LABORATORY OF EXCELLENCE RESSOURCES21
« STRATEGIC METALS IN THE 21ST CENTURY »

LABORATOIRE D'EXCELLENCE RESSOURCES21
« RESSOURCES MÉTALLIQUES STRATÉGIQUES DU 21ème SIÈCLE »

MID-TERM
ACTIVITY
REPORT
2011 - 2014

ANR-10-LABX-21-01

www.ressources21.univ-lorraine.fr

Laboratoire d'Excellence RESSOURCES21 - Université de Lorraine
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FOREWORD

RESSOURCES21 is a 'LabEx – Laboratory of Excellence' (a Geosciences research project) funded within the framework of 'Investissements d'avenir' (Investment for the future) and operated by the French National Research Agency.

RESSOURCES21 is managed by the OTELO scientific centre (Observatoire Terre Environnement Lorraine – Lorraine Earth and Environment Observatory), the scientific pole of the Université de Lorraine (France) that unites the University's different Geoscience laboratories.

Global population growth, new technologies, and the growing needs of emerging economies such as China and India, are together contributing to an ever-increasing demand for metal resources. New applications for many metals, including the rare earth elements (e.g., Nd, Dy, Eu, Tn), rare metals (e.g., Nb, Ta, Sn, W) and metals such as Sb, Ge, Ga and In, are emerging every day, both in modern manufactured products and in technology related to carbon-free energy. This issue is therefore of considerable economic and strategic importance and presents numerous challenges in geology; geochemistry; 3D-modelling; the separation of metals present in low concentrations; the recycling, environmental impact and ecotoxicology of increasingly exploited metals; and the development of new sensors and systematic monitoring means. On the basis of these challenges, RESSOURCES21 has defined the following scientific missions:

• Acquiring a complete understanding of the processes associated with the enrichment and dispersion of metals in the environment;
• Developing innovative tools for improving ore processing and metal extraction;
• Evaluating the environmental impact of these metals once dispersed across the ecosystem.

RESSOURCES21 research activities include PhD theses and post-doctoral research programmes that cover the complete metal cycle and several types of metal deposit. Two major three-year projects (one concerning the behaviour of Ni and associated elements (Co, Sc etc...) in supergene deposits, and the other concerning the rare earth elements) will combine and leverage the strengths of RESSOURCES21 over the next four years.

Education includes the training of students at all levels in the field of mineral resources, from exploration to exploitation and covering all of the associated environmental issues. Education is enhanced through the enrichment of existing courses and the setting up of new university-level training programmes such as Masters degrees. New academic programmes entirely dedicated to the metal lifecycles, from ore beneficiation to recycling, have been created, for example the Emerald Masters degree and the International Masters (Duby) in Raw Materials, Engineering and Risk Management.

Dissemination of knowledge is another of the LabEx RESSOURCES21 core objectives. This operates across different areas:

• Towards industry, via specific links with industrial partners. RESSOURCES21 laboratories already have long-established interactions with industrial partners through the GOCAD consortium for 3D-modelling and reserve estimations, and through CREGU, a subsidiary company of Areva, the world leader in uranium exploration;
• Towards the general public, through exhibitions and conferences;
• Towards political representatives. At the national and European level, the 'LabEx' label and financial means have allowed the RESSOURCES21 researchers to stabilize and enhance their standing in European networks and to increase their level of participation in the submission of European funding proposals.

A comprehensive report (in French) on the activity of the LabEx during the first two years (2012-2013) was published in early 2014 and is now available on the website. The present report provides a summary of the main activities conducted and funded by RESSOURCES21 between 2011 and 2014.

We hope that you will find this report both informative and enjoyable to read.

F. Villiéras and M. Cathelineau


UTILISATION
DES TERRES RARES
Ampoules basse consommation
Sm
Eu
Gd
Écrans plats
Y
La
Ce
Eu
Gd
Tb
Nd
Dy
Éoliennes
Smartphones et tablettes
Y
La
Ce
Eu
Gd
Tm
Yb
téléphones
NIKE
CREEKS

1

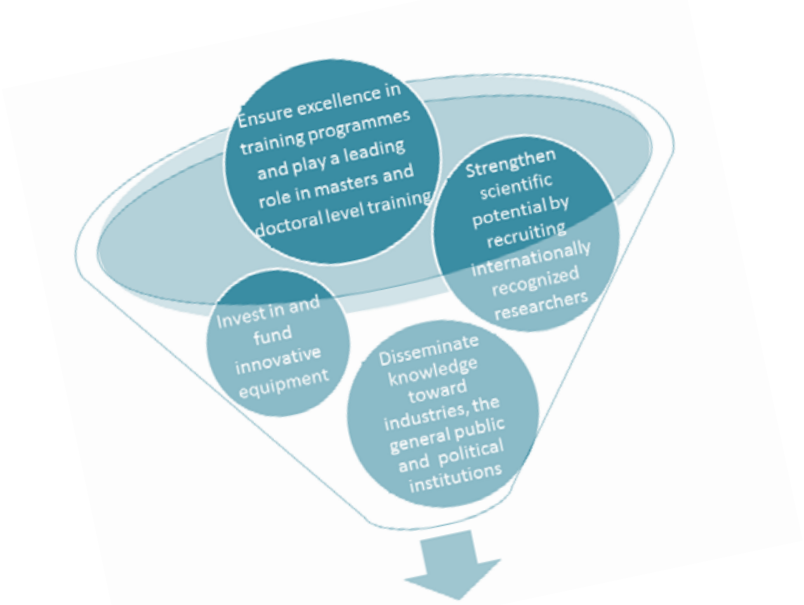
GENERAL OVERVIEW OF THE RESSOURCES21 PROJECT

WHAT IS A LABEX ?

Our country faces great challenges, and in light of these the government has made an ambitious choice: investment. A competitive call for projects led to the emergence of the 'Laboratories of Excellence' (known as 'LabEx' in abbreviated terms). These projects aim to provide leading French laboratories with the means to compete with their foreign counterparts and to develop high-quality research policies and training programmes and valuable industrial links.

Issues	• Strengthen the roles and international visibility of the best French laboratories in all disciplines and throughout the territory
Amount	• € 1 billion – interest in income to secure funding over time; planned financing for 10 years with interim evaluation
Funded projects	• Excellence of scientific laboratories / groups of internationally recognized laboratories • Projects carried by institutions or school clusters • Research and training dimension

LabEx laboratories are encouraged to foster the emergence of ambitious scientific projects, whether developed by individual research centres or by groups of research entities. The idea is that these projects will attract internationally recognized researchers and teaching staff, so as to maintain a body of scientists in France of either the highest level or the highest potential. The LabEx have a mandate to construct an integrated agenda of research, training, and promotion of projects, and they are in an optimum position to implement innovative teaching methods. The funds provided to the LabEx are public subsidies that complement the financing efforts of the participating research centres and universities.



The 100 projects selected from the first call included LabEx RESSOURCES21, which was awarded an AAA rating.

RESSOURCES21: UNDERSTANDING AND MANAGING STRATEGIC METALS IN THE 21ST CENTURY

Strategic metals are rare and non-renewable natural resources extracted from underground deposits. These metals, examples of which include nickel, antimony, gallium and other rare earth elements, possess highly specialised properties (e.g., electrical conduction, resistance to heat, torque or corrosion) that make them essential components in at least three major fields:

• Energy transition: in particular, solar energy (Ge, Ge, In), wind energy (Nd in the permanent magnets of generators), electric cars (rare earth minerals, Li batteries), low-energy light bulbs;
• The production of high-tech and high-value consumer goods such as telephones, computers, domestic appliances, aircraft, weapons and sensitive technologies (e.g., Au, Sb, rare earths);
• Increasing the strength of various tools and materials (e.g., W, Sc, Nb, Ta).

Global population growth (an estimated 9 billion people by 2050), the boom in and spread of new technologies and the growing needs of emerging economies such as China and India, are all contributing to an ever-increasing demand for rare metal resources. Given the potential threats to the supply of these metals (risk of disruption or shortages), the development of techniques and strategies for recycling metals has become a major economic and geopolitical challenge for all European countries. Unfortunately, recycling alone is not sufficient, and securing access to raw materials is essential.

To meet the challenges that lie in strategic metal supply, the Laboratory of Excellence "Strategic Metal Resources in the 21st Century - RESSOURCES21" has brought together a multidisciplinary team of researchers from the OTElo scientific centre (Observatoire Terre et Environnement Lorraine - Lorraine Earth and Environment Observatory), which is made up of four laboratories:

- LIEC (Laboratoire interdisciplinaire des environnements continentaux);
- GeoRessources;
- LSE (Laboratoire sols et environnements);
- CRPG (Centre de recherches pétrographiques et graphiques).

OBJECTIVES AND MEANS

The LabEx RESSOURCES21 research project represents an integrated scientific and educational approach for the understanding, exploitation and environmental management of strategic metal resources for the 21st century.

RESSOURCES21 addresses many challenges in terms of geology, mineral processing or environmental and ecotoxicological impact. Particular emphasis is placed on understanding the processes that lead to the formation of metal deposits, developing innovative tools for ore processing, and enhancing our understanding of the environmental impact of these metals once scattered throughout the ecosystem.

The scientific activity of the Laboratory of Excellence "Strategic Metal Resources in the 21st Century-RESSOURCES21" project focuses on three main research themes, identified as priority tasks (1,2,3), and three complementary consolidation tasks (4,5,6):

1. Understanding natural and anthropogenic strategic metal cycles and the processes of metal concentration (ore deposits) or dispersion in the environment;
2. Developing innovative tools for improved ore processing and metal extraction;
3. Evaluating the environmental impact of these metals once scattered throughout the ecosystem;
4. Developing new analytical tools for determining the distribution and concentration of trace elements within rocks and minerals, and for dating geological events;
5. 3D-Modelling of ore geometry and metal transport at corresponding scales in order to understand the distribution of metals and how ore deposits evolve over space and time;
6. Developing and refining biogeochemical environmental sensors to monitor the spread of hazardous elements and predict pollution patterns.

To achieve its goals, the LabEx RESSOURCES21 analyses the international industrial context, identifies the pertinent scientific questions and uses these findings to develop its own programmes. Several types of action are financed or co-financed:

- Short-term research programmes designed to stimulate emergent research;
- Post-doctoral and PhD fellowships;
- Integrated 3-year programmes on groups of metals that exhibit similar geochemical behaviour in the lithosphere and environment;
- Exchange of researchers (hosting internationally-renowned researchers, providing fellowships for young researchers to travel abroad in collaboration with universities or industrial partners).

Endowments to research programmes include funds for post-doctoral positions, doctoral fellowships, and operating costs, as well as the co-funding of major analytical equipment.

RESSOURCES21 AT A GLANCE

National funding: **9,000,00 euros**

Estimated project cost: **36,712,026 euros**

Duration: **104 months, from 13/04/2011 to 12/12/2019**

Project reference: **ANR-10-LABX-21-01**

Directors: **Frédéric Villiéras, Michel Cathelineau**

Trustees: **Université de Lorraine (UL), Centre National de la Recherche Scientifique (CNRS), Institut National de la Recherche Agronomique (INRA)**

Operator for the Université de Lorraine: **OTELo (Observatoire Terre et environnement de Lorraine)**

Laboratories involved: **LIEC, Georessources, LSE, CRPG**

Funding programme: **"Investissements d'Avenir" (Investments for the Future)**

Operator for research and higher education: **French National Research Agency (ANR)**

Web page: **www.ressources21.univ-lorraine.fr/**

Project status: **running**

STRUCTURE AND ORGANISATION

Since the launch of the project, the activities of RESSOURCES21 have been organised as follows:

1. The Executive Board coordinates and implements the project's activities;
2. The Research Committee sets out the priority directions for research, education, organisation and strategy, and reviews the different scientific projects as they arise;
3. The Scientific Council issues opinion on the directions proposed by the Research Committee.

Thanks to strategic seminars and thinking, we are gradually redefining the structure and organisation according to our needs. Within this new organisational framework, the Executive Board is now the central body that coordinates the activities of the project and represents the interface between political and operational levels. This structure relies on the combination of a top-down and bottom-up approach in order to connect the general strategy decided by the Executive Board and the operational committees. (An industrial committee is currently being created.)
The organisational chart below details the structure of the LabEx as well as its roles and tasks.

Board of Trustees

The LabEX R21 Board of Trustees includes the Chancellor and Supervisors from the Université de Lorraine, the CNRS-INSU (National Centre for Scientific Research -National Institute for Earth Sciences and Astronomy) and the INRA (French National Institute For Agricultural Research).

Its main mission is to validate the scientific, educational and developmental aims put forward by the Research Committee and to allocate the resources required to achieve them. It meets once a year after the Scientific Council.

The Scientific Council

The Scientific Council is composed of national and international figures renowned for their expertise in the scientific fields of LabEx R21, representatives of industrial groups and trustees, as well as members of the Research Committee.

It is a consultative organ that issues an opinion on the scientific, educational and developmental aims put forward by the different committees (emphasis on research)

The Executive Board

The Executive Board includes the LabEx Director, the development manager, the project assistant, the financial manager and the three committees directors (the Scientific Director, the Educational coordinator and the industrial coordinator)

The Executive board meets twice a month on average to co-ordinate and implement LabEx activities (Global strategy, coordination, organisation, visibility and communication of the project)

Research Comittee

The Research Committee is made up of supervisors from the six research actions and members of the Executive Board. They meet 1/month in order to:

- Expertise and evaluate the scientific exploratory projects and allocate the resources
- Advice and support the Scientific Director in the setting up of research strategy (Management and evaluation of the thematic programs)
- Ensure the visibility of our science and animate the community in their field

Education and Science communication Committee

The Research Committee is based on the pedagogical committee of OTELo. It is in charge of the:

- Definition of the pedagogical strategy to transfer knowledge from the various target audiences (students, school , general public)
- Support and Developpment of international visibility of the Existing trainings programs
- Expertise and evaluation of pedagogical exploratory projects and allocation of resources

Technology transfer Committee

The Research scientific committee will meet in average twice a year and will be in charge of:

- Raising students and doctoral students' awareness of entrepreneurship
- Expertise and evaluation of technology transfer exploratory projects and allocation of resources
- Reinforcing the synergies between the different players in the field (industrial, academic, public authorities, etc.)

OTELo scientific community

Participation in integrated programs and in exploratory projects (more than 400 people disseminated in 4 labs and 3 schools)

Executive board

Director

F. Villiéras

Scientific Director

M. Cathelineau

Educational coordinator

A-S. André Mayer

Project development manager

L. Wolff

Project assistant

I. Abildtrup

Finance and Budget Management

E. Meyer

Research activities supervisors

Metallogenesis

M-C. Boiron

B. Luais

Environmental Impact

L. Giamberini

C. Leyval

C. Schwartz

Mineral Processing

L. Filippov

J-L. Morel

G. Echevarria

Biogeochemical Sensors

C. Mustin

S. Leguedois

Analytical Boundaries

C.France-Lanord

L. Reisberg

C. Cloquet

Modelling

F. Golfier

VISION AND MISSIONS

The RESSOURCES21 Executive Board has organised several strategic workshops involving all of the research action leaders. These seminars provided an opportunity for participants to share ideas and discuss different themes in order to develop a common roadmap and vision. In order to promote dynamics and cohesion, the workshops were generally organised outside of the university infrastructure, chaired by an external person, and were followed by some kind of informal activity. New ideas, statements of a common mission and a cohesive strategic plan emerged from these seminars. The exercises were an important first step in allowing the participants to reflect on the concepts of "looking ahead", "comprehensiveness", cohesion and "targets to achieve". The management team will endeavour to ensure a continuous improvement mechanism that will allow convergence towards the strategic objectives defined. Strategic seminars will be organised at least once a year (preferably using creativity techniques to allow greater interaction with and between group members with a view to generating new ideas). Our Roadmap begins with a statement of our mission and vision: Combining sciences to reveal the natural and anthropogenic cycles of metals and using innovative techniques to manage future deposits of strategic metals.

« COMBINING SCIENCES TO REVEAL THE NATURAL AND ANTHROPOGENIC CYCLES OF METALS AND USING INNOVATIVE TECHNIQUES TO MANAGE FUTURE DEPOSITS OF STRATEGIC METALS »

MULTI DISCIPLINARY	RESEARCH	EDUCATION	PROMOTION OF RESULTS
INNOVATION	Identification and exploitation of future deposits	Development of our Labex scientific innovation courses	Transfer of technology and its integration in society: Eco-mine
INTERNATIONAL	Becoming an international benchmark in the field of natural and anthropogenic cycles of metals	International visibility and relevance of our courses	International networks and European projects


Quels projets collectifs pour une reconnaissance et une attractivité de premier plan international ?

STRATEGIC PLAN

Strategic objectives	Operational objectives
Produce new scientific and technological knowledge aimed at identifying and mining the deposits of the future («push» research)	Raise new scientific questions and challenges to develop new approaches / models / processes / questions, …
	Create a leverage-effect across the community
Act as an interface between industry players («pull» research) and public authorities (across the entire chain) and participate in the defining of European public policies	Engage industrial partners in dialogue in order to identify common challenges; to orientate and reinforce the LabEx's scientific strategy
	Work towards better integration of issues relating to mineral raw materials in French research policy
	Raise awareness concerning technology transfer
Enhance our training courses by drawing on the scientific innovations that come out of the Labex	Support and develop international visibility of the existing trainings programs in the field of Earth and Environmental Sciences at the Université de Lorraine
	Offer and fund student's projects in the field of RESSOUCES21
	Develop new training courses
Disseminate and communicate information on the importance of 'critical' raw materials in order to raise awareness in society and foster social debate	Encourage dialog between researchers, studients and industrial and societal representatives
	Launch new actions and projects in order to foster emergence of «meeting places» and shared projects involving science and society
International research visibility and research communication strategy	Foster common internal to increase recognition
	Develop and reinforce new research to foster our visibility

Operational actions

Setting up of integrated programmes: Nickel Programme 2014-2017, Rare Earth Programme 2015-2018, Programme 2016-2019, scientific animation, ..

Innovative project funding, management of project, encouragement of scientific animation...

Reinforcing the synergies between the different players in the field (industrial, academic, public authorities, ...). Consideration of the entire industrial structure allowing emergence of links with the Labex

Coordination of French actors through the French ERA-MIN group coordination, responding to strategic national and international prospectives, participation in European projects

Setting up a technology transfer committee and creation of a job-producing dynamic to further the careers of junior researchers

Representation of RESSOURCES 21 in national and international fairs and expositions, promotion of exchange between French and international students, ...

Co-funding of research internships at Master level, co-funding of PhD theses and post-doctoral fellowships

Organisation of training courses as part of our thematic workshops (summer schools, doctoral modules, Erasmus-mundus, training with industrial partners, ...)

Organisation or participation in conferences for the general public in order to sensibilise, the many uses of rare metals in everyday objects, instigation of public hearings for political representatives, ...

Developing actions and offering student internships on outreach programs (scientific dissemination workshops, information booths at public events, production of explanatory display panels, development of web documentaries,…)

Defining a research strategy: identifying a niche, defining new internal scientific challenges, developping innovations

Getting known: Participation in European projects, international hosting and mobility, organisation of conferences

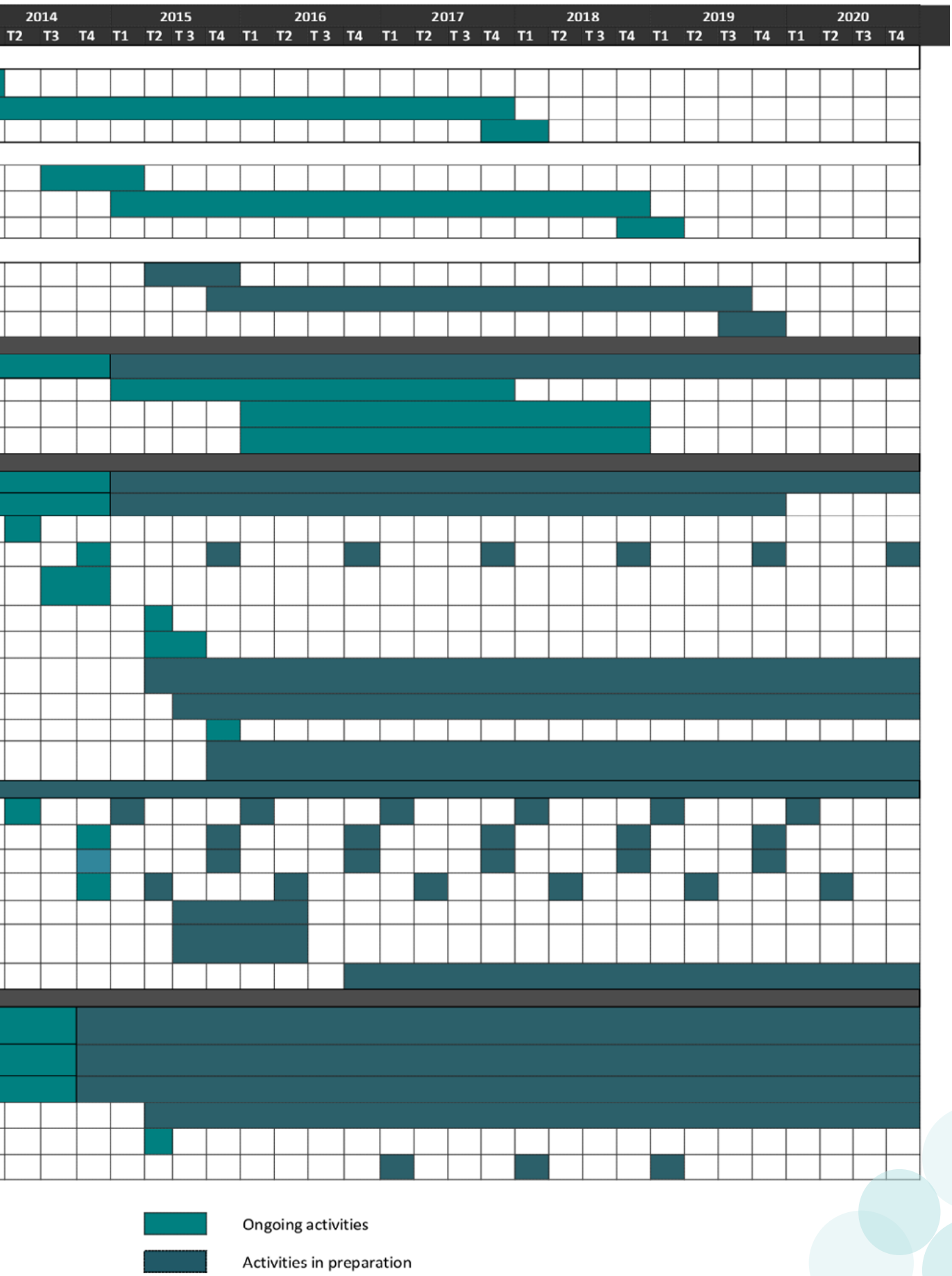
Networking through participation in European projects

Organisation of events and establishment of researcher exchange programs

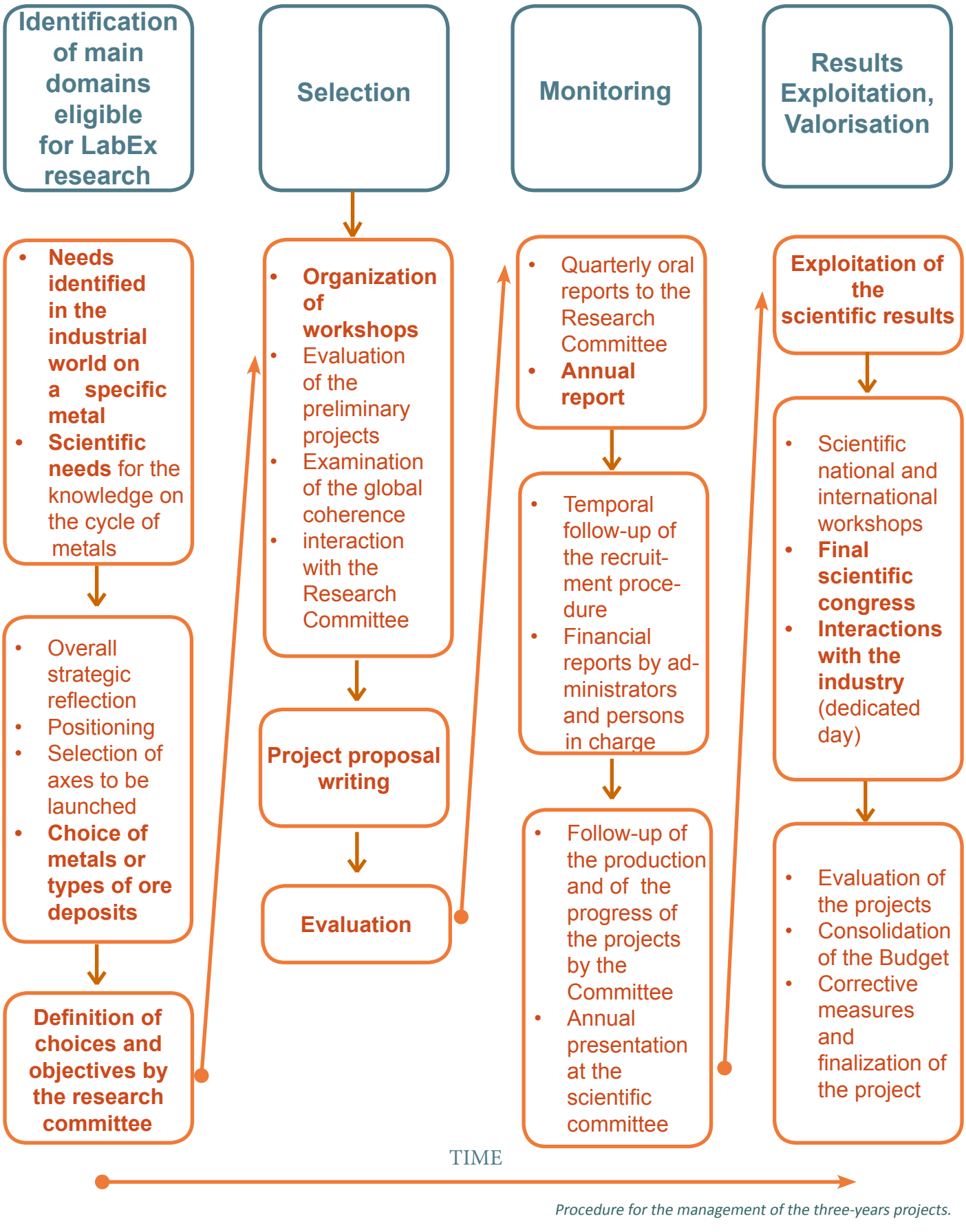
Getting recognised: Receiving external solicitations & conference invitations, governmental reguests for expertise

SCHEDULE OVERVIEW

	2013		
	T3	T4	T1
Nickel Programme 2014-2017			
Setting up of the programme: Definition of objectives and programme preparation			
Project and PhD/Post-doc execution			
Assessment and evaluation of the programme			
Rare Earth Programme 2015-2018			
Setting up of the programme: Definition of objectives and programme preparation			
Project and PhD/Post-doc execution			
Assessment and evaluation of the programme			
Third thematic integrated programme (2016-2018)			
Brainstorming and strategic thinking for definition of the third thematic programme			
Project and PhD/Post-doc execution			
Assessment and evaluation of the programme			
CHAIRS AND GUEST RESEARCHERS			
More than 10 guest researcher visits prior to 2015 and more planned			
Chair in Biogeochemistry - *Prof.Bertjan Gronenberg*			
Chair in Mineral Processing			
Chair "Biogeochemical sensors for environmental quality sensing"			
EDUCATION AND SCIENCE COMMUNICATION			
Funding student participation at congresses as representatives for the university (SIM, Québec Mines, Young Leaders Forum...)			
Funding Masters student internships			
Participation and booth at local exhibitions ("*Moments d'Invention*"- Renaissance 2013 - Nancy and *Festival du Film des Chercheurs*)			
Organisation of Student Exploration Challenge in Nancy and participation in the Québec Mines Student Exploration Challenge			
Implementation of a collaborative website (Wiki) in order to deliver accessible, general information about the economic, social and political context and the metal lifecycle (exploration, exploitation, treatment & recycling) - Student internship project			
Setting up of a education ans science communication committee (based on Otelo committee)			
Definition of the general strategies for education and science communication			
Organisation of training courses as part of themed workshops (summer schools, doctoral modules, Erasmus-mundus, training with industry, etc.)			
Call for proposal for science communication exploratory projects and execution			
Participation in the Science &You forum with experiment workshops for the general public			
Funding and/or development of dissemination and communication actions for society and schools (student projects, development of new dissemination tools and actions with other scientific departments, organisation of conferences, debates ...)			
TECHNOLOGY TRANSFER AND INDUSTRY			
Setting up meetings with industry partners in order to develop research and/or training projects			
Organising and chairing meetings of the French ERA-MIN community			
Seminar with M. ARBRE, M. Jébrak and M. McCuaig			
Setting up of a technology transfer committee, 1 to 2 meetings per year			
Definition of the general strategies for technology transfer and industry			
Launch of call for proposal, evaluation of exploratory technology transfer projects, and execution of exploratory technology transfer projects.			
Raising student and doctoral student awareness of entrepreneurship			
VISIBILITY / COMMUNICATION			
Implementation of a communication plan and development of supports/documents/tools (internet website, newsletters, flyers, booths, social networking...)			
Annual participation in the following events: PDAC, Québec Mines, Mining Indaba, CIM, SIM, Goldschmidt, IMPC, SETAC, Sustainability through Resource Conservation and Recycling			
Participation in European networks and projects (H2020, ERA-MIN, KIC,...)			
Defining a strategy of international collaboration (definition of target countries)			
Organisation of the next SGA biennial international meeting in Nancy			
International scientific meetings on Nickel REE and third thematic integrated program			


2014
2015
2016
2017
2018
2019
2020
T2 T3 T4 T1 T2 T 3 T4 T1 T2 T 3 T4 T1 T2 T 3 T4 T1 T2 T 3 T4 T1 T2 T3 T4 T1 T2 T3 T4 T1 T2 T3 T4
Ongoing activities
Activities in preparation

LARGE PROJECT SELECTION PROCESS



Procedure for the management of the three-years projects.

Procedure for short project, *see appendix, page 104*.

MARKETING COMMUNICATION STRATEGY

In the RESSOURCES21 project, we believe marketing communication to be a strategically integrated process, rather than merely a promotion of our activities. Visibility and positioning form the basis of our entire strategy, and are thus fundamental to all of the actions defined in the strategic plan. For example, when talking about research communication, we refer not only to defining a target and producing support, but also to being recognized by the scientific community for conducting the best research, producing the best science, providing the best education and developing the best networks. For example, in order to carry out the best research, we need a strategy that will foster a common project and that will in turn unite and create leverage of strengths. We will then need to develop our visibility and networking through our participation in European projects, the organization of events and through establishing researcher exchanges.

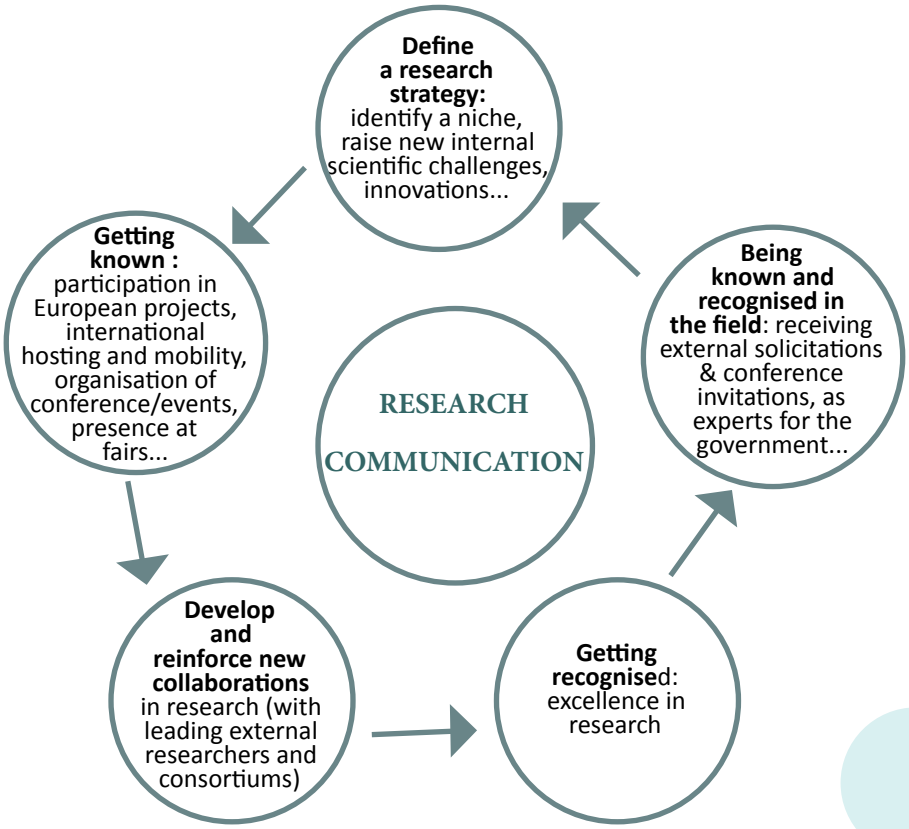
« COMMUNICATION MARKETING IS NOT ONLY ABOUT PRODUCING MARKETING TOOLS, BUT IT IS A COMPLEX PROCESS IN WHICH ALL THE ACTORS OF THE PROJECTS ARE INVOLVED »

It is thus a complex process in which all of the project's participants are involved. Marketing communication to develop excellence is therefore a comprehensive and wide-reaching process that includes all of the following:

- Developing a research strategy and fostering niche projects in order to create a leverage-effect across the community;
- Developing exchanges and attracting high-level researchers and PhD students (hosting and mobility);
- Attracting new students (supporting and developing international awareness of the existing training programmes in the field of Earth and Environmental Sciences at the Université de Lorraine);
- Reaching and informing our national and international partners and working towards better integration of issues relating to mineral raw materials in French research policy through restructuring of the research landscape;
- Developing new collaborations by becoming involved in strategic national and European projects and by participating in policy-making at an international level;
- Establishing a dialogue with and towards society in order to increase public awareness and understanding of relevant issues. This includes the creation and funding of "meeting places", shared projects involving science and society; and funding and/or developing dissemination and communication actions for the public and schools (student projects, development of new dissemination tools and actions with other scientific departments, organisation of conferences, debates, etc.).

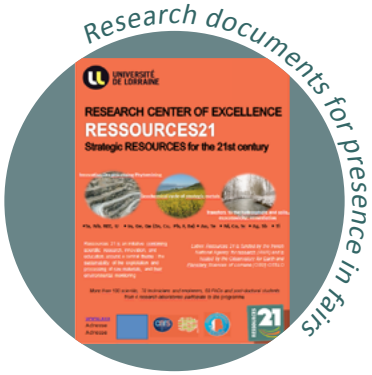
In terms of international visibility and identity, we have chosen to present ourselves as the "Université de Lorraine" rather than as a project or federation of laboratories. We believe the university image be a more perennial and recognisable marketing brand.

Communication marketing is clearly intrinsic to all our activities and the visibility actions and descriptions of events linked to research, education or dissemination of knowledge are described at the end of each chapter (I,II,II) under the heading "Visibility". We chose to focus this (next) section on the marketing tools that were established for the promotion of the project (see next page).



COMMUNICATION TOOLS

Research documents for presence in fairs



Logo

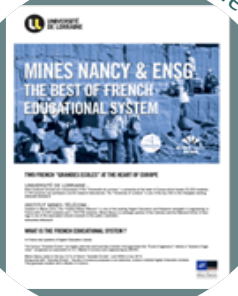
2012-2013 Activity report

Education documents for presence in fairs

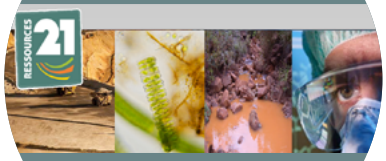
USB stick

Bi-annual newsletter (see appendix)

Education document for presence in fairs



Visual identify



Internet website

Pochette for documents for presence in fairs

Flyer

Realisation of booth structures for fairs

Installing information booths at general public events

Sweatshirts and t-shirts (RESSOURCES21 / ULJ)

Display panels for public fairs and events

Information leaflets

ACTORS AND PARTNERS

RESSOURCES21 has brought together a multidisciplinary team of researchers from the Otelo scientific centre (Lorraine Earth and Environment Observatory). More than 140 researchers, 70 technicians and engineers, and 60 postdoctoral researchers and students, drawn from four research laboratories, are involved in the project. RESSOURCES21 also benefits from the support of public entities, both industrial and commercial in nature, private companies, and external research projects.

Research structures

otelo
Observatoire Terre Environnement Lorraine

geo Ressources

Lse

LIEC

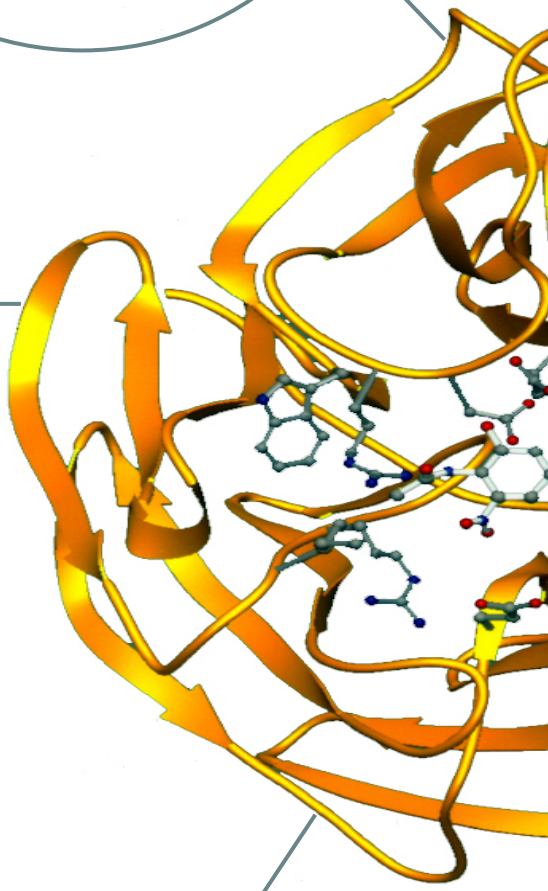
CRPG

Trustees

UNIVERSITÉ DE LORRAINE

cnrs

INRA
SCIENCE & IMPACT



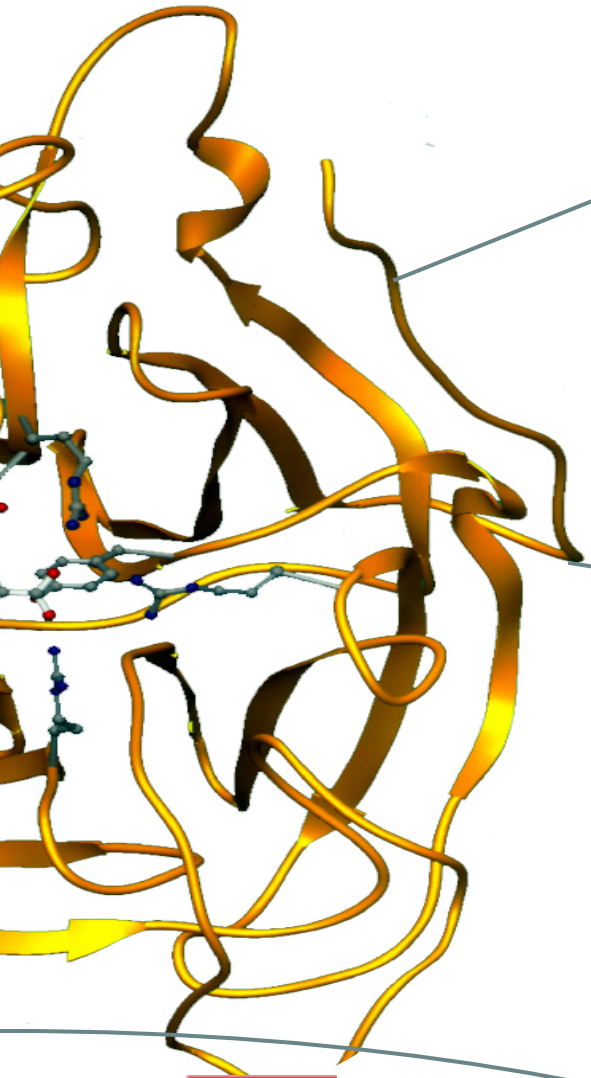
AREVA
l'avenir pour énergie

materalia
Pôle de Compétitivité Matériaux

ARKEMA
The world is our inspiration

IMERYS
TRANSFORM TO PERFORM

ERAMET



Industrial state controlled entities

lorraine.eu

ANDRA
Agence nationale pour la gestion des déchets radioactifs

INERIS
maîtriser le risque pour un développement durable

Géosciences pour une Terre durable
brgm

la Société de l'industrie minérale

BURGEAP

Excellence laboratories

VOLTAIRE

arbre

Labex
DAMAS

Serenade
Safe(r) Ecodesign Research and Education applied to Nanomaterial Development.

Industrial partners

saft

Caspeo

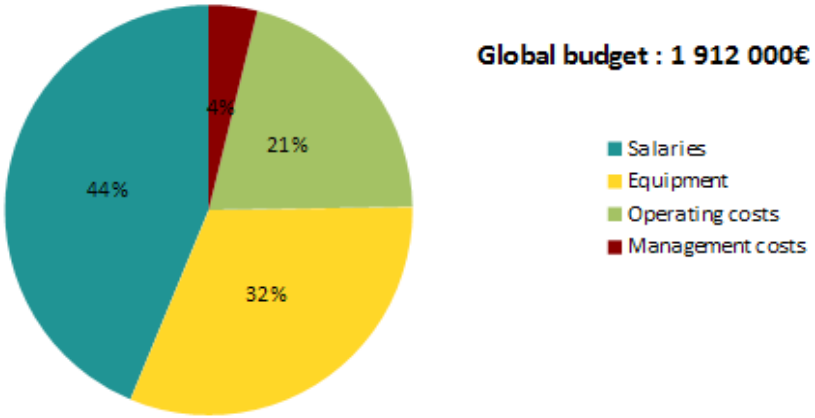
SOLVAY
asking more from chemistry®

Rhodia

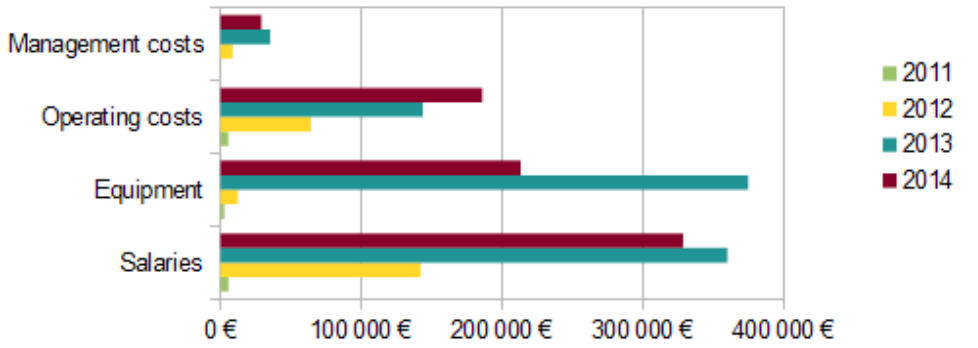
Paradigm®
ADVANCED SCIENCE FOR EVERYONE

FINANCIAL PARTITIONING

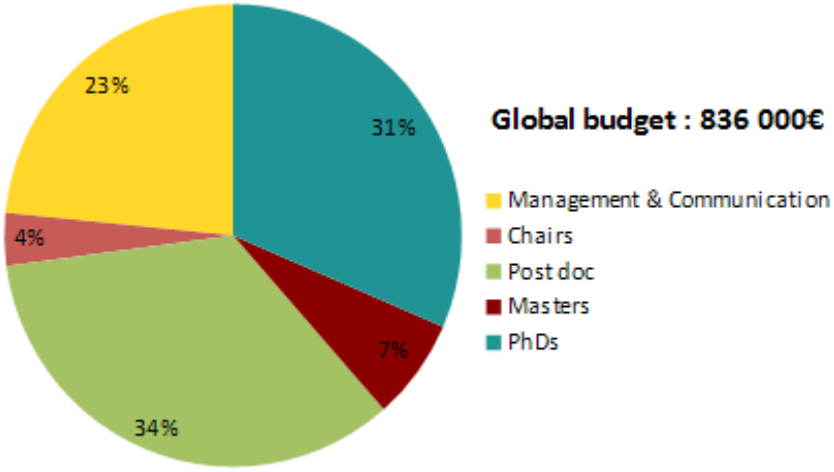
Budget repartition 2011-2014 (1)



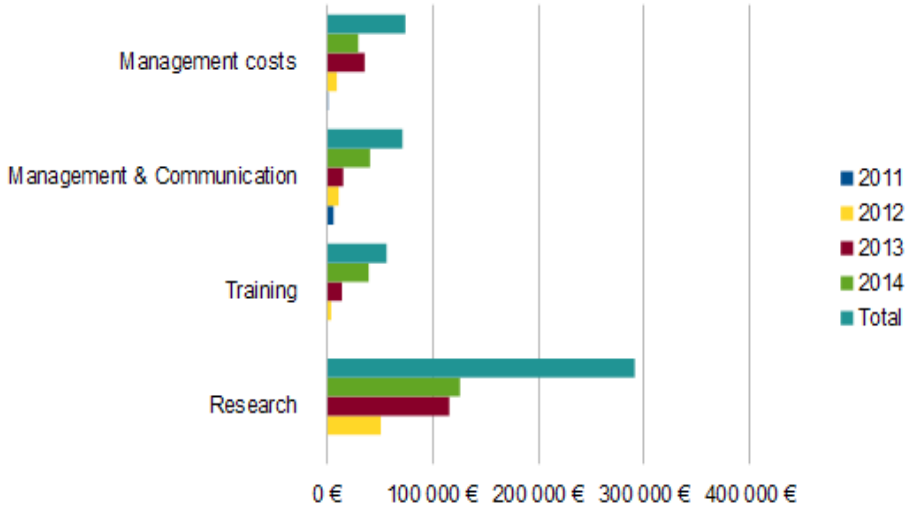
Budget repartition 2011 - 2014 (2)



Salaries 2011-2014

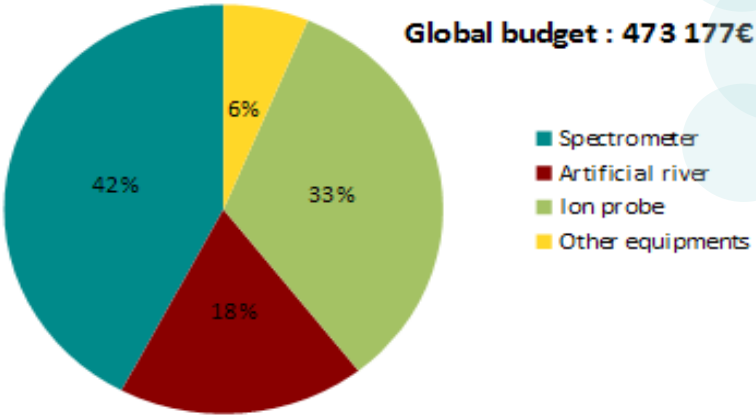


Operating costs 2011-2014

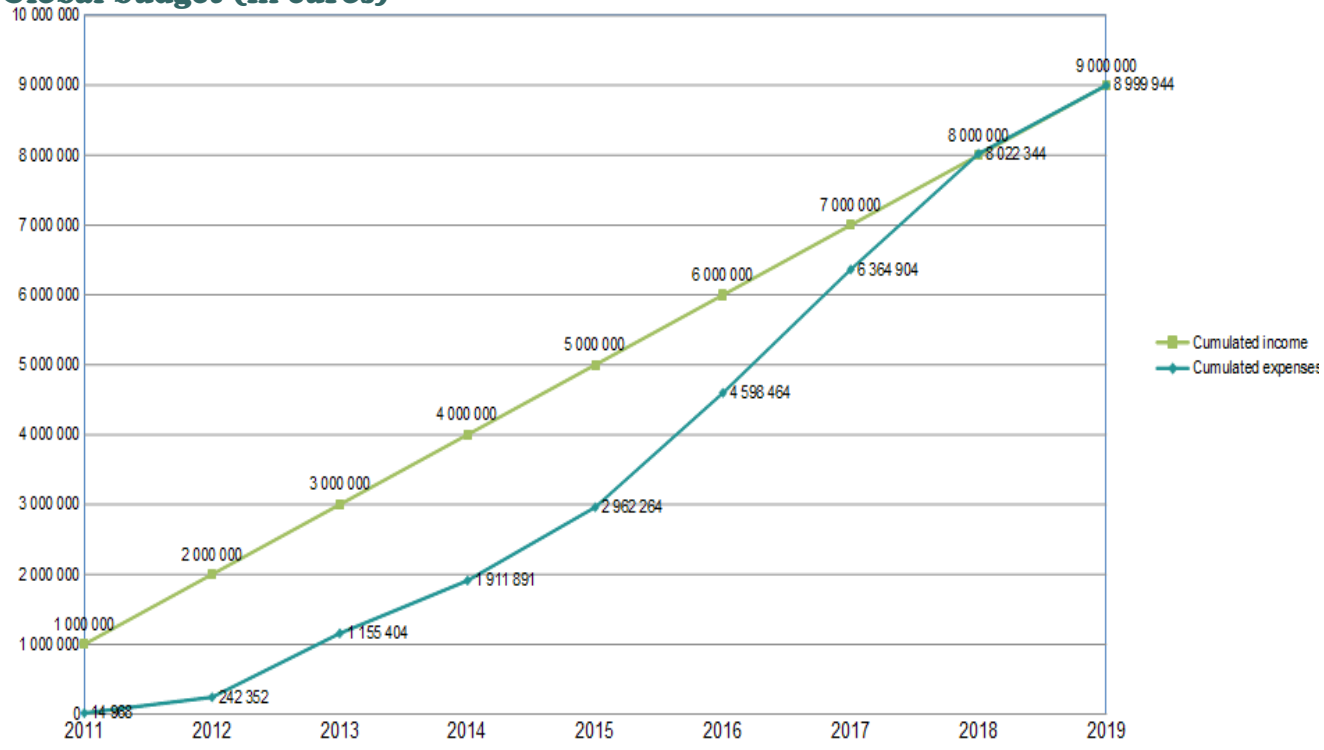


Investment 2011-2014

Global budget : 473 177€



Global budget (in euros)



II

RESEARCH

RESEARCH STRATEGY

The first two years of the project were devoted to setting up and testing the governance system and to mobilizing researchers and teaching staff from the different research units. These activities took place within the context of the merger of four universities in Lorraine and the reorganization of seven research units into four from 2013 onwards (CRPG, GeoRessources, LIEC, LSE). The organisers and coordinators launched the first calls for projects and applications, and in 2012, four post-doctoral students, six PhD students, an assistant and a project engineer were recruited.

The RESSOURCES21 doctoral and post-doctoral research programmes cover the complete metal cycle, several types of metal deposit (Ge-bearing sulphide deposits, Sn-W deposits, Sc in laterites), and environmental issues such as the behaviour of rare earth elements (REE) in the environment and the development of biosensors. Several small projects were also funded with the aim of stimulating or testing new ideas. Full details of the scientific results are given in the sections that follow. In the context of investigating the processes of metal concentration, the LabEx RESSOURCES21 teams focused their research on several groups of metals: metals required for use in the photovoltaic sector (Ga, Ge, In); rare metals associated with felsic peraluminous magmatism (Nb, Ta, Sn, W); concentrations of metals in laterites developed on ultrabasic rocks (Co, Sc); REE in carbonatites and alkaline intrusions; and the environmental impacts of REE. Alongside this, the teams have continued to work on strategic metals of high economic importance (Ni, U, Au), for which they had already acquired an international reputation, and on the impact of trace metals (Cd, Ag, Cu, Ti) and metalloids (As) in the environment.

Between the end of 2013 and mid-2014, intense brainstorming culminated in the setting up of a three-year project (late-2014 to 2017) on the behaviour of Ni and associated elements (e.g., Co and Sc) in supergene deposits. The choice of these metals was guided by the fact that Ni is one of the most important rare metals produced by French companies operating in overseas territories, (e.g. in New Caledonia) and also that Ni is accompanied by a number of other metals that have rarely been exploited up to now, such as Co, Mn, Cr and Sc. Co-beneficiation represents one of the major challenges for society as most deposits are exploited for only one metal. Mining residues, which contain low concentrations of associated metals, could constitute the reserves for tomorrow.

The second three-year programme (mid-2015 to mid-2018) will be devoted to the rare earth elements. It is a well-known fact that China's rare earth industry constitutes more than 97% of the world's REE production. In addition, the use of rare earth elements has rapidly increased in the last decade as REE have become increasingly important components in clean-energy technologies such as electric and hybrid vehicles and wind turbines. RESSOURCES21 addresses several of the most important challenges linked to the problem of ensuring an adequate supply of critical and rare metals for the coming century (SNRI 2009 and CE2010 reports).

OBJECTIVES AND CHOICE OF TARGET ELEMENTS

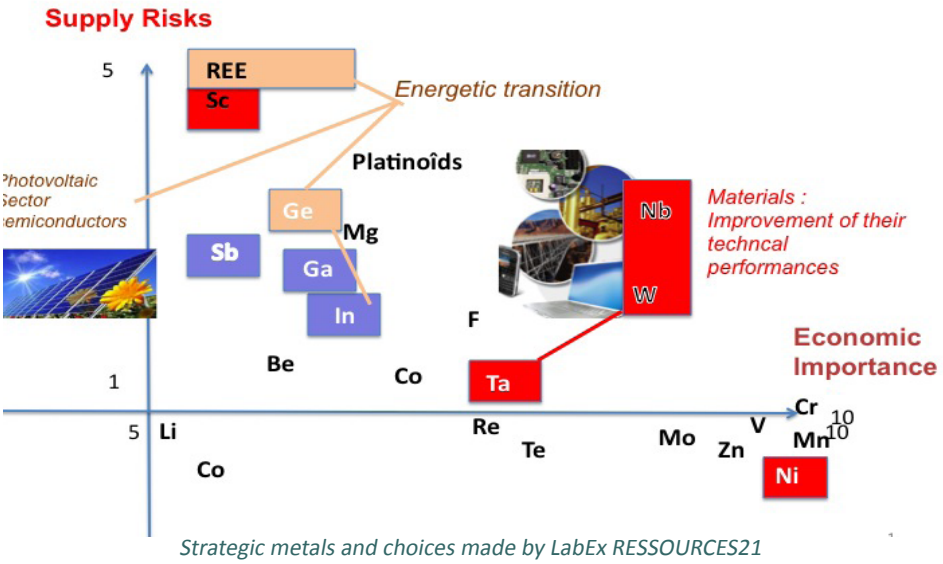
International context and justification of metal choices

Energy and mineral resources are key elements for the development of industrial economies. After more than a decade of abundance followed by a deep recession in mining activity around the world, the raw materials sector is once again in the spotlight, and is likely to remain there. This can be attributed to a combination of key factors: the new and very strong demand from the emerging Chinese and Indian economies; increased demand across the rest of the world; and the perspective of diminishing reserves of energy resources. As a consequence, the prices of many raw materials rose by a factor of 5 to 10 in the few years leading up to the international monetary crisis of 2008-2009. Although the mineral extraction industry represents only a limited part (~17%) of the global economy, access to a wide range of metals is decisive for homogeneous growth of the world's economies.

The work of the European Commission (report CE 2010) has shown that a certain number of elements, notably metals, are at risk of rapidly becoming the object of international rivalries because of their strategic importance to modern technology. Fourteen elements have been declared 'strategic' (Li, Co, Ni, Ge, Nb, In, Sb, Ta, Re, Pd, and four REE). In addition, the base metals (e.g., Cu, Zn, Fe), as well as U and Th because of their importance for the production of energy, are the subject of strong international competition. For several metals, including Ga, Ge, In, Sc, Te, and certain heavy rare earth elements, the available resources are quite limited (Watanabe, 2010).

It is therefore possible that we will soon be faced with a breakdown in the supply of metals. This is particularly true for Cu, Fe and Ni, as well as for other metals imported in large quantities from China and India. Political pressure may similarly limit the availability of certain metals. China, the world's almost exclusive producer of certain REEs (such as neodymium, essential for the production of permanent magnets in high-efficiency electric motors) is placing increasing restrictions on their export. Awareness of this issue has led national (SNRI 2009 – 'Natural Resources' Working group report) and international (the 2008 European 'Raw Materials' initiative, the 2009 Declaration of Luleå and Report CE 2010 'Critical raw materials for the EU') policy-makers to initiate programs that support the exploration, production, recycling and environmental management of primary and secondary mineral resources.

The rapidly increasing global demand for metals has spurred increased



Strategic metals and choices made by LabEx RESSOURCES21

exploration, which must be accompanied by research aimed at understanding the geological cycles of metals and the factors that lead to their concentration. These include the mechanisms leading to their incorporation as trace elements in major minerals (for example, Ge in sphalerite), and their distribution on all scales, from the mineral, to the ore body, to the region, which remain poorly understood.

Prospecting and metallogenic systems

The objectives of research in metallogeny have changed significantly over the past thirty years. The development of sophisticated ore-deposit models, essential for prospecting (e.g. Hodgson, 1987) has largely been achieved. Interest is now focused on the construction of models of 'metallogenic systems', the main objective being to understand the complete source-extraction-transport-trapping-preservation sequence and its spatial and temporal dimensions. As shown by petroleum geologists, pioneers in this domain, developing such a comprehensive understanding is the key to discovering new resources, both conventional deposits, though situated at greater depths, and non-conventional deposits (low-concentration resources and strategic metals for which the metallogenic process remains poorly known). Developing this understanding is the challenge of the 21st century for metal exploration. To meet this challenge, advances in fundamental research must be made using new approaches and tools that are somewhat analogous to those employed in the search for new hydrocarbon resources. In this light, it is clear that metallogeny is not confined simply to the study of ore bodies. Instead, this discipline raises fundamental questions concerning the processes of element transfer and concentration during the evolution of the lithosphere, in terms of conceptual understanding, quantitative characterization and integrated modelling.

Today, most systems involving strategic elements remain poorly understood, both in terms of the geodynamic context of metal enrichment and of the processes of fractionation between magma, vapour phases and sulphides. Nevertheless, the situation is starting to change.

Economic ore processing of new metal resources

Under pressure from the changing global economy, the mineral industry, regardless of the resource in question, is confronted with major technological challenges to develop techniques for the exploitation of resources with low or very low concentrations of useful substances. For example, ores with concentrations of only several hundred ppm U are currently being exploited and the development of non-conventional resources, such as pyrochlore for U or refractory minerals such as brannerite, is envisione. Other technological challenges will be raised by the potential use of marine resources for the 'small metals' (e.g., In, Ge, Te) and by the exploitation of secondary resources, such as the mining residues from which the metal of primary interest is recuperated.

These major trends engender unprecedented technological and scientific situations: very low concentrations, wide dissemination, small property contrasts between the ore and the gangue, and processes in seawater. Many of the required technological breakthroughs will involve improved separation techniques, the efficiency of which depend on enhanced selectivity. For example, new resources in REE and other rare metals have been made accessible through the development of new separation techniques using combined energetic fields or fields of high intensity..

On a more fundamental level, the development of the technology necessary for the exploitation of these primary and secondary resources will lead to renewed investigation of:

- The physics and chemistry of interactions between minerals and energetic fields (magnetic, chemical, mechanical, electric), either isolated or combined, that may intensify the fragmentation process;
- The molecular mechanisms of interaction with the flotation reagents.
- The reactivity of mineral phases: kinetics of mineral formation; element distributions; mechanisms at interfaces; mobility in supergene zone media; and processes moving away from thermodynamic equilibrium.

Environmental impact

The extraction of critical metals (CMs), their future uses, and the associated generation of waste, will raise new issues related to their potential impact on human health and the environment. Up until now, the biogeochemical cycles, bioavailability, ecodynamics and ecotoxicity of these metals have received only limited study. As is true of many other anthropogenic contaminants, disseminated metals can be found in ecosystems at varying distances from their sources, where they can have detrimental effects on living organisms. For instance, through adsorption onto suspended matter, metals can be transferred in underground and surface waters, resulting in harmful impacts on aquatic organisms and human beings.

The rare data available in the literature reveal various effects of CMs on human health. These include lung disease, genotoxic effects, carcinogenic effects and neurological effects. The most studied case is that of lanthanum, which, especially in China, is frequently used as an additive in fertilizers. La from the fertilizer accumulates in plants and is then transferred to human beings through the food chain. Understanding the links between speciation, bioavailability and ecotoxicity for such elements is crucial. At present, two main models, the Free Ion Activity Model and the Binding Ligand Model, are available for predicting bioavailability on the basis of simple speciation data, but these are applicable to only a limited number of metals. Furthermore, the models neglect the dynamic nature of equilibria in environmental systems and their predictive performance often remains limited. New models can be developed to assess metal speciation dynamics in colloidal suspensions by taking into account the geometry, hydrodynamics and electrostatics of the colloidal ligands. In soils, little is known about the fate and mobility of CMs or their interactions with the microorganisms that play an essential role in biogeochemical cycling. Critical metals are known to accumulate in microorganisms as well as in terrestrial and aquatic organisms. Specific effects related to atomic mass have even been reported. Vegetal and animal species have been shown in some cases to display bioaccumulating capacities for critical metals, although data remain relatively sparse. Examples include various fern species, squids and krill and freshwater fish such as carp. Despite the widespread and growing use of CMs in agriculture (especially in China), little information on the toxicity of these metals on organisms is available. The effects of La (together with a mixture of different CMs) on germination, growth (roots, seedlings) and the anti-oxidizing metabolism of durum wheat, have revealed significant changes in a few anti-oxidizing molecules considered as stress indicators. Cytological and cytogenetic effects of several CMs have been reported in the roots of Vicia faba. Negative direct and indirect effects of lanthanum have also been observed in crustaceans.

A review of the literature reveals a clear lack of data concerning (i) molecular and colloidal dynamic mechanisms for the transport of CMs in ecosystem compartments, (ii) the long-term ecotoxicity of environmentally relevant concentrations of CMs (hazard and risk assessment), and (iii) soil-to-plant transfers and the potential for bioremediation, mining and mineral processing strategies. There also appears to be a need to develop specific biosensors for environmental monitoring and survey.

Choice of chemical elements and organization of the research during the first three years

The first of the LabEx studies have concentrated on three groups of elements:

- Elements required for energy transition: components in photovoltaics, such as Ge (In, Ga);
- Rare metal elements used in materials (e.g., Sn-W, Nb-Ta);
- Rare earth elements used in renewable energy (e.g., magnets for wind turbines, energy-saving lights).

Three-year projects

Intense brain-storming between the end of 2013 and the end of 2014 resulted in the setting-up of two 3-year projects on the occurrence, behaviour and environmental impacts of (1) 2014-2017: Ni and associated elements (Co, Sc) in supergene deposits, and (2) 2015-2018: the REE.

2014-2017: Cycle of Ni and associated elements (Co, Sc). Leaders: G. Echevarria and M. Cathelineau

Context and positioning of the project

The first project fits into the LabEx RESSOURCES21 strategic framework by way of the setting-up of three-year projects. The proposal for a research programme on the nickel cycle was linked to two important observations:
• Before any consideration is given to the reactivation of mining operations in France, it is important to note that the main substances of economic interest for French companies and the political class (cf. report to the French senate dated March 2011) are gold in Guiana and nickel in New Caledonia, the latter being much more important than the former from an economic and societal point of view. Developing our understanding of the nickel cycle, from the exploration of ores to issues of processing and environmental impact, is therefore of high strategic importance for France. Furthermore, the numerous industrial demands related to Ni in New Caledonia are not covered by the calls for proposals from the CNRT (Centre National de Recherche Technologique).
• The potential and expertise of the LabEx RESSOURCES21 groups working on this subject is already very strong. The project will enable these teams to unite around a single theme and to enhance the visibility of this potential among the international scientific community.

General objectives of the project

The studies proposed in this project aim to contribute to a better understanding of the behaviour of the Ni-Co-(Sc) system and the lithospheric and biogeochemical cycles of these metals. The project will consider the processes of transport and entrapment of these metals in primary deposits (laterites and saprolites), and developing new concepts for the exploitation of secondary reserves (soils and technosols).

The stakes and innovative aspects of the project consist of the following:

• Testing a new concept of ore genesis to explain metal enrichment in saprolites and laterites, taking into account both the low-temperature 'hydrothermal' history linked to early deformational stages (syntectonic Ni and associated silicates) and the supergene processes. The modelling will allow us to test contrasting models (per ascensum and per descensum), in order to put forward new genetic concepts that will help improve the exploration and our understanding of ore volumes in New Caledonia.

• Scandium is a metal found in most geological formations but it has no specific deposit. Because Sc is relatively immobile under supergene conditions, it is enriched in residual soils. Preliminary studies conducted in New Caledonia point to the preferential accumulation of scandium within laterites. The objective here is to better understand: (i) the mechanisms of multi-metal incorporation, especially Sc, in goethite/hematite (e.g., Al, Cr, Co, Ni, Zn, Sc), through the development of in situ quantitative methods for the analysis of trace elements; and (ii) the effects of acid leaching or bio-hydro processing. While the preliminary studies conducted in New Caledonia suggest preferential accumulation of scandium within laterites, its geochemical behaviour during supergene alteration remains poorly understood.

• Pre-concentration of valuable elements (e.g., Ni and REE) from fine-grained low-grade ores and waste products: Most of the ores that will be discovered in the near future will certainly contain valuable elements (e.g., Ni and REE) in very low grades, typically 1% or less. These elements are finely dispersed among several minerals in the ores and new techniques will therefore be required to recover and separate them from the gangue minerals (for example in various nickel laterite ores, 75% to 90% of non-liberated Ni still remains after grinding at 15 microns). Flotation has so far been the most efficient and economical technique for recovery and separation of particles of more than 20 micron size (e.g., Ni in pentlandite). However, flotation is ineffective for fine-grained ores because of the low probability of collision between gas bubbles and particles in conventional flotation cells. Many mining companies therefore process low grade ores (such as Ni laterites) using other separation techniques, such as pyrometallurgy (smelting) or hydrometallurgy (leaching in alkaline or acid conditions e.g., with pressure acid leaching (PAL) or heap leaching (HL)). However, these procedures consume large amounts of energy and reagents (e.g., acid), have a high maintenance cost and can create environmental problems. In order to reduce the problems associated with the pyrometallurgical or hydrometallurgical procedures, we propose to pre-concentrate the valuable elements by removing gangue minerals that are not associated with these elements. This should reduce the quantity of ore that needs to be processed, resulting in a reduction of energy and acid consumption. The overall objective of the project is to develop alternative methods for the pre-concentration of valuable elements (e.g., Ni and REE) in low grade ores, and also in waste products, prior to applying pyrometallurgical or hydrometallurgical procedures.

Kerolite -Ni veins in ultrabasic units from New Caledonia.

• Understanding how hyper-accumulating plant species function and identifying the most suitable species for Ni phytoextraction or phytoremediation represent key steps towards optimizing their agronomical efficiency and value and defining strategies for the development and implementation of materials for the construction of technosoils. The objective is to optimize the biological refunctionalizing and sustainability of the ecosystem, either for phytomining or simply for stabilizing communities (plants, microbes).

• Better understanding the transfer of metals from the soil ecosystems to the hydrosphere and assessing their toxicity: The origin of high metal contents in ecosystems that have developed on ultramafic soils can be either natural or geologic, and these elements can be potentially transferred towards the aquatic environments (water columns, sediments and biological bodies). Contamination by Ni and associated metals can be exacerbated by mining procedures and by industrial activity associated with the mining operation. The objective is to develop and propose indicator tools for assessing metallic contamination of water in order to increase our understanding of mass transfers from soils to aquatic ecosystems.

In conclusion, the main research axes of this three-year project describe the surficial cycles of Ni and associated elements, including their anthropological cycles, and promote the development of new concepts and approaches.

2015-2018: Integrated project on Rare Earth Elements

The second three-year programme (mid-2015 to mid-2018) will be dedicated to the study of rare earth elements. The project is currently being finalised.

Rare earth elements (REE) are the subject of European and international research programmes and many projects are linked to enhanced exploration and to the intense search for clean and innovative ore-processing methods. Given this, the project takes into account:
• Existing know-how and the studies already supported by the LabEx RESSOURCES21 that concern processes of fractionation at the magmatic stage (carbonatite volcanoes – PhD thesis of G. Mollex), studying the impact of REE on organisms (postdoctoral research - V.Gonzales), and the effects of gadolinium issued from hospitals and released into the environment (PhD thesis of E. Perrat).
• Avoiding duplication of studies currently supported by European contracts.

The main scientific problems that were defined during the design of the 2015-2018 3-year project can be grouped according to the three main stages of the REE cycle.

Magmatic processes (Carbonatites/peralkaline intrusions):

• Enrichment factors at the magmatic stage and REE fractionation during magma crystallization;
• Spatial and temporal distribution of intrusions, relationships with partial melting of mantle rocks, and geodynamics;
• Processes of subsolidus REE redistribution in alkaline intrusions and their impact on mineralogy and ore processing.

Rare earth elements as indicators of hydrothermal processes, from the dissolution of REE-bearing phases (monazite, apatite, zircon) to their crystallization as newly-formed minerals (APS, P-thorite, P-Th-Si-U-(S)-Al system), in fluids containing Na-F-Cl-P rich fluids. REE may also used as markers of rock sources (dissolution of accessory minerals in magmatic rocks) when incorporated in gangue minerals (fluorine, carbonates) in U-F-Ba base metal deposits.

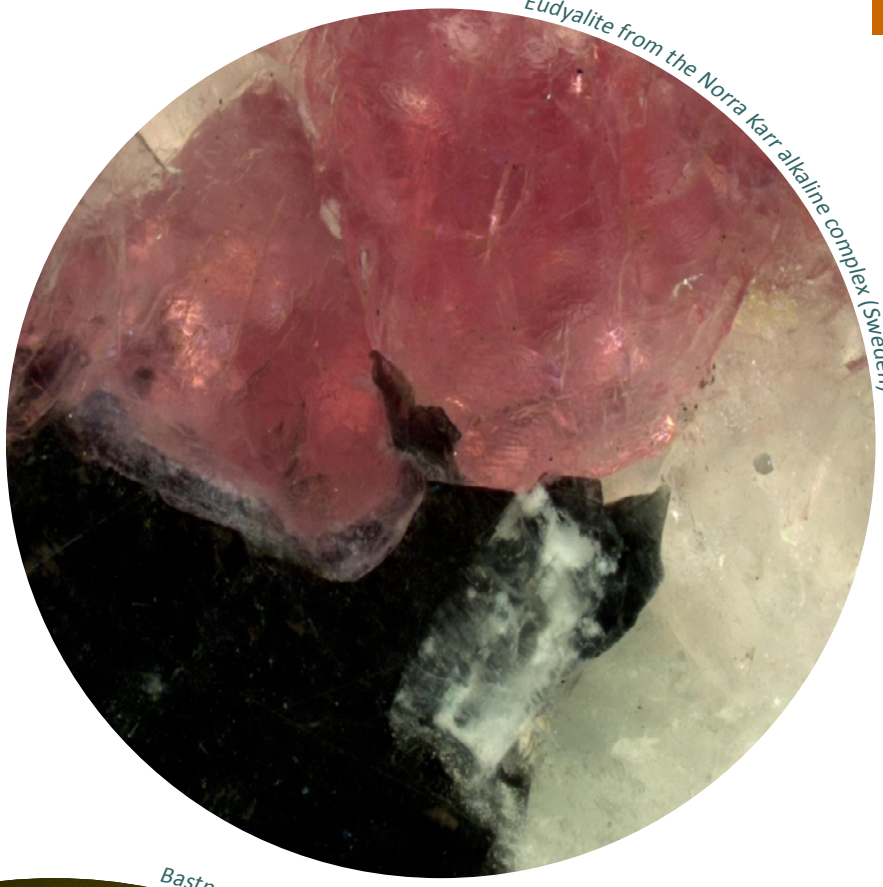
Supergene mobility and environmental impacts: in particular supergene alteration of carbonatites and alkaline rock and the impact of surficial exploitation on the environment (the mobility of REE transfer and mineralogical expression in laterites (clays, phosphates) and the impact of dissolved or colloidal forms on micro-organisms).

CONCLUSION

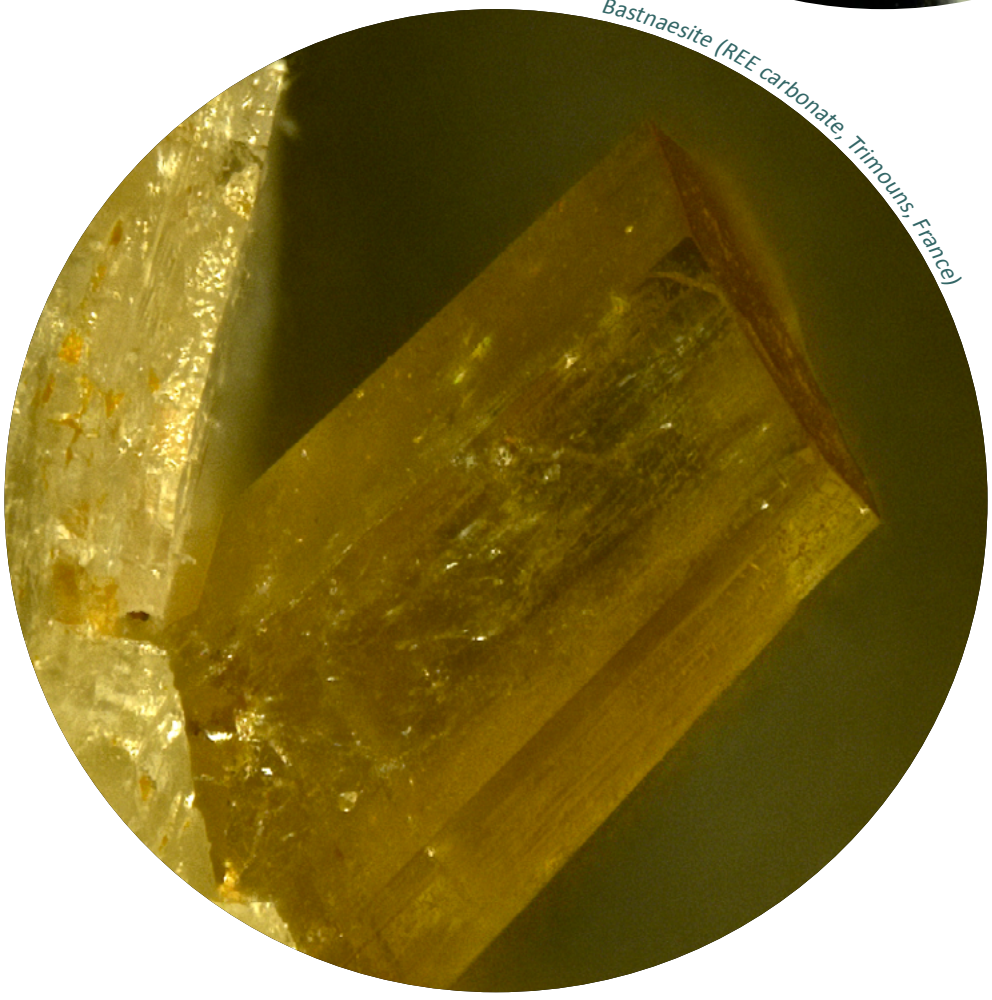
Over the next four years, the LabEx RESSOURCES21 community will be deeply involved in activities associated with the two 3-year projects. There remains place for a third project but this cannot easily be defined at present. Several options are under consideration, but these very much depend on advances made in the projects currently in progress. We can envisage:

• Enhancing the visibility of a theme that has involved considerable activity within the community, and has yielded particularly promising results, but for which the level of self-funding is insufficient;
• Developing a new theme (for example, to investigate a new group of metals or type of ore deposit);
• Reinforcing the analytical or methodological capabilities of the LabEx RESSOURCES21 teams.

The definitive choice will be made in early 2016 following prospective and strategic meetings.



Eudyalite from the Norra Karr alkaline complex (Sweden)



Bastnaesite (REE carbonate, Trimouns, France)

01

Concentration of strategic metals

MC. Boiron,
B. Luais

Georessources,
CRPG

Nickel open pit in New Caledonia

GENERAL OBJECTIVES

The two main scientific questions addressed in this work programme are:

- Where are the metals for the future ?
- What are the processes that govern the extraction of metals from their source rocks, their transport in fluid phases, and their deposition in chemical and structural traps ?

Understanding ore genesis requires knowledge of the geochemical processes that govern both the solubility of metal-bearing species in source rocks and the transport and deposition of metals in specific traps. We also need to be able to precisely relate these processes to their geodynamic context in time and space, at scales ranging from the lithospheric plate (thousands of kilometres) to local traps (metres to hundreds of metres), with a particular emphasis on the metallogenic province (10-100 km).

The main scientific issues addressed concern:

- The large metallogenic systems that produce giant deposits. In particular, to what extent did magma genesis and partial melting during the growth of the juvenile crust contribute to early fractionation processes ?
- The key factors that lead to metal remobilization by fluids and fractionation of the fluid phases and the related physical-chemical processes that produce ores;
- The specific processes that lead to extreme accumulation in the crust, especially for metals whose cycles are only partially known (e.g. Ge, In, and rare metals - Nb, Ta, REE - that are present in trace amounts in

minerals). Understanding the full cycle of extraction, including the source rocks, transport, and deposition, requires new data and approaches. The source rocks of many metals remain largely unknown, and for metals present in only trace quantities in minerals, even the mechanisms of deposition are poorly constrained (e.g., incorporation of In in sulphides, in tin or in VMS ores, Ge in hydrothermal sphalerite, and Sc in iron oxides).

The speciation in solution (in both the liquid and vapour phases) and the factors influencing deposition are also generally unknown for the strategic metals. To resolve this, there is a need for experimental metallogeny, magma characterisation, detailed mineralogical study, precise reconstruction of paleofluid compositions and conditions of migration, and dating of fluid events with respect to geodynamics. This is especially true for metals whose lithospheric cycles remain poorly understood.

In the first years of the LabEx RESSOURCES21 project, work has focused on four groups of metals that are considered to be critical raw materials due to their supply risk and the increasing demand for cutting-edge technologies:

- Metals used in the solar power industry (e.g., Ge, Ga, In);
- Rare metals (Nb, Ta) associated with peraluminous magmatism (Sn, W, Li);
- Rare earth elements in carbonatitic magma;
- Metals in altered basic rocks (Ni, Co, Sc).

I. BASE METAL SULPHIDE DEPOSITS HOSTING STRATEGIC METAL CONCENTRATIONS

Germanium cycle in sulphide minerals: elemental analyses and isotopic tracing.

(Rémi Belissont, PhD, Oct. 2012 to Oct. 2015)

The aim of this PhD project is to understand the mechanisms and thermodynamic conditions involved in the dissolution, transport, and deposition of ore minerals enriched in Ge and associated critical metals. The originality of this work lies in the coupling of multi-scale elemental (Ge and related minor/trace elements) and isotopic (Ge, Fe, S) tracers in order to study compositional variations and isotopic fractionation on scales ranging from the ore deposit to zoning in individual minerals.

The study combines mineralogical observations and in situ LA-ICP-MS analyses of minor/trace elements and bulk Ge isotopes in Ge-rich zoned sphalerite from the Saint-Salvy deposit (SW French Massif Central). This integrated approach has allowed investigation of element distributions and substitution mechanisms and an examination of the wide range of Ge isotopic fractionation in low-T processes, and provides implications for the geochemical signatures of sphalerite ore deposits. The main results of the study have recently been published (Belissont et al., 2014) and are summarised below.

1. LA-ICP-MS proves itself to be a powerful tool for measuring in-situ trace and minor elements that occur as solid solutions in sphalerite. ^{74}Ge is the most suitable Ge isotope for analysis with LA-ICP-MS because of negligible isobaric interferences. Principal component analysis (PCA) of the LA-ICP-MS dataset revealed an antithetic distribution of element clusters in sphalerite. Cu and the trace elements Ge, Sb, Ag, and As, are enriched and correlate positively in sector zoning whereas Fe, Cd, In, and Sn are enriched in dark brown rhythmic bands *(Fig. 1.1)*. Such a distribution implies crystallographic controls on the incorporation of trace elements.

2. Regardless of the zoning type, notable coupled substitutions are suggested in binary scatter plots: $2Zn^{2+} \leftrightarrow Cu^{+} + Sb^{3+}$ and $3Zn^{2+} \leftrightarrow Ge^{4+} + 2Ag^{+}$. The data also suggest the substitution $3Zn^{2+} \leftrightarrow In^{3+} + Sn^{3+}$ + vacancy, although the Sn oxidation state needs to be verified using appropriate methods (e.g., XAS, mXANES/EXAFS). Fe and Cd are mainly involved in direct $Zn^{2+} \leftrightarrow (Fe^{2+}, Cd^{2+})$ substitutions. Of note, the Cu content approaches the sum of all available tri- and tetravalent cations. In this way, Cu (occurring as Cu+) could provide charge-balance for the entire, broad set of coupled substitution mechanisms responsible for incorporation of the whole range of trace elements in Saint-Salvy sphalerite, especially Ge, Ga and Sb+. In situ mXANES demonstrated that the oxidation state of Ge is the same between sector zoning and rhythmic banding in any given sample. However, both the incorporation and partitioning of Ge between these two types of zoning are enhanced with increasing reduction of Cu+ and not Cu+ concentration. It is not clear at which level this dependence on Cu reduction occurs: whether it affects Cu+ behaviour in the fluids or instead favours the incorporation of Ge by the formation of [$(Ge^{4+}Cu^{2+})$

S2-3] clusters on the ZnS surface during growth (Belissont et al., 2014).

3. Applying multivariate statistics (PCA) to trace element features in sphalerite from Saint-Salvy and in other types of sphalerite ore has allowed us to establish a comprehensive framework with which to discriminate between different ore genetic types, as suggested by previous studies (e.g. In for magmatic-related hydrothermal systems, and Ge for low-T deposits).

4. Germanium isotopes yielded a broad range of ∂^{74}GeNIST3120a values, from -2.07±0.37 to +0.91± 0.16‰ (2σ SD), highlighting the large isotopic fractionation that occurs during sphalerite deposition in low-T hydrothermal systems. The positive correlation between bulk ∂^{74}Ge and bulk Ge content in sphalerite suggests mixing between different fluids during sphalerite precipitation (Rayleigh fractionation) in open hydrothermal systems. These results highlight the exceptional features of the Saint-Salvy deposit (France): the outstanding compositional zoning in the sphalerite crystals, their very high Ge contents compared to other Ge-bearing sphalerite ores, and the broad range of Ge isotopic compositions.

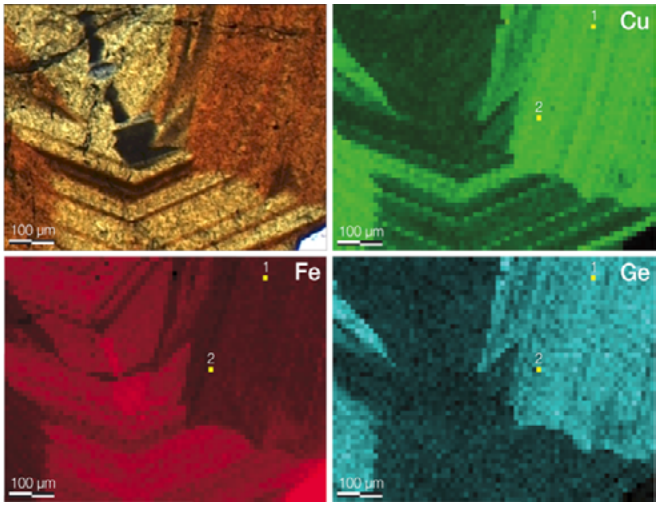


Fig. 1.1: µ-XRF mapping of Fe (left) and Ge (right) obtained on sphalérite using Grenoble ESRF Synchrotron facilities (coll. M. Munoz, ISTerre). Ge is enriched in sector zonings and is correlated to Cu (Belissont et al, 2014).

The role of the S^{3o-} ion in thermochemical sulphate reduction: geological and geochemical implications

(Truche et al., 2014)

Thermochemical sulphate reduction (TSR) plays a crucial role in the global sulphur cycle in the Earth's crust and may affect current and past sulphur isotopic records. However, the extrapolation of experimental reaction rates measured at high temperature (above 200°C) towards lower temperatures, as well as the interpretation of the sulphur isotopic fractionation recorded in natural samples, requires an accurate description of the elemental steps controlling these reactions. We have addressed this question by performing a range of specific experiments. Based on in situ Raman spectroscopy measurements, we show that, whatever the electron donor considered, the trisulphur ion S^{3-} is the dominant intermediate sulphur valence species involved in abiogenic sulphate reduction processes initiated by H_2S, over a wide range of temperatures (100-350°C) and solution compositions *(Fig. 1.2)*.

The in situ spectroscopic data unambiguously demonstrate the presence of S^{3-} at temperatures as low as 100°C. The presence of S^{3-} is crucial for achieving rapid sulphate reduction, especially in the low temperature processes that generally govern the co-precipitation of strategic metals (Ge, In) in base metal sulphides (Cu, Zn). We propose that any dissolved constituent which decreases the dielectric constant of water, or which yields favourable S^{3-} coordination, will stabilise the trisulphur ion (and thus promote TSR) at T and pH conditions that are less extreme than previously thought. The importance of S^{3-} in these processes should also be taken into account when discussing the mass-independent sulphur isotopic compositions recorded in natural and/or experimental TSR-related samples.

The second project raises the exciting possibility that thermal sulphate reduction (TSR) could provide a previously unconsidered means of producing mass-independent isotopic fractionations in sediments. To test this possibility, we are currently analysing sulphides and sulphates from sedimentary rocks affected by the TSR process, using both in situ and bulk techniques. The in situ analyses are performed on the Cameca ims 1280 ion probe at CRPG, and the development of a new set of sulphide and sulphate standards, essential for assuring the accuracy of these measurements, is currently under way. Bulk multi-isotope sulphur analyses are currently performed in Paris, at IPGP, but we are in the process of constructing a fluorination line that will allow such analyses to be performed at CRPG in the future.

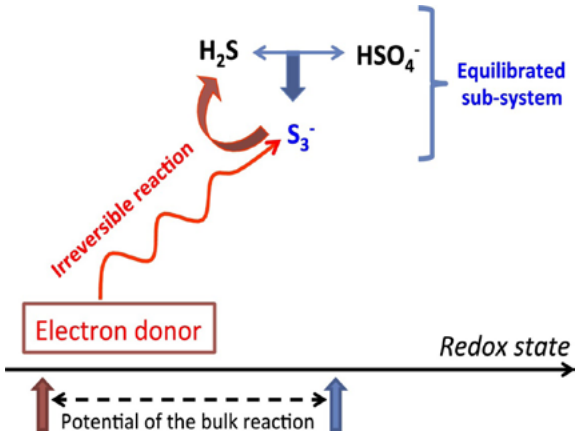
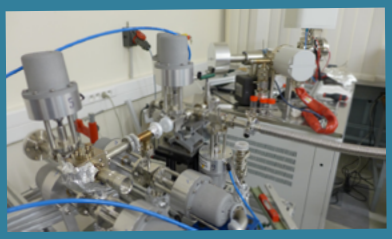


Fig. 1.2: Proposed S−3 reaction pathway for thermochemical sulphate reduction (Truche et al., 2014)

DATING K-BEARING MINERALS IN ORE DEPOSITS USING THE K-AR METHOD

The determination of the timing of processes is a fundamental parameter for any reconstruction and modelling of metallogenic processes.

However, most fluid-rock interactions in sedimentary basins lead to the formation of relatively small minerals, belonging to a limited number of mineral groups: carbonates (calcite, dolomite, ankerite, siderite), silicates (quartz, clays, feldspars (Na, or K), and sometimes phosphates. Only a few of theses phases can be dated, especially amongthe K-bearing phases (K-Ar system). As dissolution of detrital K-feldspar and mica components is concomitant with crystallisation of authigenic illite, the induced isotopic resetting of the detrital minerals must depend on their size and on the temperature of alteration (coarse mineral grains being more difficult to reset at low temperatures). For these reasons, separation of size fractions as small as possible is recommended, particularly in sandy sediments, but also for argillaceous sediments, as this will minimise contamination by coarser non- or partially-recrystallized K-bearing framework minerals (Clauer and Lerman, 2012). The objective is to set up a new K-Ar laboratory equipped with an in-house gas extraction line, an Argus spectrometer, and a preparation room including a flow-trough refrigerated centrifuge for the precise separation of fine-grained fractions down to fractions of micrometers (0.2, 0.1 microns). The line is almost finished and tests will begin in early 2015.



ENHANCED PERFORMANCE OF THE CAMECA IMS 1270 ION PROBE

In 2014, the RESSOURCES21 LabEx contributed to the funding of an extensive upgrade of the Cameca ims 1270 ion probe, an instrument that has been used for over 15 years at CRPG for performing high-resolution in-situ analyses of a wide variety of geologic materials. The upgrade included the installation of a completely new set of electronics for the control of the instrument, as well as the motorisation of the various diaphragms and slits that control the form of the ion beam. The pumping system was disassembled and fully reconstructed, facilitating achievement of the ultra-high vacuum required for precise analyses. Together, these modifications will significantly increase the mass resolution of the ion probe, opening up numerous new analytical possibilities. The renovated instrument, which is now the equivalent of the more modern Cameca ims 1280 E7 ion probe, will play an essential role in many of the LabEx RESSOURCES21 projects, as well as in other research conducted by the OTELo laboratories. An additional important development in the CRPG ion probe laboratory this year was the performance of the first high resolution K-Ca isotopic measurements on the Cameca ims 1280 HR2, opening up exciting new possibilities for in situ dating.

II. RARE METAL (W, SN, TA, NB) MINERALISATIONS

The example of the French Massif Central

(Matthieu Harlaux, PhD, Oct. 2013 – Oct. 2016)

Work carried out on rare metal mineralisations in the French Massif Central (FMC) has yielded the following results:

- The occurrence of a syn-metamorphic Namurian tungsten mineralizing event in the Cevennes region (SE part of the FMC) at around 325-315 Ma, under regional compressive deformation and coeval with regional low-pressure metamorphism and the intrusion of the Rocles peraluminous granite. Field investigation demonstrated that several tungsten mineralizing events occurred during the Neo-Variscan period; younger ages were found in other districts of the FMC. Whole-rock geochemical data obtained from aplo-pegmatites that cross-cut the Sn-W quartz veins indicate the contribution from rare-metal peraluminous magmatism at ~306 Ma, which is shown for the first time in this part of the FMC and suggests that this type of magmatism could be a general phenomena in this part of the Variscan belt.
- The role of late peralkaline hydrothermal paragenesis in the Puy-les-Vignes tungsten deposit (Limousin, NW part of the FMC). Of note, this mineralogical and geochemical study revealed the existence of a new type of rare-metal oxide, enriched in Nb, Y, HREE, W, and U, and the presence of precious metals (Au-Ag) associated with the main tungsten mineralization. This discovery could represent the first evidence within the FMC for a contribution of peralkaline magmatism in a regional context dominated by peraluminous magmatism, and it opens up new perspectives both for understanding rare-metal metallogenesis in this Variscan crustal segment and for the exploration of the rare-metal economic potential at Puy-les-Vignes and in the wider Limousin area.
- A comparative study of the capabilities of a new prototype of time-of-flight (TOF) LA-ICP-MS for multi-elemental analysis of fluid inclusions. This analytical development at ETH Zürich (Switzerland) demonstrates the advantages of the TOF in terms of precision, accuracy and limits of detection compared to the traditional types of sequential ICP-MS (quadrupole and sector-field). Combined with its quasi-simultaneous acquisition of all isotopes from ^{6}Li to ^{238}U, this makes the TOF LA-ICP-MS a very powerful and promising tool for the analysis of trace elements in geological fluids. This analytical development will be soon applied to a study of W-Sn quartz veins from the FMC.

III. GEOMODELLING APPLIED TO MINERAL RESOURCE EXPLORATION

A new tool for targeting desposits

(Pablo Mejia-Herrera, PhD, defended, in December 2014)

Understanding the history of sedimentary basins is of paramount importance in mineral exploration, allowing us to identify brine pathways, establish the physical and chemical processes involved in ore formations, and ultimately, predict the locations of potential economic mineral resources. Advanced modelling technology such as 3&4D geomodelling can be used to explore old matured mining fields with a new pair of eyes. This is usually achieved through the use of 3D reconstruction and restoration tools that take into account geometric or geomechanical constraints. For sediment-hosted ore deposits, the unfolding and unfaulting techniques allow us to visualize the impact of tectonic events during the mineralization stages and their role in the final ore-distribution. This 3D restoration procedure has been applied with success to the German and Polish Kupferschiefer, a sediment-hosted polymetallic (Cu, Ag, Au, PGE) deposit, which is one of the most important sources of copper and silver in the world. Four-dimensional restoration-decompaction modelling allows us to reconstruct the burial, deformation and natural hydro-fracturing history of intra-basin sediment-hosted ore deposits *(Fig. 1.3)*. Results highlight the role played by the Late Cretaceous-Early Paleocene uplift which affected Central Europe. This uplift provided the conditions for hydrothermal recirculation of mineralizing brines, explaining the location of Cu (Cu-Fe) sulphides ores in the area. Simulation shows good agreement between the spatial hydro-fracturing index and the location of the Cu (Cu-Fe) sulphides exploited today. A similar approach has been adopted for the Mount Pleasant Au-deposit (Western Australia). Such a methodology can be generalized to any ore deposit where damage and rock-fracturing has driven the mineralization processes involving strategic metals.

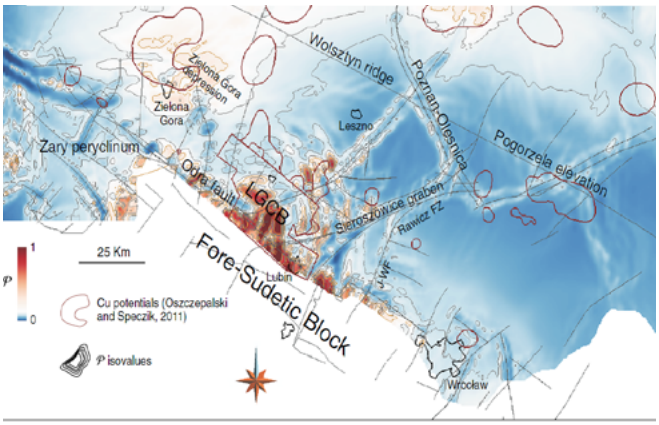


Fig. 1.3: Predicted probabilities of Cu-potentials (P) in the Fore-Sudetic Monocline. (FSM). Some main structures and Cu potential locations are highlighted. The match between high P and the Cupotential areas are close to main regional structures as the Pogorzela elevation, the Sieroszowice graben and the Odra fault system among others (from Mejia-Herrera et al., 2014)

Carbonatitic magmatism at Oldoinyo Lengaï

(Gaëlle Mollex PhD, Oct. 2013 - Oct. 2016)

Carbonatitic magmatism has existed throughout the Earth's history, but only one stratovolcano, Oldoinyo Lengaï (OL, Tanzania), produces such magmas today. These magmas are among the rarest Si-free melts on Earth and represent the main deposit for critical metals such as REE. The OL volcano exhibits three formations, representing the three phases of construction. Lengaï 1 was mainly phonolitic, Lengaï 2 was nephelinitic, and the most recent activity has been characterised by natrocarbonatite emission. The transition between the different phases is not yet fully understood, and must be investigated in detail. This project focuses on a petrological and geochemical study of cumulative xenoliths that poured out at the surface during the 2007-2008 sub-plinian eruption of Oldoinyo Lengaï. These samples were partially crystallized in the mushy zone that forms at the margin of the magma chamber, and thus provide a record of deep magma-chamber processes. Crystallization sequences and compositions of melt inclusions trapped in the different minerals (from the first to crystallize to the last) provide new information on the temporal evolution of Oldoinyo Lengai. Geothermobarometry combined with previous experimental work, indicate that advanced differentiation takes place within crustal magma chambers. During crystallization, magma composition varies from phonolitic to nephelinitic. It is proposed that crystallisation process, from a phonolitic melt, takes place at the margins of a stable magma chamber at around 300 MPa (the deepest ever crystallisation documented for OL), and that fractionation at that depth is associated with immiscibility of a calcio-carbonatitic-like melt (=high T immiscibility). Immiscibility is thus identified at much higher temperatures than before. Preliminary noble gas isotopic results (He, Ne, Ar) on these cumulates also show that interactions occur at magma chamber depth between the magma itself and the previously hydrothermally altered magma chamber margin.

This preliminary study emphasises the presence of carbonatitic melts at different stages of magma evolution, with the first identified stages of immiscibility being associated with calcio-carbonatitic magmas (which then evolve to natro-carbonatites). The OL system has thus been shown to represent a real and valuable analogue for old calcio-carbonatite-related mineralized deposits. Further objectives will be (1) to constrain the evolution of trace element concentrations (including REE) in minerals and melts during the different steps of differentiation, and (2) to identify the optimum physical conditions required to produce the highest critical metal concentrations in carbonatites. These results will be of significant value for REE exploration and exploitation.

V. NI, CO, MN, CR, AND SC CONCENTRATIONS IN LATERITIC PROFILES IN NEW CALEDONIA

Scandium in laterites

(Post-doctoral research, M. Ulrich, 2012-2013)

The mobility of Sc and associated elements, together with characterization of the host minerals, has been studied along an alteration profile in New Caledonia. The main objective of this work concerns the transport and trapping mechanisms of such metals in laterites based on our previously- gained understanding of major concentrations of Ni and Co. The geochemical data from profiles in various protoliths (dunite, harzburgite, lherzolite, gabbros and other intrusive rocks) have shown Sc enrichments along the alteration profiles, especially in the fine-grained saprolite (yellow laterite) and in the transition horizon. Sc concentrations range from 7 ppm in the harzburgitic protore to 75 ppm in the yellow laterite. LA-ICPMS for localized quantitative analysis of Sc in minerals has shown heterogeneities in concentration at the scale of tens of micrometers in the red limonite and in pisoliths *(Fig. 1.4)*. In contrast, REE contents are low in laterites compared to those developed on REE-rich rocks such as carbonatites and alkaline intrusions. REE concentrations in goethite can be as much as 1 to 10 times higher than chondritic values due to the link between enrichment and the lateritic process, but they still remain 10 times lower than measured concentrations in similar profiles from Cuba.

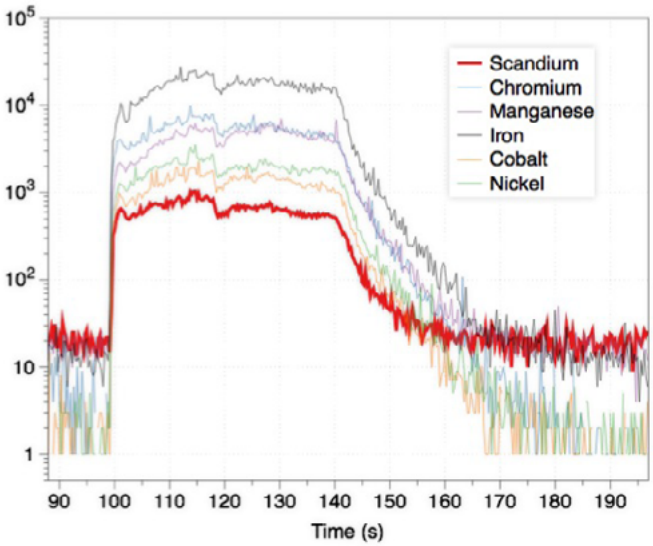


Fig. 1.4: In situ LA-ICP-MS mass spectrum of pisolites from lateritic profiles in New Caledonia, analysed for Ni, Co, Cr, Sc (Sc content around 100 ppm after calibration on external and internal standards).

02

Ore processing: developing new ecologically efficient procedures for the concentration and recycling of metals

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G. Echevarria,
JL. Morel

GeoRessources
LSE

Main objectives

Strategic metal ores are characterised by low contents of beneficiable metals, extremely fine dissemination of the metal-bearing minerals, and low property contrasts between these phases and the embedding minerals. Advanced knowledge of these materials benefits from the outputs of PRA1 and PRA4. Progress in beneficiation requires the development of both novel fragmentation processes to obtain very fine grain sizes and advanced separation techniques. The reduction of energy consumption also represents a major challenge. To this purpose, phyto-remediation techniques have been applied to metal beneficiation from low-grade ore bodies. Finally, recycling strategies need to be adapted in order to extend the life cycles of these metals.

In the quest for improved separation of constituent minerals with low reactivity contrasts, the scientific challenges converge towards the molecular-scale understanding of the mechanisms of interaction between solid matter and energy fields and/or reactants.

The PRA2 work programme pursues several objectives:

- Interaction with pulsed fields

In order to generalize promising preliminary observations, we will investigate the molecular mechanisms of combined oxido-reduction reactions and the local thermal processes that accompany micro-fragmentation upon application of high-power electromagnetic or mechanical pulsed fields. This may lead, for example, to the recovery of submicronic conductor particles of the platinum group metals, gold, etc. by preliminary embrittlement and flotation. This approach may also prove effective for the recuperation of rare metals from low-grade ores and rare earth elements from various primary and secondary waste products.

- Synergistic effects in the flotation process

The molecular mechanisms and thermodynamics of adsorption of surfactants of different molecular structure at solid interfaces will be revisited and modelled in order to improve flotation selectivity. Combining surfactants with pulsed fields or high-energy mixing/reactions should result in enhanced separability of ultra-fine materials that, because of their ability to aggregate spontaneously, are impossible to separate using conventional techniques. The mechanisms of coagulation-floculation in pulsed or sheared fields will therefore be studied. In order to achieve optimal organisation of solid, liquid and gas phases in flotation devices that use ultrasonic activation, we will study the physico-chemical and mechanical processes that occur at the solid-liquid-gas boundary.

- Reactivity of mineral phases in high ionic strength media

The prospect of extracting submarine mineral resources raises many questions. The programme will address the interactions between sulphide ores and sea water in terms of oxido-reduction reactions and geochemical alteration, as well as the influence of seawater composition on fragmentation and separation processes.

• Development of new recycling concepts

Our first approach to recycling metals from manufactured products will involve adaptation of the comminution-separation techniques currently applied to raw materials, especially those that combine flotation and magnetic separation. These methods will be supplemented with thermal and hydrometallurgical techniques aimed at extracting selected metals. In the long term, significant benefits could be obtained by integrating recycling requirements into the conception of both materials and manufactured goods. Throughout all phases of this study, we will continue to develop our collaborations with specialists in metallurgy, in particular those developed within the framework of the IRT 'Matériaux M2P' project.

• Phytomining appears to be a promising sustainable solution for low-concentration ore beneficiation.

Field experiments will be conducted at a dedicated large-scale experimental station (GISFI). This will enable the study of transfer and transformation processes in the soil-plant system at a relevant scale, thereby furthering our understanding of the cycles of critical metals.

The Steval platform fore ore processing (flotation column)

Recovery of uranium low-grade uranium ores by froth flotation: a study of the texture and synergetic effects of flotation reagents

(Co-financing of experiments carried out within the framework of a PhD project on Trekopje ores (PhD financed by Areva) Lev Filippov, Inna Filippova and A. Duverger

Ore deposits, especially those hosting low-grade ores, are becoming increasingly important areas of concern and specific research aimed at adapting beneficiation procedures is therefore justified. In the case of low-grade U-ores, conventional treatment does not typically involve mineral processing, such as the use of concentration methods, to reduce the consumption of leaching reagents. The aim of this study is therefore to develop an upgrading process to improve the operating process (alkaline heap leaching), that takes into account the mineralogical and textural variability of the ore.The Trekkopje deposit is composed of calcrete and gypcrete, and the U-bearing mineral is carnotite ($K_2 (UO_2)_2 [VO_4] 3H_2O$) *(Fig. 2.1)*. The gangue minerals are silicates such as quartz, feldspar and mica, and Ca-minerals such as calcite and gypsum (XRD and ICP-MS analysis). SEM image-processing was used to determine the textural properties and proportion of free exposed surface of mineral inclusions in clay clusters. In calcrete milled to < 200 µm, 50 % of the carnotite is found in clay clusters. These are composed of 98 % palygorskite and 2 % illite, montmorillonite, and interbedded clays (XRD and microprobe analysis). Approximately 95% of the carnotite is < 70 µm in size. Calcite is the main inclusion observed in the clay clusters (the average occurrence of calcite and carnotite inclusions is 12 % and 5 %, respectively). Moreover, the percentages of exposed surface of calcite and carnotite in clay clusters are low (3 % and 6 %, respectively). The inclusions should not therefore affect the behaviour of mixed clay particles, but we were unable to verify this using ore flotation. Three stages of mineral separation were proposed based on the abilities of the different minerals to consume leaching reagents: separation of Ca-minerals from silicates; separation of palygorskite from gangue minerals; and separation of carnotite from gangue minerals. A study of the electrokinetic properties (electrophoresis) of silicates and Ca-minerals was conducted in order to select the collectors and optimum pH range for selective flotation. Basic pH near neutral proved optimal for the separation of gangue minerals with cationic or anionic collectors (silicates IEP - pH 1-2, palygorskite IEP - pH 3, francolite IEP - pH 3-4 and IEP minerals calcium - pH 9 - 10).

Adsorption isotherms of the primary amines with a nonionic reagent, obtained by liquid chromatography, illustrate co-adsorption of the amines on the surfaces of silicates at pH 8. The presence of a nonionic reagent leads to the formation of a compact layer on the mineral surface derived from displacement of the symmetric and asymmetric vibration groups CH2 and CH3 of the infrared diffuse reflectance spectra.

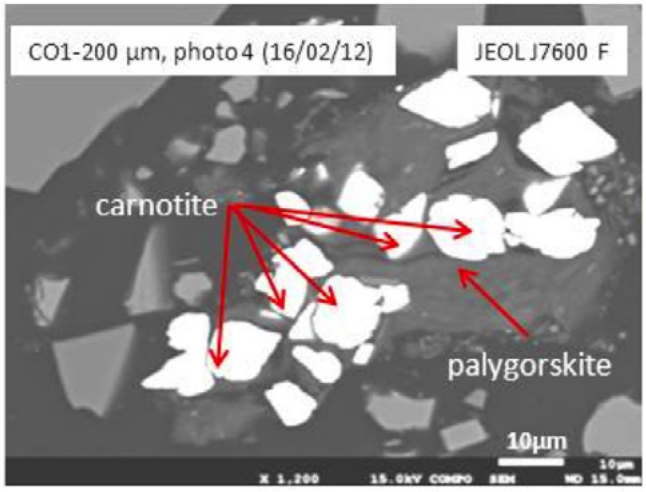

Fig. 2.1: Carnonite associated to palygorskite in Trekkopje calcrete.

Palygorskite was separated from pure Ca- minerals and silicates at pH 8 with a mixture of a primary amine and a nonionic reagent as collectors, without use of a specific depressant. A clear separation of Ca-minerals and silicates was achieved at pH 8 by combining sodium oleate with aliphatic alcohol *(Fig. 2.2)*. The observed 2-fold to 10-fold reduction in ionic reagent consumption highlights the synergistic effects of ionic and nonionic reagents. The ore flotation tests confirmed results obtained in pure mineral flotation with anionic collectors. The removal of Ca-minerals (the float product containing 20 % of the uranium) from silicates and other U-associated minerals (the non-float product containing 80 % of the uranium) was achieved using a combination of sodium oleate and an aliphatic alcohol. Through coupling of multi-scale approaches, this study has yielded important results that can be exploited to resolve the problem of processing of low-grade ores.

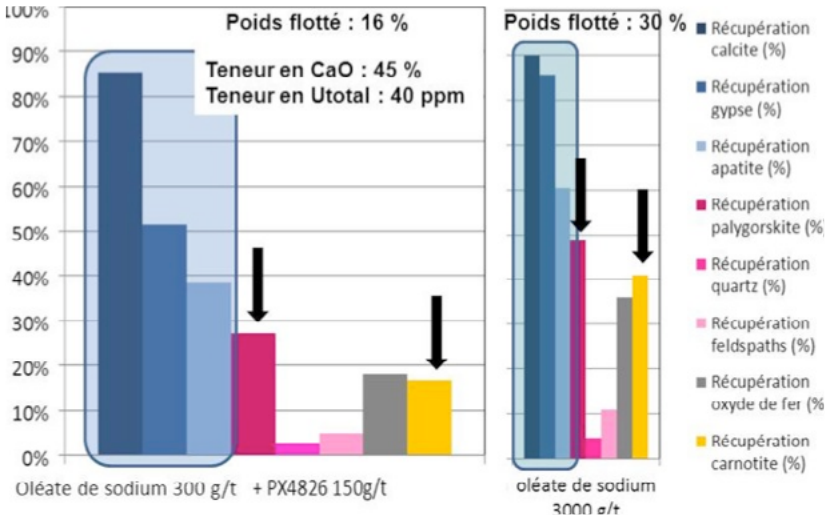

Fig. 2.2: Rate of recovery of the Trekkopje ore minerals using Na-oleate mixed with a non-ionic reagent (PX4826) at pH 8.

Mineralogy and textural properties of oceanic hydrothermal sulphides: development of a froth flotation process

(Inna Filippova, Lev Filippov)

Seafloor massive sulphide deposits (SMS), similar in composition and genesis to terrestrial volcanogenic massive sulphide deposits, are generally found in volcanic seafloor environments along major rifts and mid-ocean ridges. They are associated with extensional tectonic settings such as nascent-arc, rifted-arc and back-arc environments. SMS may represent an important source of base metals and be an alternative to onshore resources.

The size of deposits is highly variable, ranging from 0.5 to 100 Mt. The raw value of the ore varies from around 500 to 2000 $/t. A unique feature of hydrothermal sulphides is the presence of metals associated with major base metals (ex: Zn / Ge) and the flows of natural hydrogen. The operating technologies are already in development, however storage and ore processing methods still require in-depth study.

Seafloor massive sulphide deposits have particular mineralogical compositions and textural features that differentiate them from their terrestrial equivalents. When extracted from salt water and exposed to ambient air, massive sulphide samples oxidize strongly and are rapidly covered with thin layers of iron, copper and zinc-rich sulphates and oxides as well as elemental sulphur.

Mineralogical and textural studies were carried out in collaboration with Ifremer and Technip on samples from six marine campaigns (1998 to 2008). Marine sulphide ores are quite similar to continental sulphide ores (VMS, porphyries, skarns, MVT). Textural properties vary with the degree of maturity and the sample position within the hydrothermal deposit. The total porosity of this type of sample is also relatively high, particularly for immature samples such as black smoker chimneys, whose total porosity can reach 15%. Froth flotation, commonly applied to separate all types of sulphide from the gangue, is one possible technique that can be used for processing seafloor ore.

The sulphide recovery rate was in most cases excellent (>90%); however, separation of the various mineral phases was difficult, even when applying known methods used for terrestrial ore. High pH, which is known to be an effective way to depress pyrite and thus favour the flotation of copper and zinc sulphides, did not have the same effect on seafloor ore *(Fig. 2.3 a)*. Moreover, the flotation behaviour decreased rapidly with conservation time at the surface, even when samples had been stored in a confined environment. Thus, a cyclic behaviour in flotation of the same sample was shown in this work to be due to fast oxidation and removal of the oxidized macrolayer composed mainly of sulphate phases. The drastic increase in flotation recovery after two months' conservation in the N2 atmosphere can be explained by a significant proportion of elemental sulphur (up to 9 %) on the chalcopyrite surface, particularly for the finest size fraction. Further oxidation leads to formation of a thick hydrated surface layer, which requires a high dose of collector to reach a recovery level the same as that of particles with native sulphur on the surface *(Fig. 2.3 b)*.

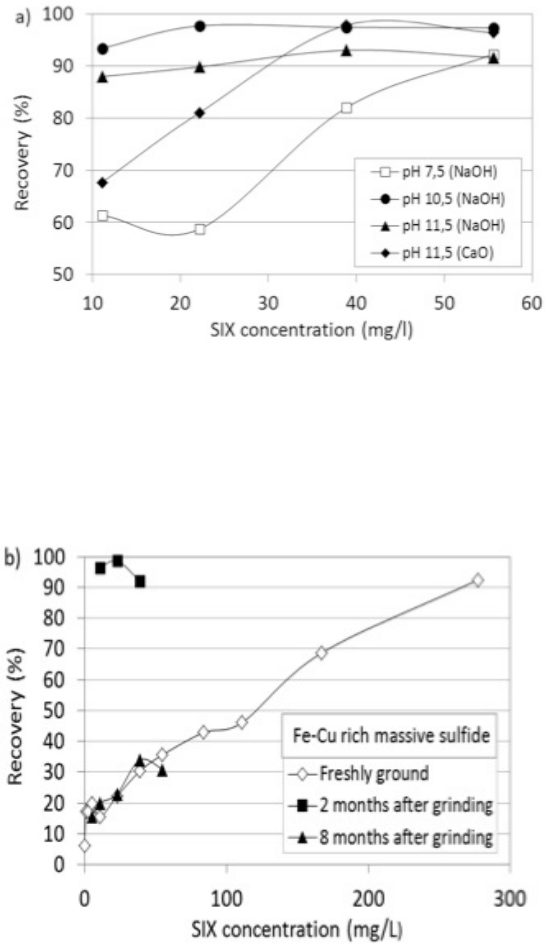

Fig. 2.3: (a) Flotation of a pyrite-rich sample (FLDR08.t2) according to the pH regulator (CaO and NaOH); (b) flotation of Fe-Cu-rich massive sulfide samples (freshly ground, 2 and 8 months after grinding, collector: sodium isopropyl xanthate; pH 6.5).

The results of this work programme concern all of the scientific sectors of phytomining, from geobotanical research of new species or taxon of hyperaccumulators, to agronomical and metallurgical processes for the recovery of metals. In 2014, the hydrometallurgy team led by Prof. M.O. Simonnot of the Laboratoire Réactions et Génie des Procédés ('Reactions and Process Engineering Laboratory'; LRGP) of the CNRS-Université de Lorraine joined the RESSOURCES21 LabEx. This team has been involved since the start of the LabEx in metal recovery from hyperaccumulator biomass. During the 2012-2014 period, a team of phytomining researchers from CRPG, LIEC, LRGP and LSE put together an ANR proposal on agromining. The project proposal was accepted in July 2014 and will start in January 2015. In October 2014, in the framework of the LabEx RESSOURCES21 Ni Project, Dr. Antony van der Ent was recruited to join the team and to reinforce the group which will implement phytomining as a rehabilitation project for Ni lateritic mines. He has extensive experience in SE Asian Ni-hyperaccumulator taxonomy and ecophysiology.

Results

Complete geobotanical inventory of Ni-hyperaccumulating plants in the balkans

Over the past decades, reports concerning the hyperaccumulating flora of the Balkans have been published either from herbarium specimens or from field-collected specimens. Most of these reports focused on the single accumulation of Ni. Other elements have progressively been taken into account when reporting on field-sampled hyperaccumulator plants. Only very recently was it discovered that Alyssum murale and other Brassicaceae had an extreme accumulation potential for K, although this element is deficient in serpentine soils. After conducting a number of surveys in Albania and continental Greece, we now have a good conception of hyperaccumulator diversity *(Fig. 2.4)*. We have even reported a new taxon, not previously recognised as a Ni-hyperaccumulator. Two families (Brassicaceae and Asteraceae) are present in the ultramafic areas of the two countries, as well as several genus of the Brassicaceae family (Alyssum bertolonii, A. heldreichii, A. markgrafii, A. murale, A. smolikanum, Noccaea goesingense, N. Ochroleuca, N. tymphaea, Bornmuellera baldaccii, B. tymphaea, Leptoplax emarginata). Two species of the Centaurea genus represent the Asteraceae hyperaccumulators. Continental Greece marks a transition between Turkey and the rest of the Balkan peninsula. We have reported the main characteristics of the fertility of the soils on which the samples were collected and have analysed the full composition of cations in the plant parts (stem, leaves and reproductive parts). Several locations for each taxon are reported whenever possible.

Results clearly indicate the different behaviour of the different species. Some species (e.g. Bornmuellera tymphaea and Leptoplax emarginata) are able to accumulate more Ni than others and this seems to be related to a lower uptake of Ca. Calcium and potassium are taken up in similar concentrations and these three elements are always higher than Mg. A reasonable hypothesis to explain the higher levels of Ni accumulation is probably a lower uptake of the other cations (i.e. Ca and K) and likely relies on either genetic or phenotypic differences. The identification of the potential of hyperaccumulator taxa for phytoextraction should be interpreted in a more complete way by analysing the cation absorption balance in whole plants. Soil analysis and the level of available Ni, Ca and K should also be taken into account in order to fully characterise the environmental conditions under which theplant has performed cation accumulation.

Fig. 2.4: Hyperaccumulator taxa from the Balkan Peninsula (Photos: G. Echevarria)

Improving the agronomy of Alyssum murale for extensive phytomining: seven years of field study (Collaboration with the Agricultural University of Tirana, Albania)

Large ultramafic areas exist in Albania that could be suitable for phytomining with native Alyssum murale. We undertook a five-year field experiment on an ultramafic Vertisol, aimed at optimizing a low-cost Ni-phytoextraction crop of A. murale, which is adapted to the Balkans. The effects of several parameters were studied on 18m^2 plots in natural conditions: (i) plant phenology and element distribution; (ii) plant nutrition and fertilization; (iii) plant cover and weed control; and (iv) planting technique (natural cover vs. sown crop). The optimal harvest time was set at the mid-flowering stage, when Ni concentration and biomass yields were highest. The application of N, P, and K fertilizers, and especially a split 100 kg ha^{-1} N application, increased the density of A. murale compared to all other species. It increased shoot yield significantly, without causing any reduction in Ni concentration. In natural stands, the control of graminaceous weeds required the use of an anti-monocots herbicide. However, after the optimisation of fertilization and harvest time, weed control procured little benefit. Finally, cropping sown A. murale was more effective than enhancing native stands and gave higher biomass and phytoextraction yields; biomass yields progressively improved from 0.3 to 9.0 t ha^{-1} and phytoextracted Ni increased from 1.7 to 105 kg ha^{-1}.

A field experiment with native Alyssum murale was undertaken on two representative Vertisols (Pojskë and Domosdovë, Albania) with the aim of testing the effect of plant density on phytomining cropping systems. Both areas were cleared in late summer 2012 and then ploughed. The soils were characterized and then 0.5 ha was planted with native seedlings at Domosdovë in September 2012, at a density of 6 plants per m^2. In a second 0.1-he plot at Domosdovë, plants that had germinated in spring 2012 were left to grow without any competition from other plants. Plots were weeded manually in autumn 2012 and in spring 2013 and individuals occupied approx.1 m^2 at maturity. Also in September 2012, a 0.3 ha plot at Pojskë was planted with seedlings of A. murale, at a density of 4 plants per m^2. Plants grown at a density of 4 and 6 plants per m^2 did not fully cover the ground; gaps were filled in naturally by a second spontaneous generation of A. murale seedlings which had germinated in autumn 2012. Other weeds were eliminated with herbicides. At Domosdovë, for 1 and 6 plants m^{-2} and at Pojskë for 4 plants m^{-2}, the biomass yield was 10.5 and 10 t ha-1, respectively, and the yields of phytoextracted Ni were 77, 41 and 112 kg ha^{-1}, respectively. We confirm that 4 plants per m^2 is the optimal plant density. Furthermore, plants responded differently in their native environment than they did in field trials in North America; A. murale can be a weed to itself and lower the Ni phytoextraction yield.

Ni extraction from biomass

Ni recovery from serpentine soils by phytomining has proved feasible. Phytomining involves the cropping of hyperaccumulating plants with high Ni contents and the valorisation of Ni by pyro- or hydrometallurgical processing. In order to evaluate the Ni contents of different plants, we analysed the organs of 14 hyperaccumulators from three genera: Alyssum, Leptoplax and Bornmuellera. The highest concentration was recorded in the leaves of Leptoplax (34.3 ± 0.7 mg g DM). In addition, we investigated biomass combustion, which is the first step in a process we designed to obtain a nickel salt. We showed that temperature and duration were important parameters for ensuring high quality ashes. At the bench scale, the optimum conditions were 550°C and 3 hours. In this way, we obtained ashes with Ni contents of up to 20 wt%. Biomass ashes can be considered a bio-ore for recovering metal value.

Optimisation of the ANSH (Ammonium nickel sulfate hexahydrate) production process enabled us to propose a number of significant improvements:

- Recycling pathways were proposed at the ash-washing step in order to save water.
- Acid leaching duration was reduced by a factor of two.
- Sodium hydroxide was replaced by calcium hydroxide, which decreases costs while avoiding the precipitation of Na_2SO_4. In addition, almost all excess H_2SO_4 was precipitated in the form of $CaSO_4 \cdot 2H_2O$, which led to the almost total removal of iron.
- Magnesium was removed prior to ANSH crystallisation instead of after. The volume of leachate was reduced by evaporation, which concentrated Ni meaning that only one step of ammonium sulphate addition was required for crystallisation.
- After the second crystallisation step, ANSH crystals were characterized by combined techniques and their purity reached 99.1 ± 0.2 %, a level higher than has previously been achieved *(Fig. 2.5)*.
- The mass balance performed on the entire process proved that ~91% of the Ni present in the ash had been recovered.

This subject was covered in the PhD thesis of Xin ZHANG, defended on 5th Dec. 2014 (co-directed by LSE and LRGP).

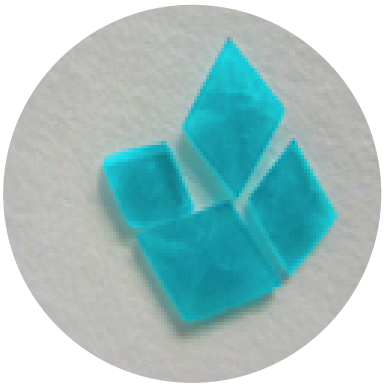

Fig. 2.5: Crystals of $Ni(NH_4)_2(SO_4)_2 \cdot 6H_20$ produced after optimization and purification (Photo X. Zhang)

Three PhD projects started in 2013:

- Tenghaobo DENG (LSE/CRPG – Sun-Yat Sen University, Guangzhou) on the study of Ni homeostasis in hyperaccumulator species from Europe with stable Ni isotope geochemistry.
- Ali KANSO (LSE/Liban University) on phytoextraction of metals in contaminated soils from Lorraine and phosphate-rich soils from Liban.
- Marie RUE, (LSE/LRGP) on the processes of metal extraction or fixation in biomass (Lorraine LORVER project, supported by AME-Région Lorraine; undertaken in collaboration with INRS-ETE (Québec, Canada), Université Agricole Tirana (Albania), and the TEIKAV Technological Institute of Kavala (Drama, Greece)).

One PhD Thesis (co-supervised by LSE, Centre for Mined Land Rehabilitation, University of Queensland) started in 2014:

- Philip NKRUMAH (LSE/University of Queensland) on the agronomy of tropical Ni-hyperaccumulators from South-East Asia.

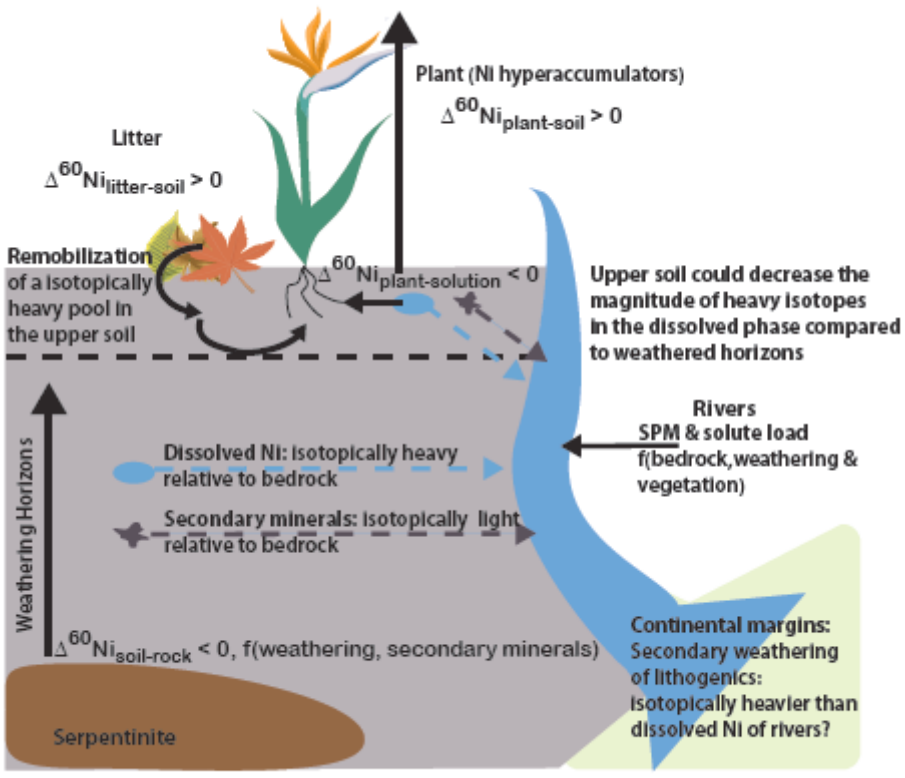

Fig.: Ni transfer and fractionation from soil to plant.

Nicolas Estrade post-doctoral fellowship at CRPG

After obtaining his PhD in isotope geochemistry in 2010 at the University of Pau in France (entitled "Isotope tracing of anthropogenic mercury sources in the environment"and focused on the use of stable mercury isotope compositions), Nicolas Estrade held a post-doctoral fellowship at the Université de Lorraine in the framework of the RESSOURCES21 LabEx.

The objective of the study was to use nickel stable isotope fractionation to understand the mechanisms that govern the transfer of Ni from soils to the hyper-accumulating plant system. Nickel stable isotopes are a promising non-traditional stable isotope system for understanding the Ni biogeochemical cycle, especially in contaminated or highly-enriched environmental compartments such as ultramafic contexts. Ni-hyperaccumulating plant species (e.g. Alyssum murale) that grow in ultramafic soils can concentrate up to several weight percent Ni in leaves and may be used in phytoremediation or phytomining. In addition to its insensitivity to redox processes, nickel homeostasis in hyperaccumulating plants is poorly understood. In this work, stable Ni isotopes were used to document the extent of isotopic fractionation during accumulation processes from soil to Ni-hyperaccumulating plant species. Sampling was carried out at four different sites in Albania and included collection of several hyperaccumulating plant species as well as tolerant ones. Using two-step chemistry to isolate Ni and the double-spiking technique to correct for instrumental mass fractionation, we recorded Ni concentrations and Ni isotope compositions along the continuum of ultramafic rock, soil (different horizons), litter, roots, stems, leaves and flowers. In typical Ni-rich soil, root and litter concentrations ranged from 1 to 3 g kg^{-1} dry matter. Nickel accumulation increased to 3-6 g kg^{-1} in stems and reached up to 20 g kg^{-1} in leaves. In contrast, the leaves of tolerant species presented Ni concentrations of ~0.1 g kg^{-1}.

The serpentinized peridotites samples presented similar isotopic compositions (0.25 ± 0.14‰), enriched in heavier isotopes compared to the Ni isotope composition of bulk silicate earth. At two locations, all horizons in two soil profiles with different degrees of weathering, presented light-isotope enrichment compared to the parent rocks ($\Delta^{60}Ni_{soil-rock}$ up to -0.63 ± 0.11‰, 2SE), suggesting that the soil pool takes up the light isotopes, while the heavier isotopes remain in the dissolved phase. By combining elemental and mineralogical analyses with the isotope compositions of the soils, the extent of fractionation was found to be controlled by the secondary minerals formed in the soil, and suggested that the higher the degree of weathering, the lower the fractionation between soil and rocks.

The degree of weathering also controls the isotopic composition of the pool available for plant uptake. DTPA-extractable fractions presented large enrichments in heavy isotopes compared to the soil ($\Delta_{60}Ni_{phyto-soil}$ up to 0.89±0.07‰, 2SE). The vegetation growing on ultramafic-derived soils is highly-specialised and includes Ni hyperaccumulating species, which can accumulate several percent per weight of Ni, and non-accumulating species. The non-hyperaccumulators (n = 2) were inclined to take up and translocate light Ni isotopes with a large degree of fractionation ($\Delta_{60}Ni_{leaves-roots}$ up to -0.60±0.07‰, 2SE). For the Ni-hyperaccumulators (n = 7), significant isotopic fractionation was measured in the plants in their early growth stages, while no fractionation occurred during later growth stages, when a plant is fully loaded with Ni. This suggests that (i) the high-efficiency translocation process involved in the hyperaccumulators does not fractionate Ni isotopes, and (ii) only the root uptake process fractionates Ni isotopes during the lifetime of the plant. In the ultramafic contexts, vegetation composed of hyperaccumulators can significantly influence the isotopic composition of the upper soil horizon, thereby affecting the isotopic balance of the Ni exported to rivers. This should be taken into account in future mass balance calculations.

Hydroponic cultivation was used to investigate the isotopic fractionation of nickel (Ni) and zinc (Zn) during plant uptake and translocation processes. The non-accumulator Thlaspi arvense, the Ni hyperaccumulator Alyssum murale, and the Ni/Zn hyperaccumulator Noccaea caerulescens, were grown in low (2 µM) and high (50 µM) Ni/Zn solutions. Plant roots preferentially absorbed light isotopes of both elements, presumably due to a low-affinity transport system. The isotopic fractionation of Ni ($\Delta^{60}Ni_{plant-solution}$ = -0.63 to -0.90‰) was greater than that of Zn ($\Delta^{66}Zn_{plant-solution}$ = -0.10 to -0.23‰) in hyperaccumulators grown in low-Zn treatments. Due to rapid absorption of Ni or Zn at the root surface, the occurrence of a concentration gradient from the root surface induced ion diffusion in the rhizosphere, which could result in a ~0.30‰ negative shift in plants for both metals. Zn reduced Ni uptake as well as its isotope fractionation ($\Delta^{60}Ni_{plant-solution}$ = -0.07 to -0.11‰ in high Zn treatment), indicating that Ni shares at least part of its uptake pathways. Ni isotopes could fractionate further during long-distance transport in the xylem due to absorption by stem symplast. Depending on the results of future studies, isotope composition analysis of transition elements could become a useful empirical tool for studying physiological processes in plants (See figure).

03

Transfer and dissemination of critical metals (CMs) in the ecosphere: mechanisms and ecotoxicological impacts

L. Giamberini,
C. Leyval

LIEC

Dissemination in the hydrosphere

Main objectives

In recent years, the production of high-tech goods has increased the global demand for raw materials to the point that securing reliable, sustainable, and undistorted access to these resources has become a primary issue within the European Union. Raw materials are fundamental to Europe's economy and growth and are essential for maintaining or improving our quality of life across a wide range of areas, including medicine, agriculture, and clean energy. The Raw Materials Initiative has defined which raw materials are critical to the EU on the basis of their economic importance and the high risk associated with their supply. In 2014, the list includes ten trace elements, the Platinum Group Metal (PGMs) and the Rare Earth Elements (REEs), which include the light (La-Eu) and heavy (Gd-Lu) lanthanides. In response to the increase in demand, the extraction, use, and disposal of REEs are all expected to rise in the near future, raising concerns about the potential impact of these elements on the environment.

Disruption of the biogeochemical cycles of some REEs is already apparent in aquatic and terrestrial ecosystems (for example, gadolinium anomalies in freshwater and tap-water and REE enrichment in soils as a consequence of agricultural practices). However, information about the potential hazards posed by REE remains incomplete and sometimes unreliable, preventing any clear assessment of the risks. Low concentrations of REEs are common in soil and water but the low mobility of these elements could promote their accumulation in the environment as a consequence of anthropogenic inputs. In the framework of investigating perturbations in the biogeochemical cycles of CMs, this work programme aims to study and better understand the occurrence, fate and behaviour of CMs in natural systems and determine the factors that are responsible for their effects on living organisms.

I. OCCURRENCE, FATE AND ECODYNAMIC OF STRATEGIC METALS IN AQUATIC ECOSYSTEMS

1. Gadolinium is a strategic metal belonging to the REE group. Gadolinium-based contrast agents (Gd-CA), which are widely employed in Magnetic Resonance Imaging (MRI), are produced by chelation of Gd to an organic chemical part. Gd-CA are injected into a patient's blood and are then eliminated from the body by urinary excretion over the course of several weeks. At present, there are no specific recycling procedures for these pharmaceuticals, and Gd-CA are found in waste water treatment plants and throughout the aquatic environment from river water to tap-water. In two recent German studies (Telgman et al. (2012) and Birka et al. (2013)), 80% of the total Gd measured in Berlin river-water was found to be in the form of Gd-CA, in particular the well-known macrocyclic molecules used in MRI.

The PhD research of Emilie Perrat (Dir. C.Cossu-Leguille & M. Parant) focuses on studying the impact of Gd-CA on living organisms under real conditions. The first phase of the research involved measuring the presence of total Gd, both natural and anthropogenic in origin, in 23 freshwater sites located across the Lorraine region.

Field sampling was conducted in February and July 2014 (during high and low water levels, respectively). Gd concentrations were found to be higher than 100ng/L in more than half of the samples. Four different geographical areas were defined on the basis of the data *(Fig. 3.1)*:

- A reference site with no anthropogenic Gd, located in the south-east of the Vosges mountains (department 88);
- Low contamination sites: ~33ng/L anthropogenic Gd, located in the south-east of Lorraine;
- Moderately contaminated sites: ~52ng/L anthropogenic Gd, located in the west of Lorraine;
- Highly contaminated sites: ~85ng/L anthropogenic Gd, located in the north-east of Lorraine, and including the main cities (Metz and Nancy), where total Gd ranges from 10 ng/L to more than 10 µg/L.

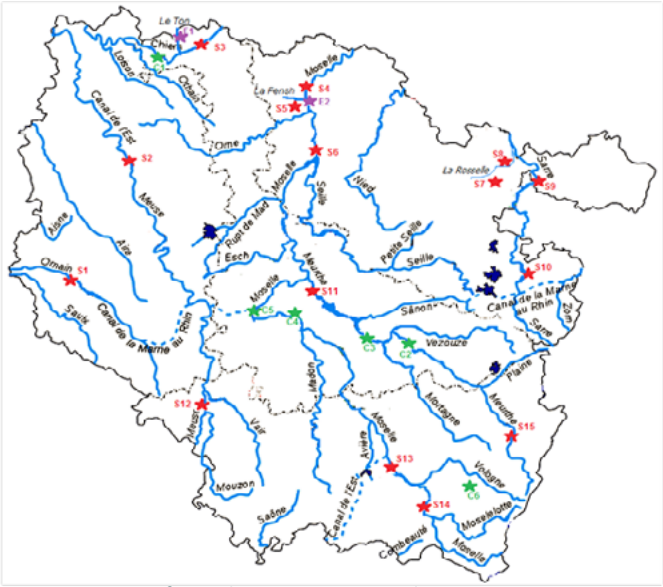

Fig. 3.1: Map of sampling sites stuying Gd concentrations in Lorraine rivers.

The first phase of the project therefore allowed us to define an environmentally realistic gradient of Gd concentrations that can be used for further ecotoxicity bioassays.

2. The use of Antimony (Sb) in the world is growing with the demand for its increasingly diverse industrial applications (e.g., microelectronics, lead-acid batteries, bullets, catalysts, and fire retardants). The rising demand for Sb, coupled with the fact that China is the world's main Sb supplier, has led to its classification as a strategic element. Environmental Sb concentrations resulting from mining activities often exceed authorized thresholds (Filella et al., 2009; Reimann et al., 2010). Sb redox states +3 and +5 are the most commonly found in the environment. Sb(V) is more mobile in neutral or alkaline soils (Johnson et al., 2005), whereas Sb(III) is able to adsorb on manganese or iron oxyhydroxides (Wilson et al., 2010).

The aim of the project of P. Bauda and G. Echevarria was to develop a combined methodology for the study of Sb environmental mobility in a mining context. In situ studies that addressed Sb mobility in the real environment were combined with in vitro experiments in order to confirm process identification under controlled and simplified conditions. In situ studies were conducted at two abandoned Sb mines (Goesdorf, Luxembourg, and La Bessade, Haute Loire, France) *(Fig. 3.2)*. At the Goesdorf site, Sb mobility was confirmed by analysis of soils, minewater and vegetation. Of the adapted plants sampled at the site, the ruderal species Senecio jacobaea was found to have accumulated the highest Sb concentrations, both in roots and aerial parts. At the La Bessade site, Sb and arsenic (As) concentration gradients were identified and natural As depollution was observed in the precipitation and/or adsorption on biogenic iron oxyhydroxides at the minewater outlet. Using the results of the in vitro studies, the initial goethite model was completed with a ferrihydrite model in which Sb was the carrier phase, and the Donnan Membrane Technique (DMT) was developed for analysis of free Sb oxyanions in solution.

3. The project developed by E. Rotureau and J.P. Pinheiro aims to explore the feasibility of using electroanalytical techniques for speciation studies of trivalent metals in natural waters. One of the project objectives is to analyse the dynamic behavior of indium, a surrogate trivalent cation, which will in turn allow us to study its speciation with organic matter such as humics and fulvics. The first results report measurements of indium speciation using an electroanalytical technique known as Scanned Stripping Chronopotentiometry (SSCP). SSCP is able to quantify both free and complexed metal ions. Because indium amalgamates in mercury (the working electrode), the SSCP technique can be directly applied to the study of indium speciation in solution. We have shown that the impact of the nature of the background electrolyte, ionic strength and pH can all be assessed by indium speciation. The results also demonstrated that a wide range of indium concentrations, from 10-10 M to 10-5 M, can be probed using the SSCP technique. This can be explained by the very high solubility of indium in the mercury electrode compared to other metals commonly analysed by SSCP (for example, Cd, Pb or Zn). Two other important differences were found concerning the electrochemical behavior of indium:

- The non-reversibility of the redox pair (In3+/(In(Hg)), despite the addition of a catalyst in solution;
- The presence of an electroactive species, other than the free indium ion.

Because of these differences, it is not possible to apply the analytical models usually used for interpretation of SSCP results and quantitative analysis of metal speciation. However, experimental studies were carried out to examine indium complexation in the presence of molecular and nanoparticle ligands (humic acids). For the reasons mentioned above and due to the paucity of published data regarding the thermodynamic constants for indium, interpretation of our results remains qualitative. Analysis of trivalent metals in solution will be improved by coupling the electroanalytical detection with another speciation technique which deals with a permeation device, known as the Donnan Membrane Technique (DMT), for robust quantification of free metals in solution.

Fig. 3.2: Minewater at La Bessade antimony/mine

1. In surface waters, chromium essentially occurs in two thermodynamically stable oxidation states: Cr(VI) and Cr(III). While hexavalent chromium is highly toxic to living organisms, trivalent chromium is considered relatively harmless. However, studies performed by different research groups (including our own) suggest that the ecotoxicity of Cr(III) for aquatic organisms may have been underestimated. The likely reason for possible errors in our current understanding of Cr(III) toxicity is that not enough attention has been paid to the complex chemistry of Cr(III) in ecotoxicological test media. In particular, following a series of rapid chemical reactions, the concentrations of Cr(III) added to test media may decrease rapidly over the duration of a test. Because of this, it is difficult to establish exposure/response relationships between Cr(III) concentrations and biological endpoints.

To circumvent these problems, the proponents of this proposal (D.A.L. Vignati and I. Aharchaou of LIEC, in cooperation with C. Fortin of INRS, Canada) have adopted a radiochemical approach to comparing the uptake and intracellular distribution of Cr(III) and Cr(VI) in the freshwater alga Chlamydomonas reinhardtii. Use of a highly sensitive radiochemical approach allows for shorter test durations (minutes to hours) and lower concentrations (nM range) compared to traditional approaches, which require days of exposure and concentrations in the µM range. This approach therefore minimises (or eliminates) problems linked to decreases in the Cr(III) concentration added to test media.
The experimental work showed that Cr(III) internalisation by C. reinhardtii follows a linear trend between 5 and 60 minutes, with comparable uptake rates apparent at whatever the pH level tested. To the best of our knowledge, our data are the first for algae and Cr(III). Data of this kind are necessary for the development of a BLM (Biotic Ligand Model) for predicting Cr(III) toxicity in freshwater. Uptake experiments with radiolabelled Cr(VI) are in progress. Regarding intracellular distribution, we observed that both of the redox forms preferentially accumulate in organelles, heat stable proteins, and in intracellular granules. Because Cr(VI) is reduced to Cr(III) upon entering a cell, these findings may indicate that Cr is handled in a similar way inside the cell regardless of the redox form in the external medium.

2. Toxicological studies of nanoparticles (NPs) have increased remarkably in recent years but our understanding of the hazards and risks they may pose remains limited because of the multiple parametric features that must be taken into account. These characteristics are related to the specific surface properties of NPs, which differ from those of the bulk material, and include particle size, shape, and surface charge, and their reductive or oxidative solubilities. In the environment, NPs may undergo changes that lead to different degrees of bioavailability and toxicity to living organisms. Research into the bioavailability, uptake, cellular accumulation and detoxication of NPs is therefore essential for understanding the ecotoxic mechanisms and the associated risks to organisms, as well as the functional role they assume in the ecosystems. In this context, our studies of the tissular and cellular localization of NPs (Leader L. Giamberini) supplement ecotoxicological investigations of different model species conducted as part of two ANR programmes.

Cerium oxides, which have long been employed in the glass industry, have undergone an exponential increase in use in their nanometric form ($nCeO_2$) in the automobile and cosmetics industries. Using RX microfluorescence and microtomography, we produced a 3D reconstruction of the digestive glands of zebra mussels exposed to $nCeO_2$ and the localisation of accumulated metals in the tissues. Internalisation and $nCeO_2$ speciation were confirmed by synchrotron beams (Collaboration A. Auffan, Cerege, Aix Marseille) and revealed a large reduction of Ce^{IV} to Ce^{III} that may be due to redox interactions between $nCeO_2$ and biological compounds *(Fig. 3.3)*. The Ce bioaccumulation was associated with biological effects linked to antioxidative and antitoxic defence mechanisms and immunological processes, as described below.

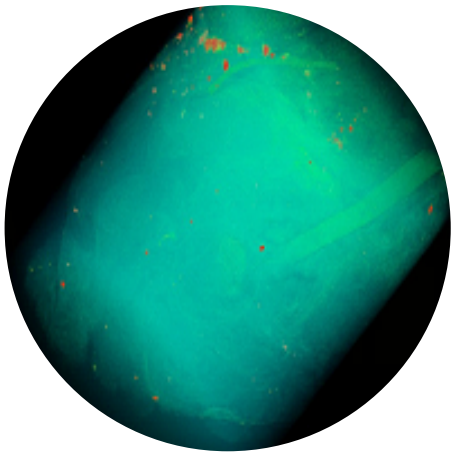

Fig. 3.3: 3D - reconstruction of digestive glands in zebra mussels exposed to CeO2 nanoparticles.

1. Lanthanides are a chemically uniform group of metals (La–Lu) that, together with yttrium(Y) and scandium(Sc), form the REE group. Because of their many applications (e.g., in agriculture, medicine, and the motor industry), global production of lanthanides has increased exponentially over the last few decades, causing disruption to their biogeochemical cycles. However, the ecotoxicological effects and mechanisms of action of these elements are still poorly understood. In particular, there is no consensus as to whether lanthanides show a coherent and predictable pattern of (eco)toxicity that relates to their atomic properties. For aquatic organisms, contradictory conclusions are found in the literature. The review recently published as part of the postdoctoral work conducted by V. Gonzales and supported by Labex R21 (Gonzales et al , 2014), showed that the variable composition of culture media used in ecotoxicology and the associated differences in lanthanide speciation are the most likely reasons for these discrepancies. In particular, the formation of insoluble species in some highly complexing media likely leads to changes in the soluble concentration of lanthanide during some tests, creating the potential for a generalised underestimation of their toxicity at the present state of knowledge. For terrestrial organisms, studies that can be used to establish trends in lanthanide toxicity are practically nonexistent, with most research instead focusing on the effects of REE mixtures.

In this framework, the bioassays conducted on different taxonomic groups (Gonzales et al., 2015) – decomposers (microorganisms), primary producers (alga), primary consumers (daphnia, ostracod and rotifer) and secondary consumers (Hydra) – revealed that ecotoxicity of lanthanides seems to increase with increasing atomic number (Ce<Gd<Lu), but a degree of variability among species was detected. The higher toxicity, at higher atomic number, was only significant in bacteria (V. fisheri) and alga (P. subcapitata). The crustaceans (D. magna and H. incongruens) were the least sensitive species while rotifer (B. calicyflorus) and cnidarians (H. attenuata) were the most sensitive, with no differences between elements observed at any of the concentrations tested. Although, at present and based on the ecotoxicological results obtained in this study, the environmental risk of these elements can be considered acceptable, some hotspots were nevertheless detected in Lorraine surface water. Their growing industrial use will promote the release of lanthanides into environment, and the associated risk could be higher in the future.

2. In the framework of the ANR Mesonnet program (PhD M. Garaud, Dir L.Giamberini & V. Felten), the short-term effects of nanometric cerium ($nCeO_2$) were investigated in environmentally realistic concentrations on two freshwater invertebrates: the amphipod G. roeseli and the bivalve D. polymorpha (Garaud et al, 2014). An integrated multibiomarker approach was adopted. Differences in the behaviour of the organisms and $nCeO_2$ in the water column led to different bioaccumulations: G. roeseli accumulated more cerium than D. polymorpha. Exposure to $nCeO_2$ led to decreases in the lysosomal system, catalase activity and lipoperoxidation in mussel digestive glands that might be the result of $nCeO_2$ antioxidant properties. However, the exposure also had a negative impact on haemolymph ion concentrations. At the same time, no strong adverse effects of $nCeO_2$ were observed in G. roeseli.

Fig. 3.4 : Examples of studied model species : Daphnia sp. and Gammarus sp.

The effects of chronic exposure to citrate-coated $nCeO_2$ (Ci-CeO2) and bare $nCeO_2$ (Ba-CeO_2) were assessed in zebra mussel under mesoscosm conditions. Bioaccumulation was weak, suggesting significant immobilization in pseudofaeces. Ci-CeO_2 bioaccumulated three times more than Ba-CeO_2, a result which is probably related to the coating and to the different behaviour of the particles in the water column and in the organisms. Integrated and discriminant analysis of biological responses showed that mussels exposed to both forms of $nCeO_2$ differed from the control and from each other due to metallothionein, antioxidative and antitoxic enzyme activity.

3. The PhD project of J. Andreï (Supervisors F. Guérold & S. Pain-Devin) forms part of the ANR Mesonnet program and is supported by Labex R21. The objective is to investigate the effects of silver nanoparticles (AgNP) in the freshwater crustacean Gammarus sp *(Fig. 3.4)*. The first phase of the work involved designing a valid experimental protocol for studying exposure to very low concentrations of AgNP (0.5 and 5 µg/L) in microcosms *(Fig. 3.5)*. Mechanisms of nanoparticle contamination (aggregation, dissolution and adsorption) in the microcosms were also studied. An experimental protocol for investigating short-term exposure to different sizes of AgNP was then designed in order to assess direct and indirect biological effects in three gammarid species. Responses were investigated at the cellular (defense, metabolism), behavioural (ventilation, locomotion) and functional (consumption and production of fine particles of organic matter) levels. The results showed that gammarids reduced their locomotion activity when exposed. The quantity of fine particles of organic matter produced was also reduced at very low levels of exposure (0.5µg/L) to the smallest AgNP (10nm). As these functional endpoints are involved in both the maintenance of an organism's metabolism and in the recycling of organic matter, it might be suggested that nanoparticle impact in gammarids could reflect potential further effects in the ecosystem functioning. However, the occurrence of such effects seems to depend not only on nanoparticle size, but also on the gammarid species. Long-term exposure experiments in mesocosm were designed in order to assess the effect of two different types of AgNP under more realistic environmental conditions. These experiments simulated chronic exposure of a multispecies system including gammarids, bivalves and microorganisms. No mortality was recorded, but some effects on the ventilation and locomotion of gammarids were observed, supporting our previous findings. Together, these results emphasise the need to better understand the biological effects of nanoparticles at low concentrations and in more complex systems in order to get closer to environmental realism.

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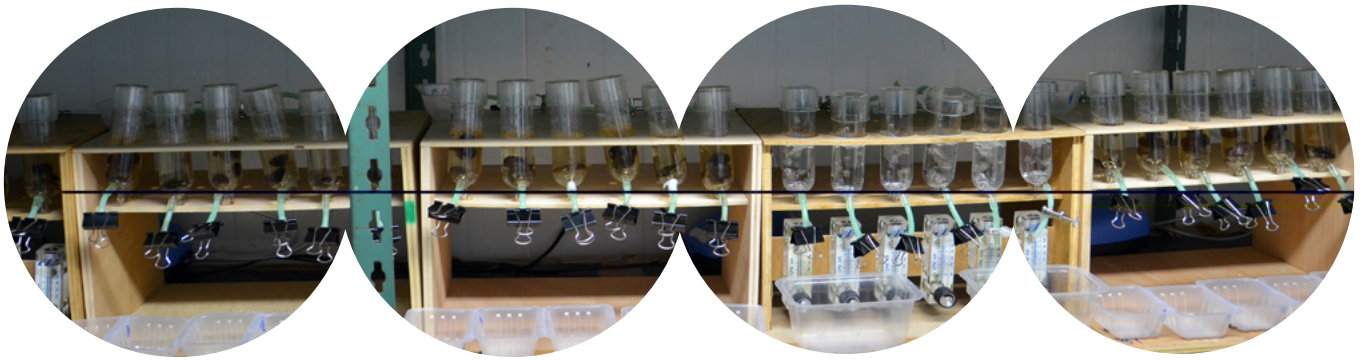


Fig. 3.5: Experimental design used to assess the effects of silver nanoparticles on the fine particulate organic matter production in Grammarus sp.

IV. WHOLE-CELL BIOSENSORS FOR UNDERSTANDING TRACE METAL (Sb, Ni) BIOAVAILABILITY IN MINING ENVIRONMENTS

The development and application of techniques to evaluate the availability of metals has received extensive scientific and legislative attention. Although metals occur naturally (weathering, volcanic eruptions), industrial production in the twentieth century (mineral extraction, manufacturing industry and waste disposal) has resulted in the input and accumulation of large quantities of metals in the environment. For instance, antimony (Sb) is the ninth most mined element in the world (over 2 million tons in world reserves; U.S. geological survey, 2010) and is used in a variety of industrial products (flame retardants, semiconductors, tracer bullets). As a consequence, the mining industry introduces to huge amounts of Sb waste in the environment. Therefore, the current policy challenge facing countries is to devise ways to insure that the supply and use of metals, such as antimony, for industrial and economical growth, is done in an environmentally friendly and sustainable manner, taking into account their impact on pollution. One of the main reasons behind such intense research is the possible linkage of metal exploitation to the evolution of biogeochemical cycles, the identification of causal factors behind toxicity, and the evaluation of environmental responses to anthropogenic pollution. Iron oxides, which are ubiquitous in the environment, and contribute up to 50% of the bulk mass of soils, have emerged as one of the major sinks for metals in terrestrial and aquatic environments due to their large surface area and reactive surface properties. Therefore, they play an important role in regulating their availability. However, while the mechanism of metal enrichment (sorption, co-precipitation) on iron oxides is reasonably well understood, the effects of bacterial activity on the mechanism of their remobilization is in its infancy, thereby limiting our ability to accurately reconstruct the evolving chemistry of the environment, in term of metal behaviour.

The two key parameters for understanding trace metal bioavailability are the characterization of the behaviour of metal-substituted iron oxides under various potential biotic conditions, and the secondary phases formed during subsequent transformation. Within this context, dissimilatory iron reduction can potentially play a central role as a biotic activity under anaerobic conditions. Thus, this research project examined the biogenic transformation of antimony (Sb) substituted iron oxide to provide a mechanistic understanding of the remobilization pathways and associated Sb bioavailability. Traditionally, the risk assessment (toxicity) for a given metal is based on measurement of its total concentration in an environmental sample (e.g., soil, sediment, water etc.). However, as the toxicity of the metal is closely linked to its uptake by biota, the determination of the total amount may differ significantly from the quantity that is bioavailable due to homeostatic regulation. The bioavailability concept is a very significant one in ecotoxicology, as it refers not to the total compound or dissolved concentration in the system obtained by chemical extraction, but rather to that fraction which is perceived by the biota. Indeed, according to Semple et al., (2007) bioavailability is defined, as “that which is freely available to cross an organism membrane from the medium the microorganism inhabits at a given point in time”. As such, the bioavailable fraction of a given metal is conceptually more easily determined by the biota itself, and one needs to interpret its biochemical response in order to establish the metal’s threshold concentration triggering a biological reaction. The outcome of this research will enhance our understanding of the fate of antimony during microbial-driven iron redox cycling in the environment. The research project has focused on two themes linked by this common objective:

• Transformation of Sb-substituted iron oxide (goethite) by bacterial activity (anaerobic iron respiration) to evaluate remobilization efficiency and mineralogical differences between secondary minerals formed via different pathways.

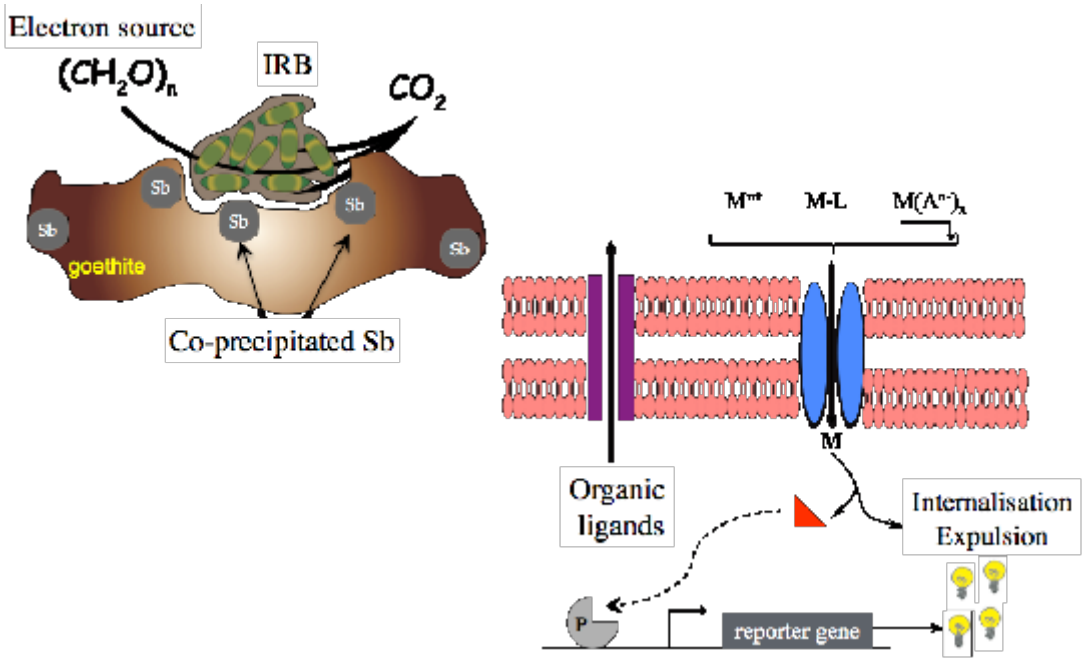


Fig. 3.5: Schematic representation of the experimental approach. An iron reducing bacteria will couple the reduction Sb-substituted goethite with the oxidation of formate. During the course of the reaction, the mobilisation in solution of Fe(II) and Sb(V) will be quantified. Additionally the bioavailability of the Sb(V) present the in aqueous phase will be estimated by recording the fluorescent signal (GFP production) emitted by whole cell biosensor.

• Characterization of Sb bioavailability via biosensors, during the transformation of Sb-substituted iron oxide by iron reducing pathways.

Main results

(i) The experimental approaches for iron oxide synthesis led to different percentages of substitution. Briefly, the first pathway was a modified method of that described for Al-goethite (Schwertmann and Cornell, 2000). The experimental method involved the mixing of a solution of FeCl3 and KSb(OH)6 with KOH to ensure alkaline conditions for precipitation of ferrihydrite-Sb and aging of the latter at 70°C to induce Sb-goethite precipitation. The second method involved the oxidation of a sulphated green rust formed in the presence of an antimonate solution (KSb(OH)6) to form Sb-goethite.

(ii) The model bacteria used in this study was an aerobic anaerobic facultative, non fermentative, obligate reparatory Fe(III) reducing bacteria: S. putrefaciens MR-1. Fe(III) iron-respiring bacteria (IRB) coupled the oxidation of formate to the reduction of ferric iron. Briefly, the cells were incubated in anaerobic conditions for two weeks with formate as the electron source and Sb-substitued goethite as the electron acceptor. Fe(II) production and Sb mobilization (Sbaq) was measured over the length of the incubation period. The results indicated that the percentage of substitution affected neither the extent of the bio-reduction nor the dissolved amount of antimonate.

(iii) The bioavailability of the amount of Sb(V) liberated during the course of the bio-reduction was assessed indirectly using E. coli JW 3434 / pars::gfp engineered by fusing ars operon (arsenic resistance gene) with a reporter (green fluorescent protein, GFP). The primary results indicated that the biosensor was able to detect Sb(OH)6 concentrations above in 3 µM. This indicates that the use of such biosensors could be pertinent to evaluate the bioavailability of Sb leached during the Sb substituted goethite bio-reduction *(Fig. 3.5)*.

Conclusions and future work

The experimental investigation used in this study allowed us, to a certain extent, to evaluate the biogeochemical cycle of iron and antimony through the use of iron-respiring bacteria and Sb substituted iron oxide. On the one hand, a given amount of Sb(V) can be removed from the aqueous medium through the co-precipitation of the metal with an iron oxide. On the other hand, bacterial activity could trigger dissolution of the mineral, leading to the release of Sb(V) in the medium. The bioavailability of the leached Sb(V) can be estimated using whole-cell biosensors. The diversity in terms of metal, iron oxide and bacterial activity, needs to be explored in order to gain an understanding of the main parameters governing their biogeochemical functioning and, subsequently, to estimate the bioavailability of a given metal. To this end, the same approach will be applied to the study of nickel (Ni) as the target metal trapped in ferrihydrite particles and nickel-sensitive whole-cell sensors (bacteria). The results obtained from studies of diverse metal-iron oxide-bacteria systems will, in the long run, enable an understanding of how these systems function and their impact on the biogeochemical cycles and bioavailability of metals.

River water sampling downstream a Waster Water Treatment Plant measuring Gd concentrations

04

International research visibility

I. SCIENTIFIC MEETINGS

LabEx RESSOURCES21 researchers are deeply engaged in international scientific meetings through their involvement as scientific session conveners and invited speakers. In relation to the ongoing RESSOURCES21 projects, international scientific meetings on Nickel and REE have been scheduled for 2017 and 2018, respectively.

Nancy will host the next SGA biennial international meeting, which will gather together more than 600 hundred researchers and students from more than 80 countries.

SGA 2015 will take place in Nancy from the 24th to the 27th of August 2015. The local scientific committee has made sure of the representation of LabEx RESSOURCES21 themes in the proposed sessions. (http://sga2015.blog.univ-lorraine.fr/)

II. INTERNATIONAL EXCHANGE

RESSOURCES21 has hosted ten foreign researchers in Nancy for stays of a few weeks to three months:

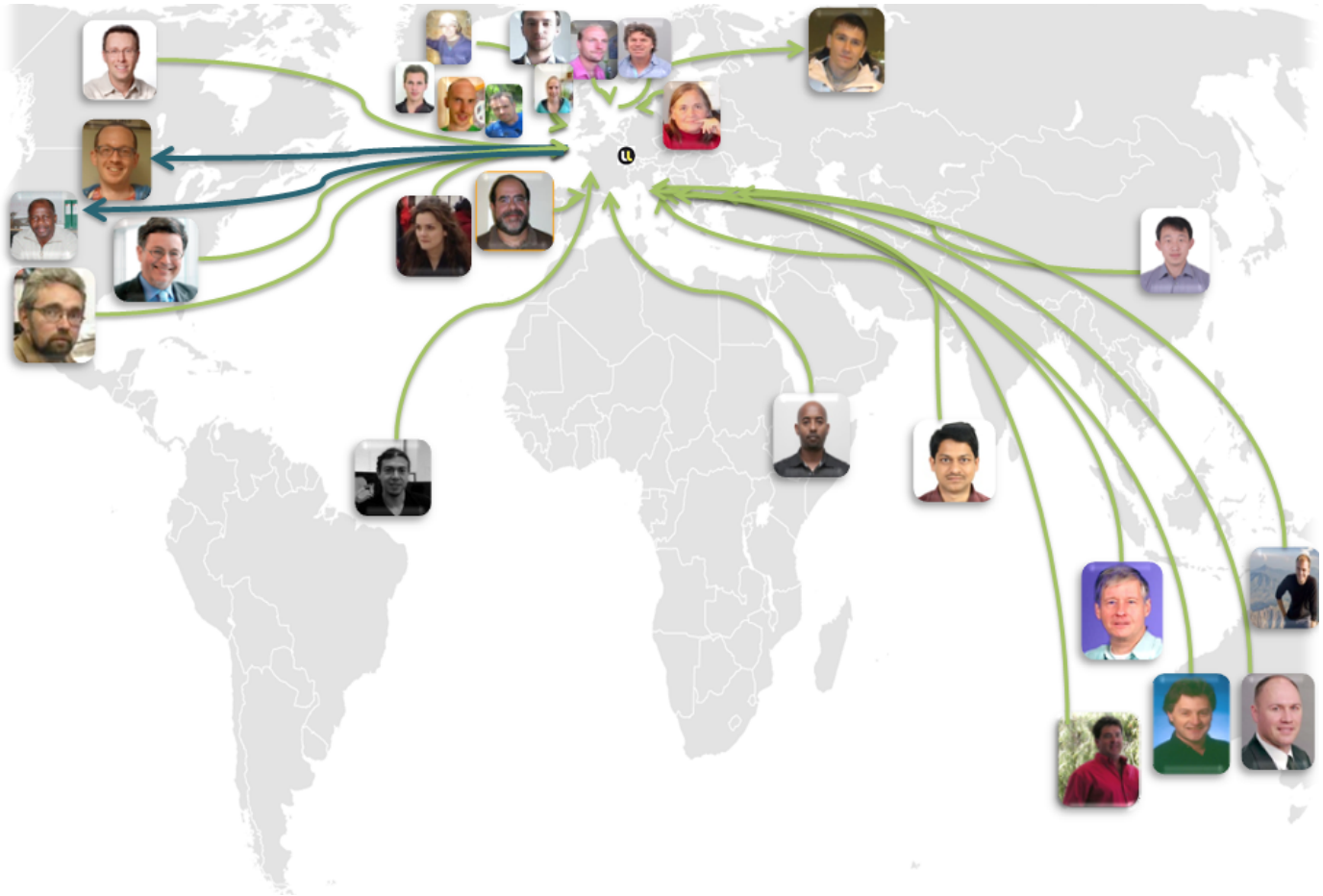
- Pr Igor Sevostianov of the Department of Mechanical and Aerospace Engineering, New Mexico State University, USA – 2 months, Spring 2012;
- Pr Li Guanglai of the College of Earth Sciences, East China Institute of Technology, Nanchang, China – 2 months, Autumn 2012;
- Dr Michel Jebrak of Quebec University, Montreal, Canada – 3 months, Winter 2013;
- Pr Campbell McCuaig, Director of CET, University of Western Australia – 2014;
- Dr Claude Fortin of INRS, Quebec, Canada – 2 months, Spring 2013;
- Pr José Paulo Pinheiro of Algarve University, Portugal – 3 months, Spring 2014;
- Pr Daniel Fornasiero, Associate Professor at the Ian Wark Research Center, University of South Australia – 2 months, 2014;
- Pr Laurent Aillères of Monash University, Australia – 3 weeks, 2014;
- Pr Monserrat Filella of the University of Geneva – 6 months, Winter & Spring 2014;
- Dr Alan Baker of the Faculty of Science School of Botany, Melbourne, Australia – 2 months, Spring & Autumn 2014.

RESSOURCES21 has also provided financial support (full and partial) for long stays abroad for researchers from Nancy:

- Yann Gunzburger, GeoRessources – Mac Gill, Montréal, Canada – 3 months 2013;
- Laure Giamberini, LIEC – CEREGE, Aix-en-Provence, France – 2014 & 2015;
- Mukendi Kongolo, GeoRessources – UQAT, Rouyn-Noranda, Canada – 3 years;
- Imad Aharchaoui – University of Laval, Quebec, Canada;

New and official links via formal agreements have been established with CET (field exploration) and the Quebec Resources Ministry. A network of laboratories working in the field of phytomining has been established and includes Spanish (CSIC), Austrian (BOKU), Albanian (UAT), and Greek (TEIKAV) partners. The network is also open to other strategic partners at the global level: USDA (USA), U. Sun Yat-Sen (China), EMBRAPA-Cerrados (Brazil), and the universities of Melbourne and Brisbane (Australia).

Recruitment at a glance



LabEx RESSOURCES21 researchers have endeavoured to (i) continue their work on the international scientific programmes in which they have been involved (FP7 PROMINE, which ended in December 2013, will be followed up with the BIOMORE project from January 2015; WAXI-AMIRA: Stage 3 launched in September 2014), and (ii) be more proactive, both in their applications to new international programmes and in developing international collaborations. This activity has led to the award of funding for several international programmes, as well as interesting new scientific collaborations.

Awarded European research programmes and networks

STOICISM

- Awarded in June 2012 - European Seventh Framework Programme for Research (FP7);
- LabEx RESSOURCES21 driving force: Lev Filippov;
- http://www.stoicism.eu/system/files/documents/STOICISM_Flyer-v3-print-17112014.pdf

In mid-2011, the European Seventh Framework Programme for Research (FP7) launched its bids for large projects under the umbrella of "Nanosciences, Nanotechnologies, Materials and New Production Technologies" (NMP), which included "NMP.2012. 4.1-1: New environmentally friendly approaches to mineral processing". The "Sustainable Technologies for Calcined Industrial Minerals" (STOICISM) project passed through two highly competitive rounds of bidding (187 proposals in total) before being given the green light for funding in June 2012. The project, which will run for over 4 years, kicked off in January 2013 in Spain. The STOICISM Consortium is led by a major industrial mineral producing company (IMERYS) and consists of 17 partners from 8 European countries. Key contributors to this multidisciplinary platform include several universities, specialised SMEs and corporations, an industry association, as well as applied technology and research institutes. The platform presentation given by Quentin Dehaine (Université de Lorraine, France) as part of the STOICISM FP7 project, entitled: "Critical metals (La-Ce-Nd-Nb, W) and Sn as co-products from the production of Kaolin in Cornwall, UK: Identification and characterisation of the most valuable fractions", received the SIM 2014 award in the PhD student category.

BioMOre

- Awarded in September 2014 - European project H2020;
- LabEx RESSOURCES21 driving forces: Lev Filippov and Jean-Jacques Royer;
- http://ec.europa.eu/eip/raw-materials/en/content/biomore-alternative-mining-concept-raw-materials-commitment.

There is considerable mineral resource potential at depth (>1km) in Europe, as demonstrated by current research (FP7 – ProMine), however, new methods are required for recovering these resources in an economical and environmentally-acceptable manner. BioMOre aims to develop new technological concepts for in situ recovery of metals from deep deposits using controlled stimulation of pre-existing fractures in combination with in-situ bioleaching. BioMOre will be launched in January 2015.

FAME - Flexible And Mobile Economic processing technologies

- Awarded in September 2014 - European project H2020;
- LabEx RESSOURCES21 driving forces: Lev Filippov.

The FAME project is devoted to improving techniques for the processing

and beneficiation of ores associated with primary rare-metal resources in Europe (skarns, greisens and pegmatites). FAME will consider flexible and modular processing technology demonstrated in relevant operational environments (industrially relevant environments in the case of key enabling technologies (TRL)). TRL6 is regarded as a feasible option for the processing of pegmatites, whereas TRL5 is considered more realistic for other types of ore body. The consortium has sixteen partners from seven European countries, including industrial, academic, and governmental institutions. The consortium has a strong industrial background and will conduct work on strategically important reference deposits operated by and/or accessible to the project partners and their associate partners within the EU28 nations and Greenland.

COST

- Awarded in November 2014 - European Cooperation in Sciences and Technologies (COST);
- LabEx RESSOURCES21 driving force: Laure Giamberini;
- www.cost.eu/COST_Actions/TDP/Actions/TD1407.

Network on Technology-Critical Elements – from Environmental Processes to Human Health Threats

The COST action will create a network of scientists working on and interested in technology-critical elements (TCEs),encompassing all aspects of the subject, from environmental issues to threats to human health. Through

assessment of the current state of knowledge and identification of gaps in our understanding, COST aims to establish priority research directions and act as a platform for new collaborations and joint research projects.

KIC Raw MatTERS

- Awarded in December 2014 - European Institute of Technologies (EIT) - Knowledge and Innovation Community;
- LabEx RESSOURCES21 driving forces: Frederic Vilieras and Laurie Wolff:
- http://eit.europa.eu/newsroom/eit-selects-new-strategic-partnerships-milestone-europe-areas-health-and-raw-materials).

Between 2012 and 2014, RESSOURCES21 was actively involved in developing the pan-European KIC Raw MatTERS programme, of which the Université de Lorraine is a partner institute. In December 2014, the project was selected by the European Institute of Technologies (EIT). As a result, the University will benefit from European financial support for a period of at least seven years to strengthen innovation across the whole raw materials value chain (exploration, extraction, processing, recycling and substitution). KIC Raw MatTERS, which was the only programme in this theme selected by the EIT, gathers together around 100 partners from academic, industrial and applied research institutions from 20 member states. KIC Raw MatTERS will strengthen the synergies between stakeholders in higher education, research, and development, with a view to promoting value creation through the support of innovation, the creation of start-ups, and the development of specialised training linked to the new scientific and technological practices in this domain.

NewOres Eramin project

- Awarded in December 2014 - ERA-MIN joint calll;
- LabEx RESSOURCES21 driving force: M. Cathelineau, LabEx RESSOURCES21 and GeoRessources;
- The participating teams from France (RESSOURCES 21 and VOLTAIRE LabEx) and Portugal share two key objectives:

(i) To re-evaluate the metallogenesis of rare metal resources in Europe, and particularly those located in France and in Portugal.

Achieving this objective requires an integrated multi-disciplinary approach that consists of conducting, in parallel, series of experiments and model simulations concerning two main geological processes:

- Magmatic processes. Through experimental studies, Eramin aims to document the exact way in which elements (and notably, the rare metals) are partitioned in the partial melts as a function of the protolith and the physical conditions of melting. Further research will then be required to decipher the mechanisms by which extreme rare metal enrichments are obtained in the RM P (either by fractional crystallization or through fluid-melt interactions);
- Hydrothermal processes. There is a need for experimental data on the speciation of rare metals, in particular tantalum, for the thermodynamical modelling of ore transportation and deposition. Physical models of fluid circulation that incorporate field-based parameters are also desirable in order to validate (or indeed invalidate) current conceptual models of ore deposition.

To complement this first objective, particular attention will be devoted to aspects that may increase the economic potential of ores. These may include, for example, assessing the potential of common ores (cassiterite, wolframite) to contain rare metals such as Ta, Nb, In, and Sc, and undertaking detailed ore characterisations, especially of low-grade ores, in order to develop innovative processing techniques.

(ii) To develop new technology for ore processing.

The processing of low grade and fine-grained ores presents a considerable challenge, due to the most part to the inefficiency of conventional technologies for low-contrast separation. The separation efficiency of these techniques weakens with decreasing particle size of the valuable minerals. Large quantities of low-grade ore and tailings also necessitate the use of specialised, economical methods for producing metal concentrates of a quality and quantity acceptable for further metallurgical processing.

Application to European call for proposals

MINERVA

Leader M. Pichavant, ISTO-Labex VOLTAIRE, RESSOURCES21, universities across Europe, and SMEs:

The essential goal of MINERVA is to build integrated and project-specific geomodels for Variscan mineralisations. The term 'exploration' encompasses activities ranging from targeting areas for ore-prospecting to determining the factors that control the location of ore-bodies and the distribution of metals within individual deposits. A geomodel is defined here as a conceptual tool that in a given exploration project, drives the entire operational strategy, integrating the various economic, industrial, scientific and technological dimensions that underlie exploration operations.

Establishment of new agreements and international collaborations

Institut de Recherches Mines et Environnements (IRME)

The Research Institute on Mines and Environment (IRME) UQAT-Polytechnique was created in 2013 by the Université du Québec en Abitibi-Témiscamingue (UQAT) and the Polytechnique Montréal. The equivalent of a LabEx structure in France, and unique in Quebec, this joint research programme was created in association with several mining partners. Focusing on the environment and the management of mining wastes, IRME aims to develop environmental solutions for the entire life cycle of a mine. Following the reception of the IRME director (Bruno Bussière) in November 2013 and scientific discussions held at the Québec Mine (November 2013 and 2014) and PDAC meetings (March 2013 and 2014), an international convention between UQAT and the Université de Lorraine was signed in December 2014. This agreement covers both research and teaching in the geosciences domain, with an emphasis placed on subjects related to the mining cycle. Mukendi Kongolo, who is currently participating in a long-term exchange at UQAT, and Laure Giamberini, who was invited to give a presentation on ecotoxicology issues at Quebec Mine 2014, have both benefited from this rising partnership.
LabEx RESSOURCES21 driving force: A.S. André-Mayer.

DIVEX

DIVEX is a research group composed of university and government researchers from seven Quebec universities (INRS-ETE, Laval, Polytechnique, McGill, UQAC, UQAM, and UQAT). Created in 2002, the DIVEX Innovation Network is entirely financed by the Quebec Research Fund - Nature and Technologies (FRQ-NT). In mid-2014, the network launched a research programme on REE occurrences in Quebec, which is scheduled to begin in May 2015. The RESSOURCES21 LabEx is an official scientific partner in this programme and a co-supervised PhD project on REE occurrences in the Grenville Paleoproterozoic province will begin in January 2015. The DIVEX programme will strengthen current interactions with the MRNF Québec (Ministère des Ressources Naturelles et de la Faune), a partnership which has been active since 2008 via educational actions (the Ecole de terrain Abitibi field school , 2008 to 2014). The collaboration has recently been extended to the environment spectra through discussion and interactions with the MEDDLCC (Ministère du Déveoppement Durable et de la Lutte contre le Changement Climatique) who are currently designing a research programme on the environmental impact of REE from exploration and mining (see invited conference at Québec Mines - Laure Giamberini).
LabEx RESSOURCES21 driving forces: A.S. André-Mayer and Laure Giamberini

Center for Exploration Targeting (CET)

CET (Center for Exploration Targeting, Perth, Australia): The Winter 2014 visit of CET director Cam McCuaig was aimed at exchanging ideas about technology transfer and the forging of industrial links, and at building an official partnership between GeoRessources and CET laboratories. A meeting with the international branch of the CNRS and INSU took place in March 2014 and led to the construction of the PICS programme. A joint post-doctoral research project is currently in progress (A. Eglinger) and A.S. André-Mayer has been awarded a 2015 Gledden Visiting Fellowship at the Western University of Australia for two months in autumn 2015.
LabEx RESSOURCES21 driving forces: A.S. André-Mayer

Phytomining network

A network of laboratories working in the field of phytomining has been established and includes Spanish (CSIC), Austrian (BOKU), Albanian (UAT) and Greek (TEIKAV) partners. The network is also open to a number of other strategic partners on an international level: USDA (USA), U. Sun Yat-Sen (China), EMBRAPA-Cerrados (Brazil), and the universities of Melbourne and Brisbane (Australia).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NANCY
IN A SUSTAINABLE

III

DISSEMINATION OF KNOWLEDGE

EDUCATION

With approximately 200 students graduating every year from 5 Masters and 2 Engineering training programmes (see Appendix, p98-99), the Université de Lorraine already offers a range of training programmes in the field of geological resources, from exploration to environmental issues. Emphasis has therefore been placed on training initiatives that will enhance (i) international outreach and visibility, (ii) research at post-graduate and PhD levels, and (iii) interactions between students involved in the field of mineral resources on a local to international scale.

STUDENT REPRESENTATION AT NATIONAL AND INTERNATIONAL EXHIBITS AND EXCHANGES

Québec Mines, November 2013 and 2014

The Université de Lorraine was represented at Québec Mines with a delegation of more than 10 people for each edition (students, PhD students, academic staff, and engineers).

PDAC, Toronto, February, 2012, 2013 and 2014

RESSOURCES21 was also present at the PDAC convention, the world's largest showcase for the field of mineral resources. The delegates presented the training opportunities and research facilities of the Université de Lorraine and its associated laboratories to a worldwide audience.

GEOLOGIA, Nancy ENSG, October, 2012, 2013, 2014

The annual GEOLOGIA fairs have provided the opportunity to promote training facilities in RESSOURCES21-related fields to Masters-level and Engineering students from Grandes Ecoles and universities all over France.

Young Leaders Forum in responsible mining development, November, Québec, 2013

More than ten people from the Université de Lorraine were selected and funded to attend the 2013 Young Leaders Forum in responsible mining development. Under the general theme of science, innovation and society, the Forum offered a series of activities, including workshops, lectures, debates, thematic tours, special meetings and networking events, which allowed them to meet with key stakeholders in the sector. Participants were also able to present their experiences in research or their professional project.

Québec Mines Défi Explo

Since 2012, Quebec Mines has held the EXPLO Quebec Mines Challenge, in which teams of three students, representing various universities, are given 12 hours to produce an exploration project. RESSOURCES21 is now supporting a team from Nancy to participate in this Masters student competition dedicated to mineral exploration.

Geologia le Défi, Nancy ENSG, October 2013, 2014

This competition for Masters students in Geosciences was created in 2013 and takes place during the annual "Geologia" fair in Nancy. The goal of each year's Défi or 'challenge' is to address an industrial issue linked to the field of geological resources. Geothermal Energy and Geological Materials were the themes tackled in 2013 and 2014, respectively. http://www.geologia.fr/accueil/les-manifestations/le-defi/

Funding student at participation to industrial fairs

RESSOURCES21 has provided partial funding for students from the School of Geology, the School of Mines (French Engineering school), and Masters degree programmes to participate in the annual meetings of the National Society of Mineral Industry, (SIM), Québec Mines and PDAC (2012, 2013 and 2014).

The Nancy SGA student chapter

The Nancy SGA student chapter has been created in 2013 by master and PhD students and aims at organizing lectures, workshop, fieldtrip, experiences exchanges in the field of mineral resources. Several lectures have been organized since 2013: projection of the movie "La sale guerre des Terres Rares" and debate with B. Lehmann, M. Cuney and A.S. André-Mayer (around 100 persons); Job opportunities in mineral exploration both in Canada and Australia with M. Jébrak and C. McCuaig (around 50 persons); a one day Fieltrip in the Vosges Mountains to visit old copper mines (15 persons), ...Each year, the student chapter is electing a student who is partially supported by RESSOURCES 21 to be part of the Université de Lorraine PDAC representation.

NEW INTERNATIONAL MASTER: LEVERAGE EFFECTS OF THE LABEX

The two new international Masters programmes are entirely dedicated to the metal life cycle from ore beneficiation to recycling: *(see focus)*

Erasmus Mundus EMERALD

EMerald is a two-year full-time English Masters programme designed to educate a new generation of young professionals by integrating a perfect knowledge of available mineral resources and modern engineering technologies. It gives the students the opportunity to develop a broad and multidisciplinary understanding of georesource processing, from the cradle to the grave and beyond. Modern computer-based technologies used in resource characterisation (digital imaging, tomography) and modelling (geostatistics, 4D GIS) form the backbone of this degree, the objective of which is to train skilled engineers capable of using the most advanced exploration and modelling technologies available. EMerald is a joint organisation of four top-level international universities in the field of georesources: University of Liège (Belgique), University of Lulea (Suède), Freiberg Technisch Universitat, Université de Lorraine (France).

http://www.emerald.ulg.ac.be/

International Masters of Science "Raw materials, Engineering, and Risk Management"

Also launched in 2013, this international MSc is driven by Nancy's two Engineering schools (Ecole de Géologie and Ecole des Mines). The course leads to a Professional Masters degree in Mines and Quarries. It covers the whole of the mineral mining cycle, from the exploration of deposits to the processing and recycling of mining waste, including mining techniques. Strong emphasis is placed on human risk prevention and environmental protection. The course includes visits to industrial sites and fieldwork.

http://www.mines-nancy.univ-lorraine.fr/content/master-international-matières-premières-minérales

RESEARCH TRAINING

The LabEx's main contributions to research training consist of:
The co-funding of research internships at Masters level (more than 8 per year), *see appendix page 97*;
The co-funding of PhD projects: nine projects funded since the beginning of the LabEx, mostly funded at 50% by LabEx and 50% by other institutions (Région Lorraine, Carnot Institute) *see appendix, page 100*.

SCIENCE COMMUNICATION

RESSOURCES21 is a multidisciplinary project aimed at developing knowledge and understanding of the entire strategic metal lifecycle (mining, extraction, processing, and recycling), taking into account issues related to geology, the environment, and ecotoxicology, for example. One of the key objectives of RESSOURCES21 is to disseminate and communicate information on the importance of 'critical' raw materials in order to raise awareness in society and foster social debate. The general public are often unaware of the significance and importance of rare metals, even though they are present in all facets of our daily lives – in our mobile phones, our computers, in low-energy light bulbs, and even in our banknotes. RESSOURCES21 has therefore organised tailored communication events that target three main audiences: the general public, school pupils and political organisations.

GENERAL PUBLIC

The LabEx researchers have presented their work at a number of open events for the general public, allowing them to demonstrate the assorted uses of rare metals in everyday objects. Members of the public were given the opportunity to discover mineral and rock specimens and technological advances, watch and participate in experiments, and meet with and talk to researchers about their work

Temporary exhibitions:
"Moments d'invention", Nancy, 11th to 16th June 2013;
"Ces métaux qui nous entourent», Maxéville, June 2014 & December 2014;
"Science en fête"; ...

Public lectures:

- Alain Cheilletz, 6 March 2014, "Les matières minérales: une mise au point", Villers les Nancy;
- Marie-Christine Boiron, 20 November 2014, "Ces métaux qui nous entourent";
- AS André-Mayer, 1 February 2015, Terrae Genesis, "Ressources minérales: Eléments d'économie et de géopolitique";
- CNRS: "Les fondamentales du CNRS", Grenoble, 2014;
- Science & You 2015: LabEx RESSOURCES21 will be present at this international cultural and scientific event open to the general public and science media.

COLLABORATIVE WIKI

To provide a better understanding and comprehensive illustration of the topic of strategic metals, Alix Marchal and Quentin Lespagnol – two second-year students at the École Nationale Supérieure de Géologie (ENSG) – developed a collaborative website, or 'Wiki', during their three-month internship with LabEx RESSOURCES21. This tool delivers easily-accessible, general information about the economic, social, and political background, and the metal lifecycle (exploration, exploitation, processing, and recycling of metals).
http://www.ressources21.univ-lorraine.fr/en/content/transfer-society

SCHOOL PUPILS

LabEx RESSOURCES21 has weaved a network of local, regional and national partnerships with actors in primary and secondary education. This has provided opportunities for researchers to organise or participate in conferences and to get in touch with the world of schools.

National conference: "Les Ressources et l'Energie" (04.12.2013)

RESSOURCES21 presented the opening seminar at the "Les Ressources et l'Energie" conference in Nancy in December 2013. Organised by "La Maison pour la Science", in partnership with the ESPE, the conference brought together around fifty primary teacher-trainers from across France and around one hundred primary school teachers from the Nancy district.

National conference "Les Ressources géologiques, nouvelles visions pour l'enseignement" (13.11.2013)

RESSOURCES21 was invited to deliver the opening presentation of the national "Les Ressources géologiques, nouvelles visions pour l'enseignement" conference, organised in Paris by the AFPSVT. The congress brought together approximately two hundred people involved in secondary education or teacher-training.

"La Maison pour la Science" and "La Main à la Pâte"

With the aim of creating strong relations with ESPE teams in Lorraine, participation in two initiatives, "La Maison pour la Science" and "La Main à la Pâte", has allowed local teachers to be hosted in laboratories and GeoRessources scientists to visit primary schools in and around Nancy.

POLITICAL REPRESENTATIVES

Public hearing for political representatives

As part of an enquiry commissioned by the 'Parliamentary Office for Evaluation of Scientific and Technological Options' (OPECST), M. Cathelineau was interviewed on the 19th of June, 2014, by M. Antoine, vice director of OPECST. The hearing concerned the issue of metal resources, in particular, the rare earth elements, and the state of research in France in the field of geosciences. The hearing, along with all the other 2014 OPCST hearings conducted in this domain, will be published in a report and made available to the public.

After the scientific (2011-2012) and pedagogical (2012-2013) tasks, comes the final, and equally important, mission of the LabEx RESSOURCES21 team: the transfer of results to industry.

RESSOURCES 21 laboratories can profit from the longstanding relationships with industry that have been established through different partnership structures: (i) the GOCAD consortium for 3D modelling and reserve estimation; (ii) CREGU, a subsidiary of Areva (the world leader in uranium exploration), hosted by and a joint supervisory body of the GeoRessources laboratory; and (iii) the Lorraine Carnot Institute for Energy and Environment (ICEEL), an innovation catalyst for businesses thanks to its skills in geosciences, materials science, process engineering, and transfer of associated technologies.

Gocad Research Group

The Gocad Research Group – part of Georessources (UMR 7359) and the School of Geology at the Université de Lorraine – is a multidisciplinary team whose primary goal is to define new approaches to build and update 3D subsurface models that account for all types of available data. This new technology is made available not only through research papers, but also through computer codes, generally implemented as plugins of Gocad, a commercial application programming interface and software commercialised by Paradigm Geophysical and initially developed in our group. These new approaches may be applied directly in the Gocad Research Group, through collaborative projects, and by academic or industrial Gocad Consortium members. More details about research activities and publications are available on the research page (http://www.gocad.org/w4/). The Gocad consortium is open to industry and universities around the world who hold an interest in subsurface modelling and integration of knowledge and data across all Earth science disciplines, and who wish to use and/or contribute to the development of this new technology. Today, the Gocad consortium consists of 18 companies and 131 universities.

CREGU

Founded in 1978, the CREGU corporation (shareholders: COGEMA - 51% and TOTAL - 49 %) is part of the CNRS-UL GeoRessources Joint Research Unit (UMR) 7359. Located at the interface of industry and research, CREGU accomplishes its various missions through industry contracts, as well as through its participation in regional, national and international programmes. CREGU activities include fundamental and applied research on mineral and energy resources, education through research, technology transfer, technical assistance and expert reports.

A training centre for engineers from enterprises and international organisations, CREGU develops its major themes through observation, analysis, understanding and modelling of the mechanisms at work in the cycle of mineral and energy resources on the five continents: (i) exploration of metals, notably uranium, and oil deposits, from ore-prospecting to the production and economic development of deposits; and (ii) remediation of sites following mine closure.

ICÉEL Carnot Institute for Energy and Environment

The level of interaction with industry is such that laboratories in Lorraine have been awarded the Carnot label, a status which rewards a laboratory for having achieved a significant number of industrial contracts through funding the outsourcing of research activities in order to maintain high levels of skill and technology. The ICÉEL Carnot Institute for Energy and Environment in Lorraine unites one institute and two research networks – Jean Lamour (Materials), Water-Soil-Earth (Geosciences) and Jacques Villermaux (Mechanics, Energy & Chemical Engineering) – that bring together fifteen laboratories affiliated to CNRS and the Université de Lorraine and the eight research transfer centres that comprise the Regional Pole of Research and Technology Transfer (PRETT) in Lorraine. ICÉEL aims to further the development of clean and safe processes with low energy consumption, to work towards the rational use of energy resources, and to support the design of innovative materials and intelligent products. The laboratories that form the ICÉEL institute bring with them a strong tradition of industrial collaboration, founded on one hundred years of experience closely linked to their ten engineering colleges. The knowledge of ICÉEL researchers in the domain of engineering reinforces their industrial partnerships and allows them to find a project environment and cutting-edge technologies adapted to their needs.
The ICÉEL Carnot Institute receives financial contributions from ANR as a function of its research and development activity within the French industrial sector. The LabEx makes a significant contribution towards these industrial links, the geoscience laboratories providing around a third of industry contracts from the academic part of ICEEL (the three poles of the Université de Lorraine).

For the training of future engineers, the School of Geology also holds important links with the STEVAL experimental station, as well as with major French industrial partners in the geosciences (e.g., AREVA, ERAMET, IMERYS), who have a permanent representation at the Councils of the School of Geology (three meetings per year).


Brainstorming session between RESSOURCES21 and ARBRE Labex

LEVERAGE EFFECTS

Enhancing links with other LabEx

Collaborative programmes have been established with LabEx Voltaire on the behaviour of rare metals in Variscan granites, leading to two complementary PhD projects and the submission of an ERAMIN project proposal. RESSOURCES21 and Voltaire also worked together on the submission of a joint proposal to the Raw Materials call from H2020 and the successful Minerva project is due to commence in March 2015. One representative from Voltaire (M. Pichavant) is invited each year to the annual RESSOURCES21 scientific presentation and two representatives from RESSOURCES21 attend the annual Voltaire scientific meeting. Scientific exchanges have also been developed with Chemisyst (Montpellier-Marcoule) and Sérénade (Aix-Marseille) in the field of metal behaviour in the environment. Although working in different fields, the two LabEx based in Lorraine, ARBRE and RESSOURCES21, share some common scientific goals, for example constraining the role of metals in tree development. Finally, the DAMAS and RESSOURCES21 LabEx were involved in the setting-up of the ERAMIN French subgroup discussion day.

Developing new industrial partnerships

• New projects with Eramet on the remediation of mine soils using adapted plants, with application to major new mining projects in Indonesia.
• Recent award of European projects in the field of ore beneficiation (BioMOre, STOICISM, FAME). Project partners include French companies such as Imerys, as well as a number of European companies (Poland, Finland).
• Setting-up of the Minerva project (RESSOURCES21/Volatire co-initiative) in response to the H2020 "Raw Materials" call for proposals. Minerva will involve several industrial partners, including Imerys, Variscan, and companies based in Portugal, the United Kingdom, and Germany.
• Award of an ERAMIN project on tungsten deposits in Western Europe (December 2014) in collaboration with LabEx Voltaire, teams from Portugal (Porto University, LNEG), and the Panasqueira mine (SOJITZ BERALT).

Being proactive in national and international research-industry networks
• Invitation to an industrial-academic brainstorming meeting on REE in Perth, Australia, May 2014.
• Taking over of leadership in the organisation of national meetings in the politics of Raw Materials ("French mirror" group of ERAMIN), and of major scientific meetings such as SGA 2015 (600 participants anticipated).
• Organisation and involvement of the Université de Lorraine in the submission of the KIC Raw materials proposal (awarded December 2014).
• Involvement of the four RESSOURCES21 research teams from Nancy (LRGP and CRPG, LIEC, LSE of Labex) and two SMEs (Soléo Services and Microhumus) in the AGROMINE project. The consortium maintains regular contacts with the main international actors in phytomining: Albania (UAT), Québec (INRS-ETE), China (SYSU), Australia (CMLR-UQ) and the United States (USDA). The results obtained in AGROMINE will greatly benefit the two SMEs and for the ECONICK start-up in Nancy, which is currently in its incubation phase. The fact that LabEx RESSOURCES21 laboratories started a comprehensive examination of its scientific strengths two years ago, through which the 3-year programme on Nickel was selected, has contributed to this success.

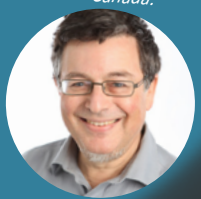
Supporting the development of start-ups
• The LORVER project, supported by La Région Lorraine and the European Union (FEDER - 6.8 M€), aims to create a production chain of non-food vegetable biomass through the recovery of degraded sites and industrial by-products. Composed of a consortium of private companies and research laboratories from the Lorraine region, LORVAR harnesses the skills and know-how required to develop an efficient operating chain, based firmly on the principles of sustainable development, to enhance the economic and environmental potential of waste resources. (http://www.lorver.org/accueil/index.html)
• ECONICK is a start-up project aimed at developing Ni-phytomining by combining Ni-phytoextraction by hyperaccumulation and Ni valorisation using pyro-hydrometallurgical processes (patent WO2012103651). In 2013, Marie-Odile Simonont received an award in the "Concours National d'aide à la création d'entreprises innovantes 2013" (MESR-Bpi France). The prize was used to finance a market study aimed at creating a business start-up.

INDUSTRIAL TRANSFER

Michel Jébrak and Cam McCuaig each spent three months in Nancy thanks to financial support from the RESSOURCES21 LabEx and the Région Lorraine. The two researchers, who are currently involved in industrial transfer structures, brought with them valuable insights from technology transfer in their respective countries and took part in brainstorming sessions organised by the RESSOURCES21 and ARBRE LabEx aimed at discussing actions to accompany the transfer of technology stemming from our research.

Cam McCuaig, director of the Centre for Exploration Targeting (CET) at the University of Western Australia. CET is currently one of the world's largest mineral exploration research groups.

Michel Jébrak, holder of an industrial research chair in mining entrepreneurship, UQAM Québec-Canada.




Michel Cosnard
Université de Lorraine
France

IV

CONCLUSION

CONCLUSION

RESSOURCES21 has enabled Geoscience laboratories in the Lorraine region to structure their research landscape in a way which places them at the cutting edge of worldwide innovation in the field of the natural and anthropogenic cycles of raw materials. In doing so, the RESSOURCES21 LabEx has been able to accomplish its three main missions:

Science

LabEx RESSOURCES21 has enhanced cross-disciplinary and collaborative work and promoted the common ambition of the team of researchers leading the project.

Means and research programmes

Through detailed analysis of the international industrial context, LabEx RESSOURCES21 has identified a number of pertinent scientific questions, each of which is addressed and developed through the financing and co-financing of different types of actions:

• Short-term research programmes designed to stimulate emergent research,

• 4 post-doctoral and 9 PhD fellowships: PhD and post-doctoral research programmes covering the entire metal cycle and several types of metal deposit (Ge-In bearing sulphide deposits, Nb-Ta-Sn-W deposits, Co-Sc in laterites), as well as environmental issues (the behaviour of rare earth elements in the environment and the development of bio-sensors).

• Two integrated 3-year programmes devoted to groups of metals (notably Ni-Co-Sc and REE-Nb-Ta) that display similar geochemical behaviour in the lithosphere and environment, each conducted through study of the mechanisms of enrichment and dispersion of metals.

Financial contributions to research programmes include funds for post-doctoral positions, doctoral fellowships and operating costs, as well as joint-funding of major analytical and technical equipment.

Results already obtained through the LabEx include the development of innovative tools (e.g., biosensors) and the application of new concepts and methodologies for a better understanding of the transport and deposition of metals (for example, experiments on sulphur species and new numerical methods for discontinuity fields), as well as fundamental advances in a number of other fields such as determination of the mode of Ge-In-Ga-Cu uptake by sulphides, the impact of REE on bio-organisms, Ni uptake by plants as a solution for remediation of soils, and Ni-phytomining.

RESSOURCES21 teams have also continued work on strategic metals of high economic importance (e.g. U, Au), for which they have already acquired an international reputation, and on the impact of trace metals (Cd, Ag, Cu,Ti) and metalloids (As) on the environment.

The raw materials workforce at the LabEx has recently witnessed some important changes, with a number of prominent researchers (e.g. M. Cuney) and professors (e.g. A. Cheillez and C. Marignac) retiring at almost the same time as the arrival of new recruits, such as CNRS researchers (J. Mercadier, A. Zegeye) and university professors (A. Galy, P. Pinheiro). Several opportunities to reinforce the human capital of the LabEx themes exist, for example by supporting researcher exchanges (the welcome of researchers of international renown), and the LabEx actively encourages and provides funding for young researchers to spend time abroad in collaboration with universities or industrial partners.

Collaborations with organisations at the national scale: links with LabEx and Carnot Institutes

Joint research programmes on the behaviour of rare metals in Variscan granites have been set up with the Voltaire LabEx, and an ERAMIN project proposal has recently been submitted. Scientific discussions and exchanges have been developed with Chemisyst (Montpellier-Marcoule) and Sérénade (Aix-Marseille) in the field of metal behaviour in the environment. Although working in different fields, the two Lorraine-based LabEx, ARBRE and RESSOURCES21, share some common objectives such as constraining the role of metals on tree development. The DAMAS and RESSOURCES21 LabEx have also worked together in the setting up of the ERAMIN French subgroup discussion day.

European dimension

The dynamism of the LabEx is measured easily by the success rate of its European project submissions (close to 80%). At the European level, the "LabEx" label and its associated financial support has allowed our researchers to secure and enhance their standing in European networks and to increase their participation in the submission of proposals to European calls (active involvement in the European ERAMIN and EODI networks - European Ore Deposit Initiative, now closed). RESSOURCES21 was also active in the organisation of the most recent meeting of the French discussion group of the ERAMIN network. LabEx RESSOURCES21 has been active in various working groups at the Université de Lorraine in preparing applications for new calls such as the EIT "KIC Raw Materials". Other highlights include the acceptance of the European FP7 STOICISM project (with IMERYS), the Erasmus Mundus Masters programme, two new projects within the framework of H2020 (BIOMORE and FAME), and the ERAMIN NewOres project. The LabEx team worked alongside the Voltaire LabEx in the setting up of the Minerva project on ores related to felsic magmatism in Western Europe, which has been submitted to the Raw Materials H2020 call for proposals. Future perspectives include an increase in the number of IUF or ERC project submissions stimulated by LabEx research.

Education and diffusion of knowledge

Training in the field of mineral resources, from their exploration to their exploitation, is already a key component of the Masters degrees and other programmes run by Nancy's two Engineering Schools. The LabEx has furthered the training opportunities in this domain through the setting up of new university-level training programmes such as the Emerald 'Duby' Masters.

The diffusion of expertise and knowledge has been effective across different areas:

• Industry - The LabEx benefits from long-lasting ties with industrial partners, such as Areva, Imerys and Eramet, established via the GOCAD consortium for 3D-modelling and reserve estimations, and CREGU, a subsidiary of Areva - the world leader in uranium exploration, hosted by and co-supervisor of the GeoRessources laboratory.

An important leverage effect of LabEx has been the development of new projects with Eramet on the remediation of mine soils using adapted plants, with application in major new mining projects in Indonesia. A major co-initiative of the RESSOURCES21 and Voltaire LabEx has been the setting up, in collaboration with European SMEs, of the "Minerva" project in response to the recent 'Raw materials' call in the framework of H2020.

• The general public - LabEx researchers have presented their research at several open meetings dedicated to a public audience, allowing the use and exploitation of rare metals in all objects of our everyday lives to be presented on a number of different occasions.

• Political representatives - Recent interactions include work with the Office parlementaire d'évaluation pour les choix scientifiques technologiques (Parliamentary Office for the Evaluation of Scientific and Technological Options).

Leverage effects of LabEx RESSOURCES21

At the national and European level, the RESSOURCES21 label and the financial means obtained as a result have allowed the researchers to secure and enhance their standing within European networks and to increase their level of participation in the submission of proposals to European calls. By careful structuring of its research community, LabEx RESSOURCES21 has provided the means with which to foster new ideas, develop researcher consortiums and establish cross-disciplinary research programmes. The main leverage effects of LabEx RESSOURCES21 have been:

• The provision of funding and incentives for developing new ideas and collaborations for the setting-out of new proposals at regional to European funding-level. This has resulted in a series of recent successes (notably, the Eramin 'NewOres' project, the H2020 'Biomore' project, and a project on Sc geochemistry in laterites with CNRT-New Caledonia) that could not have been achieved without the stimulation, organisation, help, and sometimes co-funding of LabEx.

• The creation of new training programmes, some at international level, such as the Emerald Masters degree.

• Assuming leadership in the organisation of national meetings in the politics of raw materials (ERAMIN 'French mirror' group), and of major scientific meetings such as SGA 2015 (600 participants anticipated).

• The organisation and involvement of the Université de Lorraine in the submission of the KIC Raw materials proposal.

• The development of new research programmes with French industry, for example the study of Ni behaviour in soils in new mining targets for Eramet, the systematic use of LA-ICP-MS for better understanding the incorporation of strategic metals (e.g., REE (Areva) and Ge) in minerals and substrates, and the setting up of original research on the transfers between soils and plants.

Perspectives

The two 3-year programmes will largely determine the structure of the next four years of the LabEx in terms of research. In association with the research programmes, a detailed calendar of scientific and communication actions will be organised including, for example, international workshops and meetings on Ni-Co and on REE-Nb concentrations in 2017-2018. The key objective of the RESSOURCES21 project is to establish an international reputation for knowledge and know-how that will ensure the sustainability of the financial income and scientific excellence of the LabEx teams in the many years to come.

APPENDIX

NEWSLETTER N°1 - LabEx RESSOURCES21 - JUNE 2013

RESSOURCES21

RESSOURCES21, "Rare metallic resources in the 21st century", is a research, training and advancement scheme for the environmental management of natural resources of critical metal in the 21st century. Many metals are gaining more and more applications in manufactured products and technology linked to carbon-free energy. Although just a few years ago access to metals was thought to be assured, in that they were plentiful and easy to reach, recent economic and political pressures have meant that it is now necessary to secure our supplies for the coming decades. Consequently, we must redefine the notion of resources in terms of geology, concentrations of metals and recycling, whilst taking environmental management and any potential toxic effects on ecology into account. Find out more

AN AID AGREEMENT WITH THE ANR

The RESSOURCES21 project, following a 2010 tender for projects from the Commissariat Général aux Investissements d'Avenir. The definitive version of the agreement between the ANR (Agence Nationale de la Recherche) and the University of Lorraine was signed on 18 March 2013. It aims to define the specific conditions in which the RESSOURCES21 research project will be carried out and financed. The project was selected as part of the "laboratoires d'excellence" initiative.

DEVELOPING AND ADVANCING LabEx

RESSOURCES21 recruited its engineering project manager to spearhead the development and advancement of LabEx: Laurie Wolff's remit will include implementing operations with partners, drawing up projects in response to tenders, representing LabEx at national and international conferences, and organising scientists' meetings.

A DISCUSSION ON THE MINING AND MINERAL INDUSTRIES IN CANADA

Seven researchers from Lorraine have travelled to Toronto to take part in PDAC 2013 (Prospectors & Developers Association of Canada) as part of LabEx RESSOURCES21. This is an excellent opportunity as it generally takes 5 to 8 years after registration to get a place. This year, the PDAC welcomed around 30,000 participants and 600 exhibitors all working in mining in more than 125 countries. Taking part in this event has allowed the Lorraine team to demonstrate its skills in training and research, to boost its students' recruitment possibilities on the international stage, and to establish and reinforce partnerships with the mining and mineral industry. It was also a chance to promote the university and LabEx RESSOUCES21.

NICKEL AND RARE EARTH ELEMENTS: ESSENTIAL COMPONENTS FOR NEW TECHNOLOGY

More than 50 researchers came to the nickel study day on 21 March and the rare earth elements study day on 2 April, giving them the chance to pool their knowledge regarding strategic interests and the work already completed by the LabEx teams. The nickel study day demonstrated the rich variety of approaches to nickel that the LabEx laboratories use and showed that there is a real community of people working in this area (the origins of deposits, treating ores, the environment, modelling). The aim of the rare earth metals elements study day was to draw up an inventory of activities, from metallogenesis to treating ores and environmental impacts, and consider multi-disciplinary actions that could play a part in LabEx RESSOURCES21.

THE METALS THAT MAKE OUR WORLD - RENAISSANCE NANCY 2013 - MOMENTS D'INVENTION

If you are worried about the environment or just curious, come and meet our researchers: they will unveil the secrets behind researching and using the rare metals found in everyday objects. The focus of this initiative will be to reach out to the general public by introducing attendees to samples, advances in technology and different experiments. Our researchers and students will be there to welcome you every day, from midday to midnight on the Place Charles III in Nancy, 11-16 June 2013.

SIX MONTHS TO DEVELOP NEW PARTNERSHIPS IN CANADA

Yann Gunzburger, head of conferences at the GéoRessources laboratory and the man in charge of the "Exploitation et stockages" stream of the master's degree in GPRE (Géosciences, Planètes, Ressources, Environnement), is currently on a 6-month scientific visit to the Mine Design Laboratory at McGill University in Montreal as a guest researcher. This visit has been partly financed by LabEx RESSOURCES21 and aims to develop contact with universities in Quebec and the Canadian mining industry, particularly with regards to the use of natural resources. The hope is that this will generate new research and training collaborations. One of his current scientific projects centres on understanding mechanisms and foreseeing sudden ruptures ("rock bursts") in deep mines in hard rock, which cause several accidents every year.

A PROJECT FOR CREATING INNOVATIVE BUSINESSES

The ECONICK project (for the design of an environmentally friendly, high value nickel production process) has been accepted for entry into the Incubateur Lorrain. Led by Marie-Odile Simmonot, the project aims to create a phytomining business that produces high-added-value nickel salts and gives advice on phytoextraction. The "incubation" phase allows the project and its staff to reach a state of readiness. A package of services is gradually implemented as the project develops, with the final outcome being the creation of a company and the launch of the product onto the markets in the best possible conditions. In addition to this, thanks to Oséo (and in particular its competition for new talent) the following projects will be financed: a market analysis, an intellectual property analysis and a technical evaluation of the potential for creating a start-up.

GOLD MEDAL FOR ONE OF LORRAINE'S RESEARCHERS

The council for the Société de Géologie Appliquée en Métallogénie (SGA) has decided to award Michel Cuney the SGA-Newmont Gold Medal for 2013. This international prize is given in recognition of a career that encompasses particularly original work in geology and mining. The work in question has made a major contribution to (1) science through the research and (2) development of knowledge in the area of mineral resources, exploration and discovering deposits. Michel Cuney is CNRS research director at GéoRessources and has carried out research into uranium metallogeny. La Société Géologique de France has also awarded him the Barbier Prize 2013.

PUBLICATIONS

See publications

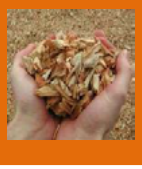
CNRS PLACEMENT WINNER

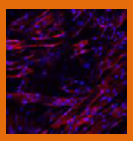
Asfawe Zegeye, a postgraduate student at LIEC, financed by R21, has been recruited into the CNRS during the CR2 selection process in April. He was ranked 5th in the list of 7 winners in Section 30.

FOCUS ON THE LORVER PROJECT

The LORVER project aims to restore the fertility of spoiled soil (a site for storing contaminated earth such as Sita-FD) and industrial wastelands (such as the GISFI experiment station in Homécourt) following past industrial activities. This will be done by reconstituting the soil to allow specific cultures to be planted for industrial purposes (fibres, energy, biochar, metals), such as poplars, hemp, nettles and hyperaccumulating plants.





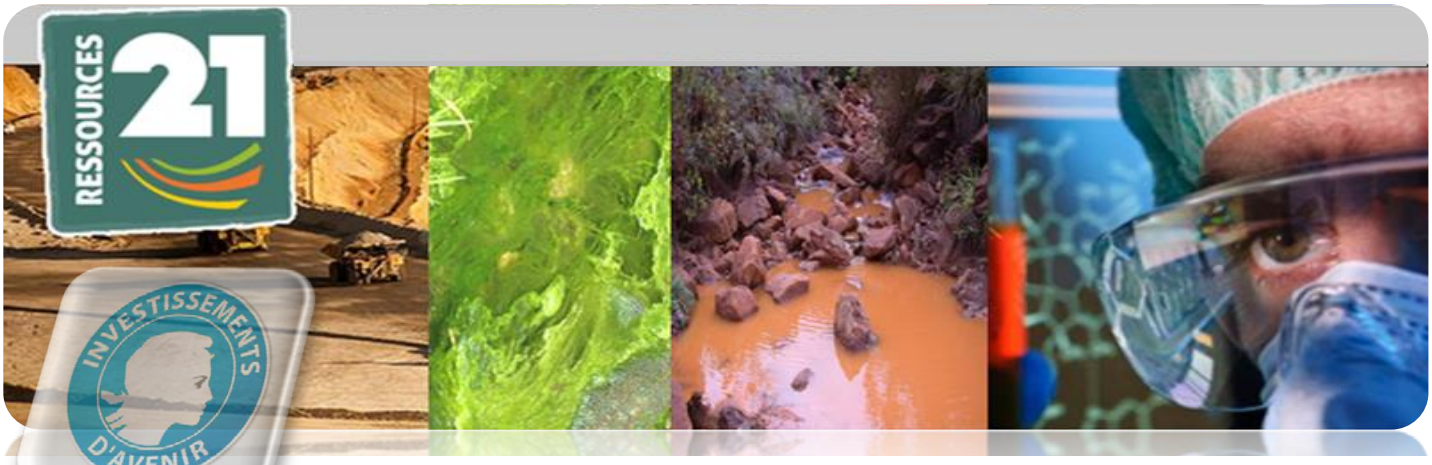
PROJECT ID

Launch	July 2012
Duration	5 years
Budget	€6.8 M financed by the Agence de Mobilisation Economique (AME) at the Région Lorraine et l'Europe (Feder)
Scientific aims	Gaining knowledge of how spoiled sites and soils function and evolve in the long term
Socio-economic aims	Creating an option that gives site owners (e.g. ArcelorMittal Real Estate France, EPFL) an alternative to using agricultural land for processing biomass that is not destined for consumption as food.
Partners	▪ 4 companies: Valterra (porteur du projet), Sita-FD, Chanvriers de l'Est and SEA-Marconi ▪ 5 laboratories based in Lorraine, including two belonging to LabEx RESSOURCES21 (LSE and LIEC), LRGP (VERTBILOR1), du LERMAB (Pôle Fibres Grand Est) ▪ 1 research centre: CRP Lippmann du Luxembourg ▪ 2 transfer structures: PROGEPI and CETELOR

Editor: Laurie Wolff

otelo Observatoire Terre Environnement Lorraine — UNIVERSITÉ DE LORRAINE — cnrs — INRA

LabEx RESSOURCES21 NEWSLETTER No. 2 - NOVEMBER 2013



RESSOURCES21

The RESSOURCES21 project is looking at how France and Europe source their supplies of strategic metals for the energy and high technology goods fields. In this context, RESSOURCES21 is focusing more particularly on primary strategic metal resources (rare earths, uranium and nickel especially) in terms of geological concentration processes, mineralurgy and environmental impact as well as the management of secondary resources and recycling. Find out more

INTERNATIONAL MASTERS

The so-called "DUBY" masters course for foreign students in "Mineral raw materials, risk engineering and management" offered by the *École des Mines* in Nancy and the *Ecole Nationale Supérieure de Géologie* - ENSG, has been approved by the Ministry. Its aim is to train mining industry managers in the countries that produce and export raw materials in order to create lasting links with these countries, the mine operators and their future managers. This new master course will start in autumn 2014 with its first intake and will take over from the CESTEMIN and the CESEV courses, which have lost their funding due to budget cuts by the ministry. These two undergraduate courses were created in Nancy in 1975 at the *Ecole Nationale Supérieure des Mines* and at the ENSG in the fields of mining technology (CESTEMIN) and exploration of mineral resources and ore processing (CESEV) respectively.

link

MICHEL CUNEY HONOURED ONCE AGAIN

After being awarded the SGA-Newmont Gold Medal for 2013, Michel CUNEY has just been appointed Honorary Professor at the East China Institute of Technology in Fuzhou, after a period spent at this university, which trains most of China's geologists in exploration and research.

RECRUITMENT

Julien Mercadier (GeoRessources) has been recruited by the CNRS as a CR2-level researcher specialising in Metallogeny.

PROFESSOR CLAUDE FORTIN AT THE LIEC

Claude Fortin, Professor at the INRS-ETE (Water-Earth-Environment Centre) in Quebec and Canada Research Chair in biogeochemistry of trace elements, recently spent two months (from 10 June to 2 August 2013) at the LIEC (Interdisciplinary Laboratory for Continental Environments). His stay gave him the chance to forge close links with researchers at the LIEC, but also to share his knowledge and maximise the synergies and complementarity of research being carried out on rare earths, critical metals and nanomaterials. Areas were identified where scientific cooperation is possible and a number of joint projects will be put forward. Reciprocal exchanges and joint supervision of students, as well as technique sharing between the two laboratories, were also seriously considered.

SUCCESS AT THE RESSOURCES21 - MOMENTS D'INVENTION – RENAISSANCE NANCY FAIR

Our researchers and students manned the RESSOURCES21 stand from 11 to 16 June 2013, welcoming the general public every day from midday to midnight. Our estimation is that approximately 150 to 180 people a day showed an interest in strategic metals, attended the demonstrations and engaged in discussion with the "researchers" present. The public's questions were particularly centered on the real place of metals in everyday life, the risks regarding the availability of supplies and the economic impact in Lorraine. This first RESSOURCES21 operation was a success and will surely be repeated, building on the solid content and experience.

SGA MINERAL DEPOSITS

RESSOURCES21's researchers gave some fifteen presentations at the SGA conference in Uppsala, Sweden, which this year was attended by almost 670 researchers and representatives of industry working in the mineral resources field. The next SGA Meeting will be held in Nancy from 24 to 27 August 2015

MEETING WITH THE MINERAL INDUSTRY SOCIETY

The Director of RESSOURCES21, Frederic Villieras, and his Scientific Director, Michel Cathelineau, met with the regional section of the SIM (Mineral Industry Society) on 5 June 2013. This event was an opportunity for the two directors to present RESSOUCES21 and the OTELo Pole of the University of Lorraine, of which it is a part. The 2013 SIM annual congress took place in Besançon at the beginning of October, and students from the ENSG and ENSMN were present at the congress exhibition. http://www.lasim.org/

SOCIETY OF ENV. TOXICOLOGY AND CHEMISTRY (SETAC)

At the SETAC Annual Meeting held in Glasgow in May 2013, Laure Giamberini chaired a session on strategic elements in the 21st century. The session included 5 presentations and demonstrated that the life cycle assessment (LCA) approach has the potential to manage the risks relating to critical materials. Product indicators could constitute measures integrated at European level to manage the use of natural resources and the related risks.

FOCUS ON RESSOURCES21's POSTDOCS

Nicolas Estrade

After a Masters in environmental chemistry, I completed a PhD thesis on "Discrimination of anthropic sources of mercury in the environment" at the University of Pau (IPREM/LCABIE) and the CRPG at the University of Lorraine. As part of the LabEx project and working with LSE and the CRPG, my job was to develop an analytical technique for measuring Ni isotopes using the double spike method to correct for instrumental mass fractionation of the machine. Once this stage was validated, we investigated the variations in nickel isotope composition in nickel hyperaccumulating soil-plant systems. These plants are of crucial importance to the LabEx project because their extraordinary capacity to take up nickel from the soil enables them to play a role in the phytoremediation of contaminated soils or to be used for "phytomining" of nickel-rich soils. The results achieved constitute a significant contribution to our knowledge of the geochemistry of nickel isotopes and have given LabEx an analytical tool it can use for future investigations.

N. Estrade is now conducting postdoctoral research in Canada

Marc Ulrich

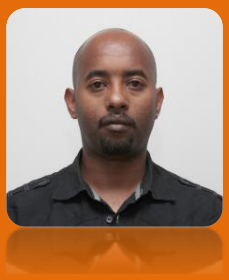
After completing a PhD in geology in 2010 and 2 years in a temporary lecturing post in Grenoble, I joined the GeoRessources laboratory in Nancy in September 2012. My research aims to understand the distribution of metals of economic interest such as nickel, cobalt, chromium and manganese in the laterite profile in New Caledonia. A particular focus of the study is the behaviour of scandium, one of the group of rare earth elements, and at risk of a shortage of supply in the short term. My postdoctoral work is being funded mainly by LabEx Ressources 21, with a financial contribution from Koniambo SAS, a mining company in New Caldeonia which is participating in the project.

M. Ulrich has taken up a post as a lecturer at the University of Strasbourg

Veronica GONZALEZ

I obtained my PhD in January 2012 (Soil Science Department. University of Almeria, Spain) and I currently have a postdoctoral position funded by LabEx RESSORCES21. My PhD was based on contamination problems, toxicity and remediation of soil contaminated by mining activities between the University of Almeria and Amsterdam University. During my postdoctoral training in LabEx, my main activities have been centered in rare earth elements (REEs); a group of elements with a broad spectrum of application in different industry sectors but with scarce ecotoxicological information. In this way, a detailed literature search was performed in order to identify research needs (results presented in the Congress SETAC, Glasgow 2013); after, a comparative study of REEs ecotoxicity has been performed by using different aquatic organisms. Final aim is to apply these results in understanding the environmental fate and ecotoxicity of these less studied elements in the environment.

V.Gonzalez is currently in her second postdoctoral year



Asfaw ZEGEYE

My research work concerns the biochemical cycles of elements, iron in particular, with a particular interest in bacteria-mineral interaction. My research combines solids analysis techniques and molecular approaches in order to determine reaction rates and mechanisms and the secondary products of these biogeochemical transformations. My postdoctoral project with Ressource21, working in the LIEC laboratory, is seeking to establish a relationship between the bioreduction of antimony-doped iron oxide and the bioavailability of this element using bacterial biosensors.

A. Zegeye has taken up a position as a CNRS CR2-level researcher at the LIEC

Responsable de publication : Laurie Wolff

NEWSLETTER N°3 - LabEx RESSOURCES21 – July 2014

RESSOURCES21

RESSOURCES21 is a project funded as part of the French "Future Investment" national programme, with a budget of 9 million Euros (2011-2019). It pursues three main aims: (i) Understanding the formation processes of metal deposits, and the natural and anthropogenic cycle of strategic metals; (ii) Develop innovative tools and procedures to better manage and exploit these deposits; (iii) To grasp the environmental impact of these metals once they have been scattered throughout the ecosystem. Three broad areas of action complement the main aims: (i) Pushing the frontiers of analysis in terms of finding and measuring trace elements within rocks and dating geological events; (ii) Modelling the geometry, movement and physical chemistry processes at regional level to understand the distribution of metals and their development in space and time; (iii) Designing and developing new biogeochemical sensors to monitor the scattering of dangerous elements and predict pollution. More info at www.ressources21.univ-lorraine.fr/en/project/download.html

RECRUITMENT

Two students from the National School of Geology (Quentin Lespagnol and Alix Marchal) have been recruited for a 3 month placement to produce a web-documentary providing general information on strategic metal.

PRIZE

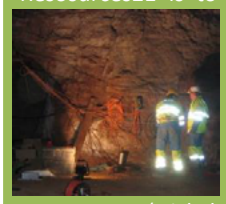
Arnaud Botella received a prize for his poster on hybrid grid generation during the RP2E Graduate School open day. These grids are an essential element in achieving the modelling-related aims of LabEx on coupled calculations in complex geological models.

PUBLICATIONS

See our Ressources21 publications at https://www.zotero.org/groups/ressources21

AN UPDATE ON OUR SCIENTIFIC PROGRAMMES

One of the main aims set by LabEx Resources21 is to produce new scientific and technological knowledge to enable the identification and exploitation of future deposits. The launch of the integrative programmes (Nickel Programme 2014-2017 and Rare Earth Programme 2015-2018) have allowed the community to focus on its priorities, thus encouraging interaction around the same topic. The Nickel programme will launch in September 2014 and the preparation workshops for the Rare Earth Programme have already revealed a community supporting new scientific questioning.



RESEARCHERS FROM LORRAINE STILL CONTRIBUTE TO THE DEVELOPMENT OF PHYTOMINING

New Scientist 2961, 22 March 2014, page 49)

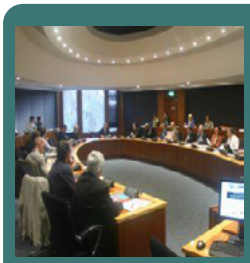
Aida Bani from the Agricultural University in Tirana, who studied under Jean-Louis Morel and Guillaume Echevarria in the noughties, has published an article on her research in New Scientist. Echevarria originally set up these tests, now producing conclusive results. The development of phytomining in Albania is the result of our investment since the 1990's.

As part of this collaborative work, testing was extended to Cuba and Brazil where researchers like former LSE post-doc and current colleague of Guillaume Echevarria, Leide de Andrade from the Embrapa Research Institute, are working.

New Scientist states that Eramet wants to use phytomining to rehabilitate biodiversity.

The journal also mentions that the ECONICK project (for the design of an environmentally friendly, high value Nickel production process) is now being incubated at Lorraine. This start-up is led by Marie-Odile Simmonot and aims to create a phytomining business that produces high-added-value nickel salts.

FRANCO-AUSTRALIAN MEETING ON RARE EARTH ELEMENTS IN PERTH



Michel Cuney and Lev Filippov represented Geosciences in Nancy and "RESSOURCES21" at this workshop organised by the French Embassy. Many industrial and academic stakeholders attended, giving us a better grasp of the challenges this field faces due to markets, prospection and rare earth element production processes as well as an overview of the chemical and physical chemical challenges involved in flotation and leaching etc. The associated issues were presented by several bodies (CSIRO, UniSA, ANSTO, CEAtech and Georesources). Visits to laboratories and research centres emphasised the importance of the research work in mineral raw materials. Thanks to the conferences on the subject, there are many mining projects underway, with the Université de Lorraine, Chimie ParisTech and Curtin University began working together by exchanging researchers and setting up joint research projects.

QUÉBEC MINES 2013

A 10-person delegation from Lorraine represented the University and RESSOURCES21 at the Québec Mines 2013 congress. The students were able to extend their networks, giving them a chance to find a placement or a job in Canada. Three of them were given the chance to take part in the MINES EXPLO contest where several teams compete to deliver the best exploration project). The job of the lecturers was to strengthen Franco-Québec ties and to make it easier for French students to find jobs in Canada. The Franco-Québec Youth Office (OFQJ) funded the involvement of 7 students from Lorrain in the International Forum for Young Leaders in Mining Development that took place during the show. The delegation from the Université de Lorraine and French companies were received by the French Consul in Québec.

RESEARCHERS' MOVIE FESTIVAL

The Researchers' Film Festival is a CNRS / Université de Lorraine event that took place between 10th and 15th June 2014 at the Parc de la Pépinière in Nancy. Scientists and directors got the chance to answer questions from the audience. 60 films screened for free, researchers and festival-goers discussing until the early hours, passers-by who stopped then stayed, film-making school-children, science (and comedy) shows. The first evening was dedicated to "The Continental Waltz" with Christian France-Lanord (from CRPG) as the speaker.

OTELO and RESSOURCES21 presented an exhibition for the general public, built around 5 totems focusing on the future challenges of rare metals that are essential to our daily lives and the environmental impact of these metals, including tracking them.

FOCUS ON GUEST RESEARCHERS

Prof Jose Paulo PINHEIRO, specialist in environmental physical chemistry, lecturer at the University of the Algarve (Portugal) spent a month in Nancy hosted at the LIEC in March 2014. His stay enabled him to share his knowledge and (i) explore electroanalytical methods for developing sensors and speciation analysis of trivalent metals, especially rare earth elements, in natural waters (*ii*) and to study the behaviour of a model trivalent metal (Indium) in solution in the presence of natural particles (humic acids). The last point was the subject of a Masters placement in 2014 supervised by Paulo Pinheiro and Elise Rotureau. The work carried out so far has enabled the operating conditions to be perfected for assessing the speciation of *In* by voltametry, and to acquire a first data set on the interactions between Indium and various molecular or particulate ligands. Jose Paulo PINHEIRO's was recruited after as a lecturer at ENSG.

Currently a researcher/lecturer at the University of Geneva, **Prof Montserrat FILELLA** joined the LIEC from January to June 2014. The aim of her stay was to set up research into the geochemistry of strategic elements. Mme Filella was involved in the integration into ecotoxicology studies of metal speciation such as those she set up in aquatic environments, as well as experimental cultivation environments. She supervised a campaign of antimony sampling in water from old mineworkings, and established the conditions for effective speciation in bioavailability studies. As part of a Masters, Montserrat FILELLA set up and ran the protocols for the chemical and physical separation of the components in aquatic organic matter, in the context of biodiversity and ecotoxology studies. Projects underway will be continued through other joint efforts.

Prof Daniel Fornasiero from the Ian Wark Research Center, Associate Professor at the University of South Australia spent 8 weeks as part of the "Resource and Residue Recovery" team at the Georesources laboratory thanks to funding by the Emerald programme. He was involved in 3 modules for Emerald Masters students and presented two conferences on different aspects of modelling flotation microdynamics for mixed and composite particles. He also took part in discussions on progress in the team's research projects and existing thesis work. As part of the Ressources21 "Nickel" project, he developed a research programme on the properties and recovery of fine particles contained in lateritic ores.

Dr Laurent Aillères is a Professor at Monash University in Australia. He was the guest of GeoRessources for 3 weeks as part of the EMERALD Masters. We took the opportunity to discuss implied-surface based 3-D modelling methods and discuss future collaboration (an article is currently being written with Gautier Laurent, a former doctoral student and current post-doc at Monash). Laurent Aillères produced an applied mining exploration module in M1 Emerald based on geophysical and geomodelling data.

Publishing Editor: Isabelle Abildtrup, Graphic design: Laurie Wolff

Publication by: Frédéric Villieras

PhD Thesis

(in blue, PhD thesis linked to research financed by LabEx Ressources21 or to precursor actions to the LabEx programmes)

Defended PhD thesis

• BONNEAU François (2011-2014), Prise en compte d'informations indirectes dans la simulation de réseaux de fractures. Dir. CAUMON, G., SAUSSE, J., RENARD, P.

• BONNET Julien, 2014, Cycle géologique et processus métallogéniques de métaux rares (Ge, Ga, I). Inter-Carnot ICEEL et BRGM. Encadrant : A.S. André ; Mayer, Co-encadrants L. Bailly et J. Cauzid, Thèse Carnot ICEEL-BRGM, 11 Décembre 2014.

• BONNETTI Christophe, 2013 Genèse des gisements d'uranium associés aux bassins sédimentaires continentaux. L'exemple du bassin d'Erlian (Chine). Thèse université de Lorraine -Nancy (Bourse AREVA), direction M. Cuney, S. Bourlange, F. Malartre 13 Décembre 2013

• CALAND Fabrice (17/09/2013) - Décomposition tensorielle de signaux luminescents émis par des biosenseurs bactériens pour l'identification de Systèmes Métaux-Bactéries- Thèse de l'Université de Lorraine - spécialité Géosciences - Direction conjointe LIEC-CRAN (C. Mustin, D. Brie)

• DARGENT Maxime, 2014, Etude expérimentale de la spéciation des ions uranyles en solutions chlorurées acides et de la cinétique de précipitation de l'uraninite par réduction (H2, CH4, C, H2S). Encadrants : J. Dubessy et L. Truche, financement Carnot. soutenue en Décembre 2014.

• DIONDOH Sandrine, Contexte géologique et géodynamique des minéralisations Au et U de la ceinture panafricaine d'Afrique centrale dans le massif du Mayo Kebbi (Tchad). Encadrant : Encadrants : O. Vanderhaeghe, 2010-2015.

• DUVERGER - Agathe - Valorisation par flottation des minerais à faible teneur en uranium : étude de la texture et des effets synergiques des réactifs de flottation – L. Filippov – 09/12/2013

• DYJA Vanessa, 2014, Interaction entre fluides de différents réservoirs lors de lévolution d'un prisme orogénique en contexte de déformation partitionnée: les Cordillères bétiques internes (Espagne). Implications sur le transfert de métaux dans la croûte. (mars 2014). Encadrants : C. Hibsch, M. Cathelineau, Thèse MNESR

• EGLINGER Aurelien, 2013 Cycle de l'uranium et évolution tectono-métamorphique de la ceinture orogénique Pan-Africaine du Lufilien (Zambie). direction: A.S. André ; Mayer, O. Vanderhaeghe, bourse MNESR, Thèse Université de Lorraine, 13 décembre 2013

• FLEURANCE Stéphanie, 2012, Métallogenèse de l'uranium associée à des processus superficiels : l'exemple de la Jordanie centrale, Thèse soutenue le 13 décembre 2012, Directeurs de thèse : M. Cuney et F. Malartre

• JOMINI Stéphane. 2014- Ecotoxicité des nanoparticules (TiO2, ZnO, CeO2) et des résidus de dégradation de nanomatériaux sur les communautés bactériennes : Impact sur la diversité taxonomique et fonctionnelle. Direction de thèse. Pascale Bauda. Christophe Pagnout

• LACH Philippe, 2012, Signature géochimique des éléments des terres rares dans les oxydes d'uranium et minéraux associés dans les gisements d'uranium : analyse par ablation laser couplée à l'ICP-MS et étude géochronologique. Thèse soutenue le 7 décembre 2012, Directeurs de thèse : M. Cuney, J. Dubessy et MC Boiron

• LAURENT Gautier (2010-2013), Compatibilité des structures en modélisation 3D. Dir. CAUMON, G.

• LE MIGNOT Elodie, 2014, L'or Ouest Africain : Marqueur d'un transfert majeur manteau-croûte. WAXI. Encadrant : A.S. André ; Mayer, Co-encadrante L. Reisberg. Thèse MNESR, soutenue en Décembre 2014.

• LECOMTE Andrei, 2014, Relations spatiales et génétiques entre uranium, sulfures et matières organiques : application aux shales et schistes noirs, soutenance janvier 2014, Thèse CARNOT ICEEL

• MERLAND Romain - Génération de grilles de type volumes fini : adaptation à un modèle structural, pétrophysique et dynamique - G. Caumon, B. Lévy et P. Collon-Drouaillet - 18/04/2013

• MEJIA Pablo (2014) Apport de la modélisation 3D et de la restauration structurale dans la compréhension de gisements de matières premières minérales. Dir. JJ Royer

• MUNARA Askar, 2012, Formation des gisements d'uranium de type roll : approche minéralogique et géochimique du gisement uranifère de Muyunkum (Bassin de Chu-Sarysu, Kazakhstan). Thèse soutenue le 9 Juillet 2012, Directeurs de thèse : Michel Cathelineau, Cédric Carpentier

• PARRELLO Damien (2014) Conception de biosenseurs fluorescents multicolores pour l'identification in vivo des interactions bio-physico-chimiques dans les systèmes minéral-bactérie.

• PELLERIN Jeanne (2010-2014), Maillages conformes de domaines géologiques. Dir. LEVY, B., CAUMON, G.

• POIREL Jessica (2013) - Réponses adaptatives des communautés bactériennes telluriques aux métaux et métalloïdes : liens avec la disponibilité des polluants métalliques dans les sols. Direction : Corinne Leyval (DR CNRS) et Dr Patrick Billard

• TOE Wilfried Antoine Bassou, 2012, Minéralisations uranifères de la ceinture orogénique Pan-Africaine du Damara (Namibie) : implications à la fusion partielle, de la migration et de la mise en place des magmas sur le remaniement de la croûte continentale. Thèse soutenue le 11 décembre 2012, Directeurs de thèse :. AS Andre-Mayer et O. Vandehaeghe.

Ongoing PhD Thesis

• AHARCHAOU. Imad 2013-Mécanismes de toxicité des éléments trivalents : importance des espèces solubles et insolubles Dir. Davide A.L. Vignati et Eric Battaglia. Financement bourse ministérielle.

• ANDREI. Jennifer 2012-Effets des nanoparticules manufacturées sur les invertébrés d\'eau douce et leurs fonctions au sein des écosystèmes. Approche intégrée chez le gammare. Direction de thèse Pr. François GUÉROLD et Dr. Sandrine PAIN-DEVIN. Co financé Labex R21. Région Lorraine

• BAHAR Tidjani Bahar (2012-), Impact de la présence d'une phase bactérienne sur les processus d'écoulement et de transport multiphasique en milieu poreux. Dir. GOLFIER, F., OLTEAN, C.

• BELISSONT Rémi, Cycle du Germanium et éléments accompagnateurs dans les concentrations métalliques : Traçage élementaire, isotopique et approche expérimentale Encadrants : MC. Boiron, B. Luais, M. Cathelineau, Thèse Labex R21 et région Lorraine, 2012- 2015.

• BENIOUG, Marbe (2011-), Etude expérimentale et numérique de la croissance microbienne en milieu poreux. Dir. BUES, M., GOLFIER, P., OLTEAN, C.

• BERTHELOT Charlotte, Phytoremédiation de sols pollués - Étude des mécanismes de tolérance aux métaux lourds chez les plantes et leurs symbiotes associés. UL, BVF, LIEC, direction Leyval C.

• BERTRAND Carole 2013 - Nanomatériaux à travers un gradient de salinité: exposition et effets ecotoxicologiques au cours de leur cycle de vie (production, utilisation, fin de vie). Directrice L Giamberini, C Mouneyrac (Université Catholique de l'Ouest). Codirecteurs S. Devin, L Poirier (UCO).Financement ANR nanosalt

• BOTELLA Arnaud (2013-), Maillage hybride de domaines géologiques. Dir. CAUMON, G.

• CARROUE Simon. Genèse des granites potassiques archéens riches en uranium dans la ceinture de Barbeton, direction Jean Françoic Moyen, codirection, M. Cuney. Thèse Univ. St Etienne.

• CHEN Fengjuan (2013-), Modélisation microporomécanique de roches poreuses hétérogènes et applications aux roches oolithiques. Dir. GIRAUD, A., GRGIC, D.

• DELONCA Adeline (2011-), Incertitudes sur les paramètres des éboulements rocheux et leurs impacts sur l'évaluation du risque. Dir. VERDEL, T., GUNZBERGER, Y.

• FAIVRE Maxime (2012-) Modélisation du comportement hydrogéomécanique d'un réseau de faille sous l'effet des variations de l'état de contrainte. Dir. GOLFIER, F., GIOT, R.

• GARAUD Mael. 2011- Effets des nanoparticules manufacturées (CeO2, TiO2 et Ag0, nanotubes de carbone) sur la biologie du bivalve d'eau douce Dreissena polymorpha. Direction de thèse. Laure Giambérini & Vincent Felten. Financement ANR Mesonnet

• HAFEZNIA Yaser, Minéralisations de Sainte-Marie aux Mines, Encadrants : M. Ohnenstetter et S. Bourlange, 2010-2014.

• HARLAUX Mathieu, Les minéralisations tardi-orogéniques varisques à tungstène et métaux associés (Sn, Nb, Ta) : source des fluides et des métaux, relations avec les évènements tectonomagmatiques fini-carbonifères sur l'exemple de gisements du massif central français. Encadrants M. Cuney ; codirection, C. Marignac, J. Mercadier. Thèse Labex R21- Carnot ICEEL, 2013- 2016

• KANBAR Hussein 2012-2015 Devenir des métaux dans les enregistrements sédimentaires, cas des barrages de Moyeuvre et Homécourt sur l'Orne. Dir Frederic Villiéras, Antoine El Samrani université du Liban, Codirectrice E. Montarges-Pelletier codir, codir EMP

• LE MEUR, Mathieu 2012-2015 Nature et réactivité des matières en suspension de la Moselle, influence de la minéralogie et des propriétés de surfaces sur le mode de fixation des métaux».directeur Frédéric Villiéras, Codirectrice E. Montarges-Pelletier

• MAMANE Marah, Les gisements d'uranium du Niger, synthèse métallogénique, Direction M. Cathelineau, 2013- 2016

• MARTIN Jennifer 2011-2014. Dynamique de spéciation de cations métalliques aux interphases molles biotiques (bactéries) et abiotiques (hydrogels). Directeur J. Duval, Codirectrice E Rotureau. Financement bourse ministérielle.

• MARTZ Pierre (2014) Evolution spatio-temporelle des migrations de fluides et de la formation des minéralisation uranifères de Cigar Lake et de leurs extensions. Encadrants M. Cathelineau, J. Mercadier

• MIYAKI Andrey -2014- Formation des minéralisations de Ni saprolitique en Nouvelle Calédonie : Minéralogie, séquence paragénétique et modélisation des interactions fluides roches et de la dynamique des transferts de métaux. Encadrants : F. Golfier, M. Cathelineau, L. Truche- Thèse Labex programme Nickel.

• MOLLEX Gaelle, Les concentrations en Terres Rares dans les carbonatites : exemple de l'Oldoinyo Lengaï, Tanzanie. Direction L. France et P. Burnard, Thèse Labex R21 et région Lorraine, 2013- 2016

• NGUYEN Anhtuan (2012-), Modélisation des massifs rocheux fracturés appliquée aux mines à ciel ouvert et aux carrières : du relevé de fractures à la modélisation de la stabilité des ouvrages avec prise en compte des incertitudes. Dir. MERRIEN-SOUKATCHOFF, V., VINCHES, M.

• PAUL Bertrand (2013-), Modélisation de la propagation de fissures hydrauliques par la méthode des éléments finis étendue. Dir. GOLFIER, F., MASSIN, P.

• PERRAT Emilie 2013- Impacts environnementaux des agents de contraste au gadolinium : situation locale, approche cellulaire et in vivo. Dir. C Cossu Leguille et Marc PARANT. Financement Région Lorraine et Ressource 21.

• REICHART Guillaume (2010-), Fonctionnement thermo-hydro-chimique des réservoirs de mines profondes ennoyées. Dir. BUES, M., COLLON-DROUAILLET, P.,

VAUTE, L.

• RONGIER Guillaume (2013-), Simulation Multipoints de corps géologiques connectés. Dir. SAUSSE, J., COLLON-DROUAILLET, P.

• RUIU Jérémy (2011-), Analyse géologique orientée objets d'images digitales de dépôts sédimentaires. Dir. CAUMON, G., VISEUR, S.

• BARRE Guillaume (2014-) - Rôle de la spéciation du soufre sur les processus redox affectant son cycle géochimique dans la lithosphère. Encadrant L. Truche, thèse MNERT

• TURLIN François (2014-), Les gisements de REE magmatiques, traceurs des processus de croissance et de différenciation de la croûte continentale : Exemple de la province orogénique protérozoïque du Grenville, Québec. Dir. Anne-Sylvie André-Mayer Co-encadrants O. Vanderhaeghe (GET, Toulouse) et Felix Gervais (Polytechnique Montreal). Thèse Region-Labex

• LEGROS Héléne (2014-), Granites et metallogenèse à métaux rares (W, Ta, Nb, Li, Sn) du SE de la Chine et comparaison avec la chaîne varisque européenne. Thèse inter Carnot ICEEL-BRGM. Encadrants : J. Mercadier, T. Augé

Publications

(in blue, publications linked to research financed by LabEx Ressources21 or to precursor actions to the LabEx programmes)

PRA1+ 4+5: Geological processes and ore deposits, exploration and exploitation 2012

Abidi, R; Slim-Shimi, N; Marignac, C; Hatira, NI; Gasquet, D; Renac, C; Soumarin, A. Gleeson, S., 2012, The origin of sulfate mineralization and the nature of the BaSO4-SrSO4 solid-solution series in the Ain Allega and El Aguiba ore deposits, Northern Tunisia. Ore Geology Reviews, 48, 165-179.

André-Mayer A.S. and Sausse J. (2012) Fissural permeability in the Roşia Poieni copper deposit: Influence on ore repartition at the open pit scale. Romanian Journal of Earth Sciences, vol. 86, issue 1, p. 1-15.

Berkesi, M; Guzmics, T; Szabo, C; Dubessy, J; Bodnar, RJ; Hidas, K; Ratter, K (2012) The role of CO2-rich fluids in trace element transport and metasomatism in the lithospheric mantle beneath the Central Pannonian Basin, Hungary, based on fluid inclusions in mantle xenoliths. Earth and Planetary Science Letters, 331, 8- 20. DOI: 10.1016/j.epsl.2012.03.012

Carbone, C; Marescotti, P; Lucchetti, G; Martinelli, A; Basso, R; Cauzid, J (2012) Migration of selected elements of environmental concern from unaltered pyrite-rich mineralizations to Fe-rich alteration crusts. Journal of geochemical exploration, 114, 109- 117.

Cathelineau M., Boiron M.C., Fourcade S., Ruffet G., Clauer N., Belcourt O, Coulibaly Y., Banks D. A., Guillocheau F., 2012. A Kimmeridgian to Berriasian major fluid event at the basin/basement unconformity in western France: 39Ar-40Ar and K-Ar dating, fluid chemistry, and related geodynamic context. Chemical Geology, 322-323, 99-120.

Chattaraj, P.K., Das, R., Duley, S. & Vigneresse, J.L., 2012. Structure-stability diagrams and stability-reactivity landscapes: a conceptual DFT study. Theoretical Chemical Acta 131, 1089, DOI 10.1007/s00214-012-1089-y

Cherpeau, N; Caumon, G., Caers, JK; Levy, B (2012) Method for stochastic inverse modeling of fault geometry and connectivity using flow data. Mathematical Geosciences, 44:2, 147-168.

Collon-Drouaillet, P; Henrion, V; Pellerin, J. (2012) An algorithm for 3D simulation of branchwork karst networks using Horton parameters and A-star. Application to a synthetic case. Geological Society of London - Special Publications, 370 (1)

Cuney M., Emetz A., Mercadier J., Mykchaylov V., Shunko V., Yuslenko A. 2012, Uranium deposits associated with Na-metasomatism from central Ukraine: a review of some of the major deposits and genetic constraints.Ore Geology Reviews, 44, 82-106.

Deschamps, F; Godard, M.; Guillot, S.; Chauvel, C.; Andreani, M.; Hattori, K.; Wunder, B.; France, L. 2012, Behavior of fluid-mobile elements in serpentines from abyssal to subduction environments: Examples from Cuba and Dominican Republic, Chemical Geology, 312-313, 93-117

Duley, S; Vigneresse, JL; Chattaraj, PK (2012) Fitness landscapes in natural rocks system evolution: A conceptual DFT treatment. JOURNAL OF Chemical Sciences, 124, 29- 34.

Escoube, R.; Rouxel, O.; Luais, B.; Ponzevera, E.; Donard, O., 2012, An intercomparison study of the germanium isotope composition of geological reference materials, Geostandards and Geoanalytical Research, 36, 2, 149-159

Feneyrol, J.; Onhenstetter, D.; Giuliani, G.; Fallick, A.E.; Rollion-Bard, C.; Robert, J.L.; Malisa, E.P., 2012, Evidence of evaporites in the genesis of the vanadian grossular 'tsavorite' deposit in Namalulu, Tanzania; The Canadian Mineralogist, 50, 745-769

Giot, R; Giraud, A; Guillon, T; et al. (2012) Three-dimensional poromechanical back analysis of the pulse test accounting for transverse isotropy. Acta Geotechnica7(3): 151-165 DOI: 10.1007/s11440-012-0158-7

Giraud, A; Nguyen, NB; Grgic, D (2012) Effective poroelastic coefficients of isotropic oolitic rocks with micro and meso porosities. International journal of engineering science 58: 57-77 DOI: 10.1016/j.ijengsci.2012.03.025

Harthong, B; Scholtes, L; Donze, F (2012) Strength characterization of rock masses, using a coupled DEM-DFN model. Geophysical journal international 191(2) : 467-

480 DOI: 10.1111/j.1365-246X.2012.05642.x

Huber, C; Bachmann, O; Vigneresse, JL; Dufek, J; Parmigiani, A., 2012, A physical model for metal extraction and transport in shallow magmatic systems. Geochemistry Geophysics Geosystems (2012) 13.

Isseini M. , André-Mayer A. S., Vanderhaeghe O., Barbey P., Deloule E., 2012, A-type granites from the Pan-African orogenic belt in south-western Chad constrained using geochemistry, Sr–Nd isotopes and U–Pb geochronology Original Research ArticleLithos, Volume 153, 39-52

Leisen M., Boiron M.C., Richard A., Dubessy J., 2012, Determination of Cl and Br concentrations in individual fluid inclusions by combining microthermometry and LA-ICPMS analysis: Implications for the origin of salinity in crustal fluids. Chemical Geology, 330-331, 197-206.

Leisen M., Dubessy J., Boiron M.C., Lach P., 2012. Improvement of the determination of element concentrations in quartz-hosted fluid inclusions by LA-ICP-MS and Pitzer thermodynamic modeling of ice melting temperature. Geochimica Cosmochimica Acta, 90, 110-125.

Lemarchand J., Boulvais Ph., Gaboriau M., Boiron M.C., Tartèse R., Cokkinos M., Bonnet S. 2012, Giant quartz vein formation and high-elevation meteoric fluid infiltration into the South Armorican Shear Zone: geological, fluid inclusion and stable isotope evidence.Journal of the Geological Society, London, 169, 17-27.

Luais, B., 2012, Germanium chemistry and MC-ICPMS isotopic measurements of Fe–Ni, Zn alloys and silicate matrices: Insights into deep Earth processes. Chemical Geology, 334, 295–311.

Mainhagu, J; Golfier, F; Oltéan, C; Buès, M (2012) Gravity-driven fingering in fractures: experimental investigation and dispersion analysis by moment method for a point-source injection, Journal Contaminant Hydrology, 132: 12-27. doi:10.1016/j.jconhyd.2012.02.004

Mercadier, J; Richard, A; Cathelineau, M., 2012, Boron- and magnesium-rich marine brines at the origin of giant unconformity-related uranium deposits: delta B-11 evidence from Mg-tourmalines. Geology, 40, 231-234.

Morteani G., Eichinger F., Götze J., Tarantola A., and Müller A. (2012) Evaluation of the potential of the pegmatitic quartz veins of the Sierra de Comechigones (Argentina) as a source of high purity quartz by a combination of LA-ICP-MS, ICP, cathodoluminescence, gas chromatography, fluid inclusion analysis, Raman and FTIR spectroscopy. Quartz: deposits, mineralogy and analytics in Springer Book, DOI: 10.1007/978-3-642-22161-3_5, 119-137.

Richard A., Rozsypal C., Mercadier J., Banks D.A., Cuney M., Boiron M.C., Cathelineau M., 2012. Giant uranium deposits originate from exceptionally U-rich brines. Nature Geoscience, 5, 142-146.

Saadat, F; Sevostianov, I; Giraud, A (2012) Approximate Representation of a Compliance Contribution Tensor for a Cylindrical Inhomogeneity Normal to the Axis of Symmetry of a Transversely Isotropic Material. INTERNATIONAL JOURNAL OF FRACTURE 174(2): 237-244 DOI: 10.1007/s10704-012-9688-0

Sadequi, M., Bouabdellah, M., Boushaba, A., Marcoux, E., Cheilletz, A., 2012, Mineralogy, fluid inclusion, and oxygen isotope constraints on the genesis of the Lalla Zahra W-(Cu) deposit, Alouana district, northeastern Morocco. Arabian Journal of Geosciences, 2,

Scholtes, L; Donze, F (2012) Modelling progressive failure in fractured rock masses using a 3D discrete element method. International journal of rock mechanics and mining sciences 52: 18-30 DOI: 10.1016/j.ijrmms.2012.02.009

Sevostianov, I; Giraud, A (2012) On the Compliance Contribution Tensor for a Concave Superspherical Pore. International journal of fracture 177(2): 199-206 DOI: 10.1007/s10704-012-9754-7

Seydoux-Guillaume A. M., , Montel J. M., Bingen B., BosseV., , de Parseval P. , Paquette J.L., Janots E., Wirth R., 2012, Low-temperature alteration of monazite: Fluid mediated coupled dissolution–precipitation, irradiation damage, and disturbance of the U–Pb and Th–Pb chronometers. Chemical Geology, Volumes 330–331, 140-158

Solovova, IP; Ohnenstetter, D; Girnis, AV., 2012, Melt inclusions in olivine from the boninites of New Caledonia: Postentrapment melt modification and estimation of primary magma compositions. Petrology, 20, 529-544.

Sun, R; Dubessy, J., 2012, Prediction of vapor-liquid equilibrium and PVTx properties of geological fluid system with SAFT-LJ EOS including multi-polar contribution. Part II: Application to H2O-NaCl and CO2-H2O-NaCl System. Geochimica Cosmochimica Acta, 88, 130-145

Tarantola, A; Diamond, LW; Stunitz, H; Thust, A; Pec, M., 2012, Modification of fluid inclusions in quartz by deviatoric stress. III: Influence of principal stresses on inclusion density and orientation. Contributions to mineralogy and petrology, 164, 537-550

Uher, P; Giuliani, G; Szakall, S; Fallick, A; Strunga, V; Vaculovic, T; Ozdin, D; Greganova, M., 2012, Sapphires related to alkali basalts from the Cerova Highlands, Western Carpathians (southern Slovakia): composition and origin. Geologica Carpathica, 63, 71-82

Vanderhaeghe O. 2012, Thermal-mechanical evolution of orogenic belts at convergent plate boundaries: a reappraisal of the orogenic cycle, Journal of Geodynamics, 56-57 pp. 124-145.

Vigneresse J. L., 2012, Chemical reactivity parameters (HSAB) applied to magma evolution and ore formation. Lithos, Volume 153, Pages 154-164.

2013

Berger, J., H. Diot, L. Khalidou, D. Ohnenstetter, O. Féménias, M. Pivin, D. Demaiffe, A. Bernard, and B. Charlier. 2013, Petrogenesis of Archean PGM-bearing chromitites and associated ultramafic–mafic–anorthositic rocks from the Guelb el Azib layered complex (West African craton, Mauritania). Precambrian Research 224 (2013): 612–628.

Blaise T., Barbarand, J., Kars M., Ploquin F. Aubourg Ch. Brigaud B., Cathelineau M. El Albani, A., Gautheron C., Izart A., Janots D., Michels R., Pagel M., Pozzi JP, Boiron MC Landrein Ph. (2013) Reconstruction of low burial (< 100 °C) in sedimentary basins: A comparison of geothermometer sensitivity in the intracontinental Paris Basin, Marine and Petroleum Geology, sous presse

Bonneau, F; Henrion, V; Caumon, G; Renard, P; Sausse, J (2013) A methodology for pseudo-genetic stochastic modeling of discrete fracture network. Computers & Geosciences, 56 (12-22)

Caumon, G; Gray, GG; Antoine, C; Titeux MO (2013) 3D implicit stratigraphic model building from remote sensing data on tetrahedral meshes: theory and application to a regional model of La Popa Basin, NE Mexico. IEEE Transactions on Geoscience and Remote Sensing, 51:3: 1613-1621

Caumon, M.-C., Dubessy, J., Robert, P. and Tarantola, A., (2013) Fused-silica capillary capsules (FSCCs) as reference synthetic aqueous fluid inclusions to determine chlorinity by Raman spectroscopy. Eur. J. Mineral., 25: DOI: 10.1127/0935-1221/2013/0025-2280.

Cepedal A., Fuertes − Fuente M., Martin − Izard A., Garcia − Nieto J., Boiron M.C., 2013, An intrusion −related gold deposit (IRGD) in the NW of Spain, the Linares deposit: Igneous rocks, veins and related alterations, ore features and fluids involved. Journal of Geochemical Exploration, 124, 101−126.

Conin M., Bourlange S., Henry P., Boiselet A., 2013, Distribution of resistive and conductive structures in Nankai accretionary wedge reveals contrasting stress paths, Tectonophysics

Decree, S; Marignac, C; De Putter, T; Yans, J; Clauer, N; Dermech, M; Aloui, K; Baele, JM., 2013, The Oued Belif Hematite-Rich Breccia: A Miocene Iron Oxide Cu-Au-(U-REE) Deposit in the Nefza Mining District, Tunisia. Economic Geology, 108, 1425- 1457.

Demaiffe, D., J. Wiszniewska, E. Krzeminska, I. S. Williams, S. Brassinnes, D. Ohnenstetter, and E. Deloule. 2013, A hidden alkaline and carbonatite province of early carboniferous age in northeast Poland: zircon U-Pb and pyrrhotite Re-Os geochronology. Journal of the Geology 121, 91–104.

Devineau, K; Devouard, B; Leroux, H; Tissandier, L (2013) Incorporation of Zn in the destabilization products of muscovite at 1175 degrees C under disequilibrium conditions, and implications for heavy metal sequestration. American Mineralogist, 98, 932- 945. DOI: 10.2138/am.2013.4213

Didier, A. Bosse, V; Boulvais, P; Bouloton, J; Paquette, JL; Montel, JM; Devidal, JL., 2013, Disturbance versus preservation of U-Th-Pb ages in monazite during fluidrock interaction: textural, chemical and isotopic in situ study in microgranites (Velay Dome, France). Contributions to Mineralogy and Petrology, 165, 1051-1072. DOI: 10.1007/s00410-012-0847-0

Durand-Riard, P; Guzofski, CA; Caumon, G; Titeux, MO (2013) Handling natural complexity in 3D geomechanical restoration, with application to the recent evolution of the outer fold-and-thrust belt, deepwater Niger Delta. AAPG Bulletin, 97:1, 87-102.

Eglinger, A., André-Mayer, A.S., Vanderhaeghe, O., Mercadier, J., Cuney, M., Decrée, S., Feybesse, J.L., Milesi, J.P. 2013, Geochemical signatures of uraninite: From unconformity to syn-metamorphic uranium deposits in the Panafrican Lufilian Copperbelt. Ore Geol. Rev., 54, 197-213.

Feneyrol, J. Giuliani, G., Ohnenstetter, D. Fallick, AE., Martelat, JE. Monie, P., Dubessy, J., Rollion-Bard, C; Le Goff, E; Malisa, E; Rakotondrazafy, AFM; Pardieu, V; Kahn, T; Ichangi, D; Venance, E; Voarintsoa, NR; Ranatsenho, MM; Simonet, C; Omito, E; Nya (2013) New aspects and perspectives on tsavorite deposits. Ore Geology Reviews, 53, 1- 25.

Fleurance S., Cuney M., Malartre F. , Reyx, J., 2013, Origin of the extreme polymetallic enrichment (Cd, Cr, Mo, Ni, U, V, Zn) of the Late Cretaceous–Early Tertiary Belqa Group, central Jordan Original Research ArticlePalaeogeography, Palaeoclimatology, Palaeoecology, 369- 1, 201-219.

Giraud, A; Sevostianov, I (2013) Micromechanical modeling of the effective elastic properties of oolitic limestone. Internation journal of rock mechanics and mining sciences 62: 23-27 DOI: 10.1016/j.ijrmms.2013.04.001

Grgic, D; Giraud, A; Auvray, C (2013) Impact of chemical weathering on micro/macro-mechanical properties of oolithic iron ore. International Journal Of Rock Mechanics And Mining Sciences. 64: 236-245 DOI: 10.1016/j.ijrmms.2013.09.005

Lach P, Mercadier J., Dubessy J., Boiron M.C., Cuney M., 2013, In-situ quantitative measurement of rare earth elements in uranium oxides by Laser Ablation-Inductively Coupled Plasma-Mass Spectrometry. Geostandards and geoanalytical research. 37, 277-296.

Laurent, G; Caumon, G; Bouziat, A; Jessell, M (2013) A parametric method to model 3D displacements around faults with volumetric vector fields. Tectonophysics, 590:83—93.

Le Carlier, Ch., Alexandre, P., Ruffet, G. Cuney, M., Cheilletz, A. (2013). A two-stage exhumation in Western French Massif Central: new geochronological evidences of syn-collisional extension. LITHOS, 175, 1-15.

Lomine, F; Scholtes, L; Sibille, L; et al. (2013) Modeling of fluid-solid interaction in granular media with coupled lattice Boltzmann/discrete element methods: application to piping erosion International Journal For Numerical And Analytical Methods In Geomechanics 37(6), 577-596 DOI: 10.1002/nag.1109.

Montel, JM; Giot, R (2013) Fracturing around radioactive minerals: elastic model and applications. Physics And Chemistry Of Minerals, 40, 635- 645.

Nizkaya, T; Angilella, JR; Buès, MA (2013) Inertial focusing of small particles in wavy channels: Asymptotic analysis at weak particle inertia. Physica D Nonlinear Phenomena 268:91-99

Oltéan, C; Golfier, F; Buès, MA (2013) Numerical and experimental investigation of buoyancy-driven dissolution in vertical fracture. Journal of Geophysical Research: Solid Earth, 118(5), 2038–2048, doi: 10.1002/jgrb.50188

Orgogozo, L; Golfier, F; Buès, MA; Quintard, M; Kone, T (2013) A dual-porosity theory for groundwater contaminant transport in biofilm-coated porous media. Advances in Water Resources, 62(Part B), 266-279

Pellerin, J; Levy, B; Caumon, G; Botella, A (2014) Automatic Surface Remeshing of 3D Structural Models at Specified Resolution: a Method Based on Voronoi Diagrams. Computers & Geosciences 62: 103-116

Pignatelli, I., Mugnaioli, E. Hybler, J. Mosser-Ruck, R. Cathelineau M.and Michau N. (2013) A multi-technique characterization of cronstedtite synthesized by iron–clay interaction in a step-by-step cooling procedure, Clays and Clay Minerals, Vol. 61, No. 4, 277–289, 2013

Rabeau, O; Royer, JJ; Jebrak, M; Cheilletz, A (2013) Log-uniform distribution of gold deposits along major Archean fault zones. Mineralium Deposita 48(7): 817-824.

Rasoamalala, V., Salvi, S., Béziat, D., Ursule, J.P., Cuney, M., Moine, B., Guillaume, D., Andriamampihantona, J. (2013) Geological setting of the bastnaesite and monazite deposits in the Ambatofinandrahana area, central part of Madagascar: an overview. Journal of African Earth Sciences

Richard A., Cauzid J., Cathelineau M., Boiron M.C., Mercadier J., Cuney M., 2013, Synchrotron-XRF and XANES investigation of uranium speciation and element distribution in fluid inclusions from unconformity-related uranium deposits. Geofluids, 13,110-121

Richard, A., P. Boulvais, J. Mercadier, M. C. Boiron, M. Cathelineau, M. Cuney, and C. France-Lanord. «From evaporated seawater to uranium-mineralizing brines: Isotopic and trace element study of quartz–dolomite veins in the Athabasca system.» Geochimica et Cosmochimica Acta 113 (2013): 38–59

Scholtes, L; Donze, FV (2013) A DEM model for soft and hard rocks: Role of grain interlocking on strength. Journal Of The Mechanics And Physics Of Solids 61(2): 352-369 DOI: 10.1016/j.jmps.2012.10.005

Sevostianov, I; Giraud, A (2013) Generalization of Maxwell homogenization scheme for elastic material containing inhomogeneities of diverse shape. International Journal Of Engineering Science, 64, 23-36 DOI: 10.1016/j.ijengsci.2012.12.004

Siebenaller L., Boiron M.C., Vanderhaeghe O., Hibsch C., Jessell M., André-Mayer A.S., France-Lanord C., Photiades A. (2013) Fluid record of rock exhumation across the brittle-ductile transition during formation of a Metamorphic Core Complex (Naxos Island, Cyclades, Greece). Journal of metamorphic geology, 31, 313-338

Tartese, R; Boulvais, P; Poujol, M; Gloaguen, E; Cuney, M (2013) Uranium mobilization from the variscan Questembert syntectonic granite during fluid-rock interaction at depth. Economic Geology, 108, 379- 386

Toe W., Vanderhaeghe O. André-Mayer A.S., Feybesse J.L. and Milesi J.P. (2013) From migmatites to granites in the Panafrican Damara Belt. Journal of African Earth Sciences, 85, 62–74.

Truche, L., Jodin-Caumon, M-C, Lerouge, C., Berger, G., Mosser-Ruck, R., Giffaut, E., Michau, N., (2013). Sulphide mineral reactions in clay-rich rock induced by high hydrogen pressure. Application to disturbed or natural settings up to 250°C and 30 bar. . Chem. Geol. 351, 217-228.

Welsch, B., F. Faure, V. Famin, A. Baronnet, and P. Bachèlery. 2013, «Dentritic crystallization: A single process for all the textures of olivine in basalts?» Journal of Petrology 54, no. 3 539–574

2014

Andre-Mayer, A.S., Ramiandrisoa, N., Vanderhaeghe, O., Reisberg, L., Rabeandrasana S., Zimmermann, C. (2014) Re-Os geochronological constraints on the Dabolava mesothermal gold occurence, Madagascar: Implications for the Ikalamavony sub-domain deposition age. J. Afr. Earth Sci. 94, 119–127 (2014)

Belissont, R., Boiron, M.-C., Luais, B. & Cathelineau, M. (2014) LA-ICP-MS analyses of minor and trace elements and bulk Ge isotopes in zoned Ge-rich sphalerites from the Noailhac - Saint-Salvy deposit (France): Insights into incorporation mechanisms and ore deposition processes. Geochim. Cosmochim. Acta 126, 518–540 (2014)

Benko Z., Molnár F., Lespinasse M., Váczi T., (2014) Evidence for exhumation of a granite intrusion in a regional extensional stress regime based on coupled microstructural and fluid inclusion plane studies: An example from the Velence Mts., Hungary ,Journal of Structural Geology, , ,http://dx.doi.org/10.1016/j.jsg.2014.04.001

Benko Z., Molnar F., Lespinasse M., Billstrom K., Pecskay Z., Nemeth T., (2014) Triassic fluid mobilization and epigenetic lead-zinc sulphide mineralization in the Transdanubian Shear Zone (Pannonian Basin, Hungary),Geologica Carpathica,65, 3,177-194,doi: 10.2478/geoca-2014-0012

Bonneau, F., Henrion, V., Caumon, G., Renard, P. & Sausse, J. (2014) A methodology for pseudo-genetic stochastic modeling of discrete fracture networks. Computers & Geosciences 56, 12–22 (2013)

Bonnetti C., Malartre F., Huault V., Cuney M., Bourlange S., liu X., Peng Y., (2014) Sedimentology, stratigraphy and palynological occurrences of the late Cretaceous Erlian Formation, Erlian Basin, Inner Mongolia, People's Republic of China. Cretac. Res. 48, 177–192 (2014)

Cai Y., Verdel T., Deck O. (2014) On the topography influence on subsidence due to horizontal underground mining using the influence function method,Computers and Geotechnics,61,328–340,10.1016/j.compgeo.2014.06.003

Cuney, M. & Barbey, P. (2014) Uranium, rare metals, and granulite-facies metamorphism. Geoscience Frontiers (2014). doi:10.1016/j.gsf.2014.03.011

Dargent M., Dubessy J., Truche L., Nguyen-Trung C., Robert P., 2014, Experimental study of uranyl(VI) chloride complex formation in acid LiCl aqueous solutions under hydrothermal conditions (T = 21 °C - 350 °C, Psat) using Raman spectroscopy. Eur. J. Min, 25, 765-775

Decree, S., Deloule, E. , De Putter, T., Dewaele, S., Mees, F., Baele, J.M., Marignac, C. (2014) Dating of U-rich heterogenite: New insights into U deposit genesis and U cycling in the Katanga Copperbelt. Precambrian Res. 241, 17–28 (2014)

Decrée S. , Marignac C., Liégeois J-P., Yans J., Ben Abdallah R., Demaiffe D. (2014) Miocene magmatic evolution in the Nefza district (Northern Tunisia) and its relationship with the genesis of polymetallic mineralizations. Lithos 192–195:240–258

Delonca, A., Gunzburger, Y., Verdel, T. (2014) Statistical correlation between meteorological and rockfall databases,Natural hazards and Earth system sciences

(NHESS),14,01/12/14,10.5194/nhess-14-1-2014

Eglinger A., Feirrana C., Tarantola A., André-Mayer A.S., Vanderhaeghe O., Boiron M.C., Dubessy J., Richard A. and Brouand M. (2014)Metamorphic evolution of U-bearing highly saline Ca-Na fluids in syntectonic Pan-African quartz veins in the Domes region (Lufilian belt, Zambia).,Contribution to Mineralogy and Petrology., 167:967

Eglinger A., Tarantola A., Durand C., Ferraina C., Andre Mayer A.S., Vanderhaeghe O., Paquette J-L., Deloule E. (2014) Uranium mobilization by fluids associated with Ca–Na metasomatism: A P–T–t record of fluid–rock interactions during Pan-African metamorphism (Western Zambian Copperbelt). Chem. Geol., doi:10.1016/j.chemgeo.2014.07.028

Giraud, A. & Sevostianov, I. (2014) Micromechanical modeling of the effective elastic properties of oolitic limestone. Int. J. Rock Mech. Min. Sci. 62, 23–27 (2013)

Gloaguen, E. et al. (2014) Tracing the magmatic/hydrothermal transition in regional low-strain. zones: The role of magma dynamics in strain localization at pluton roof, implications for intrusion-related gold deposits. J. Struct. Geol. 58, 108–121 (2014)

Jaguin J., Boulvais P., Boiron M.C., Poujol M., Gapais D., Ruffet G., Briant N. (2014) Stable isotopes (O, C) and fluid inclusion study of quartz-carbonate veins from the Antimony Line, Murchison Greenstone Belt. American Journal of Science. vol 314, 1140-1170

Laurent, G., Caumon, G., Bouziat, A. & Jessell, M. (2014) A parametric method to model 3D displacements around faults with volumetric vector fields. Tectonophysics 590, 83–93 (2013).

Lecomte A., Cathelineau M., Deloule E., Brouand M., Peiffert C., Loukola-Ruskeeniemi K., Pohjolainene E., Lahtinen H. (2014) Uraniferous bitumen nodules in the Talvivaara Ni-Zn-Cu-Co deposit (Finland): influence of metamorphism on uranium mineralization in black shales. Miner. Depos. 49, 513–533 (2014)

Mejía-Herrera, P., Royer, J.-J., Caumon, G. & Cheilletz, A. (2014) Curvature Attribute from Surface-Restoration as Predictor Variable in Kupferschiefer Copper Potentials: An Example from the Fore-Sudetic Region. Natural Resources Research (2014). doi:10.1007/s11053-014-9247-7

Mouélé I. M., Dudoignon P., El Albani A., Cuney M., Boiron M. C., Gauthier-Lafaye F. (2014) Percolation of diagenetic fluids in the Archean basement of the Franceville basin. CR Geoosciences, 346, 13-19

Mouélé I. M., Patrick Dudoignon, Abderrazak El Albani, Alain Meunier, Philippe Boulvais, François Gauthier-Lafaye, Jean-Louis Paquette, Hervé Martin, Michel Cuney (2014) 2.9–1.9 Ga paleoalterations of Archean granitic basement of the Franceville basin (Gabon) Journal of African Earth Sciences 09/2014; 97:244–260

Pellerin J., Levy B., Caumon G., Botella A. (2014) Automatic surface remeshing of 3D structural models at specified resolution: A method based on Voronoi diagrams,Computers and Geosciences,62,103-116,10.1016/j.cageo.2013.09.008

Pellerin J., Levy B., Caumon G. (2014)Toward Mixed-element Meshing based on Restricted Voronoi Diagrams,Procedia Engineering,82,279-290,10.1016/j.proeng.2014.10.390

Rasoamalala, V., Salvi, S., Béziat, D., Ursule, J.P., Cuney, M., Moine, B., Guillaume, D., Andriamampihantona, J. 2014. (2014) Geological setting of the bastnaesite and monazite deposits in the Ambatofinandrahana area, central part of Madagascar: an overview.,Journal of African Earth Sciences,94,128-140

Richard, A., Mark A. Kendrick, Michel Cathelineau (2014) Noble gases (Ar, Kr, Xe) and halogens (Cl, Br, I) in fluid inclusions from the Athabasca Basin (Canada): Implications for unconformity-related U deposits Precambrian Research (Impact Factor: 4.44). 03/2014; DOI: 10.1016/j.precamres.2014.03.020

Rongier, G., Collon-Drouaillet, P. & Filipponi, M. (2014) Simulation of 3D karst conduits with an object-distance based method integrating geological knowledge. Geomorphology 217, 152–164 (2014)

Sevostianov, I. & Giraud, A. (2014) Generalization of Maxwell homogenization scheme for elastic material containing inhomogeneities of diverse shape. Int. J. Eng. Sci. 64, 23–36 (2013)

Sevostianov, I. & Giraud, A. (2014) On the Compliance Contribution Tensor for a Concave Superspherical Pore. Int. J. Fract. 177, 199–206 (2012)

Truche L., Bazaekinnaa E. F., Barrea G., Thomassot E., Berger G., Dubessy J., Robert P. (2014) The role of S−3 ion in thermochemical sulphate reduction: Geological and geochemical implications. Earth and Planetary Science Letters 396, 190–200 (2014)

Ulrich M., Munoz M., Guillot S., Cathelineau M., Picard C. Quesnel B., Boulvais P., Couteau C. (2014) Dissolution-precipitation processes governing the carbonation and silicification of the serpentinite sole of the New Caledonia ophiolite. Contrib. Mineral. Petrol. 167, 952 (2014)

Vigneresse, J-L., Truche, L. and Chattaraj P.K. (2014) Metals (copper) segregation in magmas. Lithos 208-209, 462-470

PRA2: Ore processes and phytomining

2013

Bani, A., Imeri, A., Echevarria, G., Pavlova, D., Reeves, R.D., Morel, J.L., Sulçe, S. 2013. Nickel Hyperaccumulation in the Serpentine Flora of Albania. Fresenius Environmental Bulletin. 22 (6), 1792-1801.

Chardot-Jacques, V., Calvaruso, C., Simon, B., Turpault, M.-P., Echevarria, G., Morel, J.L. 2013. Chrysotile Dissolution in the Rhizosphere of the Nickel Hyperaccumulator Leptoplax emarginata. Environmental Science & Technology 47 (6), 2612–2620.

2014

Bani, A., Echevarria, G., Montargès-Pelletier, E., Gjoka, F., Sulçe, S., Morel, J.L. (2014) Pedogenesis and nickel biogeochemistry in a typical Albanian ultramafic toposequence. Environmental Monitoring and Assessment 1–12 (2014). doi:10.1007/s10661-014-3709-6

Deng, T-H-B, Cloquet, C., Tang, Y-T, Sterckeman, T., Echevarria, G., Estrade, N., Morel, J.L., Qiu, R-L. (2014). Nickel and Zinc Isotope Fractionation in Hyperaccumulating and Non-accumulating Plants. Environ. Sci. Technol. 48, 11926-11933

Filippov, L. O., Severov, V. V. & Filippova, I. V. (2014) An overview of the beneficiation of iron ores via reverse cationic flotation. Int. J. Miner. Process. 127, 62–69 (2014)

Kanari N., Filippova I., Diot F., Mochón J., Ruiz-Bustinza I., Allain E. Yvon J. (2014) Utilization of a waste from titanium oxide industry for the synthesis of sodium ferrate by gas-solid reactions. Thermochim. Acta 575, 219–225 (2014)

Menad N., Kanari N., Save M. (2014) Recovery of high grade iron compounds from LD slag by enhanced magnetic separation techniques, International Journal of Mineral Processing,126,1-9

Lucisine, P., Echevarria, G., Sterckeman, T., Valance J., Rey, P., Benizri, E. (2014). Effect of hyperaccumulating plant cover composition and rhizosphere-associated bacteria on the efficiency of nickel extraction from soil. Applied Soil Ecology 81, 30–36 (2014)

Shahid, M. et al. (2014) EDTA-Enhanced Phytoremediation of Heavy Metals: A Review. Soil. Sediment. Contam. 23, 389–416

Zhang, X, Houzelot, V., Bani, A., Morel, J.L., Echevarria, G., Simonnot, M.O. (2014). Selection And Combustion Of Ni-Hyperaccumulators For The Phytomining Process. Int. J. Phytoremediat. 16, 1058–1072 (2014)

2015

Bani, A., Echevarria, G., Sulçe, S., Morel, J.L. (2015). Improving the agronomy of Alyssum murale for extensive phytomining: A five-year field study. International Journal of Phytoremediation 17 (2), 117-127

Bani, A., Zhang, X., Echevarria, G., Laubie, B., Benizri, E., Simonnot, M.O., Morel, J.L. (2015). The effect of plant density in nickel phytomining field experiments with Alyssum murale in Albania. Australian Journal of Botany, in press

Jiang, C-A., Wu, Q.T., Goudon, R., Echevarria, G., Morel, J.L. (2015). Effect of P supply on metal uptake by Ni-hyperaccumulator co-cropped with Lupinus albus. Australian Journal of Botany, in press

Rue, M., Vallance, J, Echevarria G., Rey, P. Benizri, E. (2015). Rhizosphere microbial communities under mono- or multispecies hyperaccumulator plant cover in a serpentine soil. Australian Journal of Botany, in press

Van der Ent, A., Baker, A., Reeves, R., Chaney, R., Anderson, C., Meech, J., Erskine, P., Simonnot, M-O., Vaughan, J., Morel, J-L., Echevarria, G., Fogliani, B., Qiu, R.L. and Mulligan, D. (2014) 'Agromining': farming for metals in the future? submitted to: Environmental Science and Technology on Dec. 12, 2014. Accepted (position paper co-authored with invited Prof. Alan Baker and Dr. Antony van der Ent)

PRA3 + 6: Metals and environment

2012

Ahmad, MA; Prelot, B; Razafitianamaharavo, A, Douillard, JM; Zajac, J; Dufour, F; Durupthy, O; Chaneac, C; Villieras, F (2012) Influence of Morphology and Crystallinity on Surface Reactivity of Nanosized Anatase TiO2 Studied by Adsorption Techniques. 1. The Use of Gaseous Molecular Probes. Journal Of Physical Chemistry C, 116, 24596- 24606. DOI: 10.1021/jp307707h

Barbaroux, R; Plasari, E; Mercier, G; Simonnot, MO; Morel, JL; Blais, JF (2012) A new process for nickel ammonium disulfate production from ash of the hyperaccumulating plant Alyssum murale. Science Of The Total Environment, 423, 111- 119. DOI: 10.1016/j.scitotenv.2012.01.063

Bartoli, F; Coinchelin, D; Robin, C; Echevarria, G (2012) Impact of active transport and transpiration on nickel and cadmium accumulation in the leaves of the Ni-hyperaccumulator Leptoplax emarginata: a biophysical approach. Plant And Soil, 350, 99- 115. DOI: 10.1007/s11104-011-0885-9

Bigorgne, E; Foucaud, L; Caillet, C; Giamberini, L; Nahmani, J; Thomas, F; Rodius, F (2012) Cellular and molecular responses of E. fetida coelomocytes exposed to TiO2 nanoparticles. Journal Of Nanoparticle Research, 14, - . DOI: 10.1007/s11051-012-0959-5

Bongoua-Devisme, AJ; Mustin, C; Berthelin, J (2012) Responses of Iron-Reducing Bacteria to Salinity and Organic Matter Amendment in Paddy Soils of Thailand. Pedosphere, 22, 375- 393.

Caland, F; Miron, S; Brie, D; Mustin, C (2012) a blind sparse approach for estimating constraint matrices in paralind data models. 2012 proceedings of the 20th european Signal Processing Conference (Eusipco), 839- 843.

Carbone, C; Marescotti, P; Lucchetti, G; Martinelli, A; Basso, R; Cauzid, J (2012) Migration of selected elements of environmental concern from unaltered pyrite-rich mineralizations to Fe-rich alteration crusts. JOURNAL OF GEOCHEMICAL EXPLORATION, 114, 109- 117. DOI: 10.1016/j.gexplo.2012.01.003

Coinchelin, D; Bartoli, F; Robin, C; Echevarria, G (2012) Ecophysiology of nickel phytoaccumulation: a simplified biophysical approach. Journal Of Experimental Botany, 63, 5815- 5827. DOI: 10.1093/jxb/ers230

Cornut, J; Clivot, H; Chauvet, E; Elger, A; Pagnout, C; Guerold, F (2012) Effect of acidification on leaf litter decomposition in benthic and hyporheic zones of woodland streams. Water Research, 46, 6430- 6444. DOI: 10.1016/j.watres.2012.09.023

Couleau, N; Techer, D; Pagnout, C; Jomini, S; Foucaud, L; Laval-Gilly, P; Falla, J; Bennasroune, A (2012) Hemocyte responses of Dreissena polymorpha following a short-term in vivo exposure to titanium dioxide nanoparticles: Preliminary investigations. Science Of The Total Environment, 438, 490- 497. DOI: 10.1016/j.scitotenv.2012.08.095

Duval, JFL; van Leeuwen, HP (2012) Rates of Ionic Reactions With Charged Nanoparticles In Aqueous Media. Journal Of Physical Chemistry A, 116, 6443- 6451. DOI : 10.1021/jp209488v

Foltete, AS; Masfaraud, JF; Ferard, JF; Cotelle, S (2012) Is there a relationship between early genotoxicity and life-history traits in Vicia faba exposed to cadmium-spiked soils?. Mutation Research-Genetic Toxicology And Environmental Mutagenesis, 747, 159- 163. DOI: 10.1016/j.mrgentox.2010.12.011

Gismondi, E; Cossu-Leguille, C; Beisel, JN (2012) Acanthocephalan parasites: help or burden in gammarid amphipods exposed to cadmium?. ECOTOXICOLOGY, 21, 1188- 1193. DOI: 10.1007/s10646-012-0873-8

Gismondi, E; Rigaud, T; Beisel, JN; Cossu-Leguille, C (2012) Effect of Multiple Parasitic Infections on the Tolerance to Pollutant Contamination. Plos One, 7, - . DOI: 10.1371/journal.pone.0041950

Gismondi, E; Beisel, JN; Cossu-Leguille, C (2012) Polymorphus Minutus Affects Antitoxic Responses of Gammarus Roeseli Exposed to Cadmium. Plos One, 7, - . DOI : 10.1371/journal.pone.0041475

Gismondi, E; Rigaud, T; Beisel, JN; Cossu-Leguille, C (2012) Microsporidia parasites disrupt the responses to cadmium exposure in a gammarid. Environmental Pollution, 160, 17- 23. DOI: 10.1016/j.envpol.2011.09.021

Hafeez, F; Spor, A; Breuil, MC; Schwartz, C; Martin-Laurent, F; Philippot, L (2012) Distribution of bacteria and nitrogen-cycling microbial communities along constructed Technosol depth-profiles. Journal Of Hazardous Materials, 231, 88- 97. DOI: 10.1016/j.jhazmat.2012.06.041

Immel, F, Renaut, J; Masfaraud, JF (2012) Physiological response and differential leaf proteome pattern in the Eurpean invasive Asteraceae Solidago canadensis colonizing a former cokery soil. Journal of Proteomics, 75, 1129-1143. DOI : 10.1016/j.jprot.2011.10.026

Jomini, S; Labille, J; Bauda, P; Pagnout, C (2012) Modifications of the bacterial reverse mutation test reveals mutagenicity of TiO2 nanoparticles and byproducts from a sunscreen TiO2-based nanocomposite. Toxicology Letters, 215, 54- 61. DOI: 10.1016/j.toxlet.2012.09.012

Krapf, MEM; Lartiges, BS; Merlin, C; Francius, G; Ghanbaja, J; Duval, JFL (2012) Polyethyleneimine-mediated flocculation of Shewanella oneidensis MR-1: Impacts of cell surface appendage and polymer concentration. Water Research, 46, 1838- 1846. DOI: 10.1016/j.watres.2011.12.061

Manusadzianas, L; Caillet, C; Fachetti, L; Gylyte, B; Grigutyte, R; Jurkoniene, S; Karitonas, R; Sadauskas, K; Thomas, F; Vitkus, R; Ferard, JF (2012) Toxicity of copper oxide nanoparticle suspensions to aquatic biota. Environmental Toxicology And Chemistry, 31, 108- 114. DOI: 10.1002/etc.715

Merlin, J; Duval, JFL (2012) Metal speciation in a complexing soft film layer: a theoretical dielectric relaxation study of coupled chemodynamic and electrodynamic interfacial processes. Physical Chemistry Chemical Physics, 14, 4491- 4504. DOI: 10.1039/c2cp23611g

Minguez, L; Buronfosse, T; Giamberini, L (2012) Different Host Exploitation Strategies in Two Zebra Mussel-Trematode Systems: Adjustments of Host Life History Traits. PLOS ONE, 7, - . DOI: 10.1371/journal.pone.0034029

Minguez, L; Boiche, A; Sroda, S; Mastitsky, S; Brule, N; Bouquerel, J; Giamberini, L (2012) Cross-effects of nickel contamination and parasitism on zebra mussel physiology. Ecotoxicology, 21, 538- 547. DOI: 10.1007/s10646-011-0814-y

Minguez, L; Buronfosse, T; Beisel, JN; Giamberini, L (2012) Parasitism can be a confounding factor in assessing the response of zebra mussels to water contamination. Environmental Pollution, 162, 234 - 240. DOI: 10.1016 / j.envpol.2011.11.005

Pagnout, C; Jomini, S; Dadhwal, M; Caillet, C; Thomas, F; Bauda, P (2012) Role of electrostatic interactions in the toxicity of titanium dioxide nanoparticles toward Escherichia coli. Colloids And Surfaces B-Biointerfaces, 92, 315- 321. DOI: 10.1016/j.colsurfb.2011.12.012

Palais, F; Dedourge-Geffard, O; Beaudon, A; Pain-Devin, S; Trapp, J; Geffard, O; Noury, P; Gourlay-France, C; Uher, E; Mouneyrac, C; Biagianti-Risbourg, S; Geffard, A (2012) One-year monitoring of core biomarker and digestive enzyme responses in transplanted zebra mussels (Dreissena polymorpha). Ecotoxicology, 21, 888-905. DOI: 10.1007/s10646-012-0851-1

Qiu, RL; Zhang, DD; Diao, ZH; Huang, XF; He, C; Morel, JL; Xiong, Y (2012) Visible light induced photocatalytic reduction of Cr(VI) over polymer-sensitized TiO2 and its synergism with phenol oxidation. Water Research, 46, 2299- 2306. DOI: 10.1016/j.watres.2012.01.046

Raous, S., Echevarria, G., Sterckeman, T., Hanna, K., Thomas, F., Martins, E.S., Becquer, T. 2013. Potentially toxic metals in ultramafic mining materials: identification of the main bearing and reactive phases. Geoderma. 192:111-119.

Rizvi, SH; Gauquelin, T; Gers, C; Guerold, F; Pagnout, C; Baldy, V (2012) Calcium-magnesium liming of acidified forested catchments: Effects on humus morphology and functioning. Applied Soil Ecology, 62, 81- 87. DOI: 10.1016/j.apsoil.2012.07.014

Sahli, L; Afri-Mehennaoui, FZ; El Okki, ME; Ferard, JF; Mehennaoui, S (2012) Assessment of sediment quality and pore water ecotoxicity in Kebir Rhumel basin (NE-Algeria): a combined approach. Water Science And Technology, 65, 393- 401. DOI: 10.2166/wst.2012.802

Sellami, R; Gharbi, F; Rejeb, S; Rejeb, MN; Henchi, B; Echevarria, G; Morel, JL (2012) Effects Of Nickel Hyperaccumulation On Physiological Characteristics Of Alyssum Murale Grown On Metal Contaminated Waste Amended Soil. International Journal Of Phytoremediation, 14, 609- 620. DOI: 10.1080/15226514.2011.619225

Sornom, P; Gismondi, E; Vellinger, C; Devin, S; Ferard, JF; Beisel, JN (2012) Effects of Sublethal Cadmium Exposure on Antipredator Behavioural and Antitoxic Responses in the Invasive Amphipod Dikerogammarus villosus. Plos One, 7, - . DOI: 10.1371/journal.pone.0042435

Tang, YT; Cloquet, C; Sterckeman, T; Echevarria, G; Carignan, J; Qiu, RL; Morel, JL (2012) Fractionation of Stable Zinc Isotopes in the Field-Grown Zinc Hyperaccumulator Noccaea caerulescens and the Zinc-Tolerant Plant Silene vulgaris. Environmental Science & Technology, 46, 9972- 9979. DOI: 10.1021/es3015056

Tang, YT; Deng, THB; Wu, QH; Wang, SZ; Qiu, RL; Wei, ZB; Guo, XF; Wu, QT; Lei, M; Chen, TB; Echevarria, G; Sterckeman, T; Simonnot, MO; Morel, JL (2012) Designing Cropping Systems for Metal-Contaminated Sites: A Review. Pedosphere, 22, 470- 488. DOI:

Thion, C; Cebron, A; Beguiristain, T; Leyval, C (2012) Long-term in situ dynamics of the fungal communities in a multi-contaminated soil are mainly driven by plants. Fems Microbiology Ecology, 82, 169- 181. DOI: 10.1111/j.1574-6941.2012.01414.x

Town, RM; Duval, JFL; Buffle, J; van Leeuwen, HP (2012) Chemodynamics of Metal Complexation by Natural Soft Colloids: Cu(II) Binding by Humic Acid. Journal Of Physical Chemistry A, 116, 6489- 6496. DOI: 10.1021/jp212226j

Van Heghe, L; Engstrom, E; Rodushkin, I; Cloquet, C; Vanhaecke, F (2012) Isotopic analysis of the metabolically relevant transition metals Cu, Fe and Zn in human blood from vegetarians and omnivores using multi-collector ICP-mass spectrometry. Journal Of Analytical Atomic Spectrometry, 27, 1327- 1334. DOI: 10.1039/c2ja30070b

Vellinger, C; Parant, M; Rousselle, P; Usseglio-Polatera, P (2012) Antagonistic toxicity of arsenate and cadmium in a freshwater amphipod (Gammarus pulex). Ecotoxicology, 21, 1817- 1827. DOI: 10.1007/s10646-012-0916-1

Vellinger, C; Felten, V; Sornom, P; Rousselle, P; Beisel, JN; Usseglio-Polatera, P (2012) Behavioural and Physiological Responses of Gammarus pulex Exposed to Cadmium and Arsenate at Three Temperatures: Individual and Combined Effects. Plos One, 7, - . DOI: 10.1371/journal.pone.0039153

Vellinger, C; Parant, M; Rousselle, P; Immel, F; Wagner, P; Usseglio-Polatera, P (2012) Comparison of arsenate and cadmium toxicity in a freshwater amphipod (Gammarus pulex). Environmental Pollution, 160, 66- 73. DOI: 10.1016/j.envpol.2011.09.002

2013

Aboudrar, W; Schwartz, C; Morel, JL; Boularbah, A (2013) Effect of nickel-resistant rhizosphere bacteria on the uptake of nickel by the hyperaccumulator Noccaea caerulescens under controlled conditions. Journal Of Soils And Sediments, 13, 501- 507. DOI: 10.1007/s11368-012-0614-x

Ahmad, MA; Prelot, B; Dufour, F; Durupthy, O; Razafitianamaharavo, A; Douillard, JM; Chaneac, C; Villieras, F; Zajac, J (2013) Influence of Morphology and Crystallinity on Surface Reactivity of Nanosized Anatase TiO2 Studied by Adsorption Techniques. 2. Solid-Liquid Interface. Journal Of Physical Chemistry C, 117, 4459- 4469. DOI: 10.1021/jp3077084

Amorosi C., Mustin C., Frache G., Bertani P., Toniazzo V. Ruch D., Ball V., Averous L. Michel M. (2012) Design of Flexible Free Standing Plasma Polymer−Based Films As Hosts for Enzyme Immobilization J. Phys. Chem. C, 2012, 116 (40), 21356–21365

Bongoua-Devisme, AJ; Cebron, A; Kassin, KE; Yoro, GR; Mustin, C; Berthelin, J (2013) Microbial Communities Involved in Fe Reduction and Mobility During Soil Organic Matter (SOM) Mineralization in Two Contrasted Paddy Soils. Geomicrobiology Journal, 30, 347- 361. DOI: 10.1080/01490451.2012.688928

Bourgeault, A; Ciffroy, P; Garnier, C; Cossu-Leguille, C; Masfaraud, JF; Charlatchka, R; Garnier, JM (2013) Speciation and bioavailability of dissolved copper in different freshwaters: Comparison of modelling, biological and chemical responses in aquatic mosses and gammarids. Science Of The Total Environment, 452, 68- 77. 81 DOI: 10.1016/j.scitotenv.2013.01.097

Caland F., Miron S., Brie D., Mustin C. (2012) A blind sparse approach for estimating constraint matrices in Paralind data models. European Signal Processing, EUSIPOO 2012, p 839 − 843

Cébron A., Arsene-Ploetze F., Bauda P., Bertin P.-N., Billard P., Carapito C., Devin S., Goulhen-Chollet F., Poirel J., Leyval C, 2013. Rapid impact of phenanthrene and arsenic on bacterial community structure and activities in polluted sand batches. Microbial Ecology. Accepted

Cébron A., Arsène-Ploetze F., Bauda P., Bertin P.N., Billard P., Carapito C., Devin S., Goulhen-Chollet F., Poirel J., Leyval C. (2013) Rapid impact of phenanthrene and arsenic on bacterial community structure and activities in polluted sand batches. Microb. Ecol. Accepté.

Chardot-Jacques, V; Calvaruso, C; Simon, B; Turpault, MP; Echevarria, G; Morel, JL (2013) Chrysotile Dissolution in the Rhizosphere of the Nickel Hyperaccumulator Leptoplax emarginata. Environmental Science & Technology, 47, 2612- 2620. DOI: 10.1021/es301229m

Chatellier, X; Grybos, M; Abdelmoula, M; Kemner, KM; Leppard, GG; Mustin, C; West, MM; Paktunc, D (2013) Immobilization of P by oxidation of Fe(II) ions leading

to nanoparticle formation and aggregation. Applied Geochemistry, 35, 325- 339. DOI: 10.1016/j.apgeochem.2013.04.019

Colas, F; Baudoin, JM; Danger, M; Usseglio-Polatera, P; Wagner, P; Devin, S (2013) Synergistic impacts of sediment contamination and dam presence on river functioning. Freshwater Biology, 58, 320- 336. DOI: 10.1111/fwb.12060

Devin S., Burgeot T., Giambérini L., Minguez L., Pain-Devin S, 2013. The Integrated Biomarker Response revisited: optimization to avoid misuse. Environmental Science And Pollution Research. sous presse

Dika, C; Ly-Chatain, MH; Francius, G; Duval, JFL; Gantzer, C (2013) Non-DLVO adhesion of F-specific RNA bacteriophages to abiotic surfaces: Importance of surface roughness, hydrophobic and electrostatic interactions. Colloids And Surfaces A-Physicochemical And Engineering Aspects, 435, 178- 187. DOI: 10.1016/j.colsurfa.2013.02.045

Duval, JFL (2013) Dynamics of metal uptake by charged biointerphases: bioavailability and bulk depletion. Physical Chemistry Chemical Physics, 15, 7873- 7888. DOI: 10.1039/c3cp00002h

El Khalil, H; Schwartz, C; El Hamiani, O; Kubiniok, J; Morel, JL; Boularbah, A (2013) Distribution of major elements and trace metals as indicators of technosolisation of urban and suburban soils. Journal Of Soils And Sediments, 13, 519- 530. DOI: 10.1007/s11368-012-0594-x

Esnault L., Libert M., Bildstein O., Mustin C., Marsal F., Jullien M. (2013) Impact of iron−reducing bacteria on the properties of argillites in the context of radioactive waste geological disposal. Applied Clay Science, 2013, 83–84,42–49

Fatombi, JK; Lartiges, B; Aminou, T; Barres, O; Caillet, C (2013) A natural coagulant protein from copra (Cocos nucifera): Isolation, characterization, and potential for water purification. Separation And Purification Technology, 116, 35- 40. DOI: 10.1016/j.seppur.2013.05.015

Garnier, J; Quantin, C; Guimaraes, EM; Vantelon, D; Montarges-Pelletier, E; Becquer, T (2013) Cr(VI) genesis and dynamics in Ferralsols developed from ultramafic rocks: The case of Niquelandia, Brazil. Geoderma, 193, 256- 264. DOI: 10.1016/j.geoderma.2012.08.031

Ghach W., Etienne M., Billard P., Jorand F.P.A., Walcarius A. (2013) Electrochemically-Assisted bacteria encapsulation in thin hybrid sol-gel film. J. Mater. Chem. B, 1, 1052-1059

Gismondi, E; Cossu-Leguille, C; Beisel, JN (2013) Do male and female gammarids defend themselves differently during chemical stress?. Aquatic Toxicology, 140, 432- 438. DOI: 10.1016/j.aquatox.2013.07.006

Huguier, P; Manier, N; Meline, C; Bauda, P; Pandard, P (2013) Improvement of the Caenorhabditis elegans growth and reproduction test to assess the ecotoxicity of soils and complex matrices. Environmental Toxicology And Chemistry, 32, 2100- 2108. DOI: 10.1002/etc.2282

Huot, H; Simonnot, MO; Marion, P; Yvon, J; De Donato, P; Morel, JL (2013) Characteristics and potential pedogenetic processes of a Technosol developing on iron industry deposits. Journal Of Soils And Sediments, 13, 555- 568. DOI: 10.1007/s11368-012-0513-1

Kassir, M; Roques-Carmes, T; Hamieh, T; Razafitianamaharavo, A; Barres, O; Toufaily, J; Villieras, F (2013) Surface modification of TiO2 nanoparticles with AHAPS aminosilane: distinction between physisorption and chemisorption. Adsorption-Journal Of The International Adsorption Society, 19, 1197- 1209. DOI: 10.1007/s10450-013-9555-y

Laporte, MA; Denaix, L; Pages, L; Sterckeman, T; Flenet, F; Dauguet, S; Nguyen, C (2013) Longitudinal variation in cadmium influx in intact first order lateral roots of sunflower (Helianthus annuus. L). Plant And Soil, 372, 581- 595. DOI: 10.1007/s11104-013-1756-3

Lorin-Nebel, C; Felten, V; Blondeau-Bidet, E; Grousset, E; Amilhat, E; Simon, G; Biagianti, S; Charmantier, G (2013) Individual and combined effects of copper and parasitism on osmoregulation in the European eel Anguilla anguilla. Aquatic Toxicology, 130, 41- 50. DOI: 10.1016/j.aquatox.2012.11.018

Lovy, L; Latt, D; Sterckeman, T (2013) Cadmium uptake and partitioning in the hyperaccumulator Noccaea caerulescens exposed to constant Cd concentrations throughout complete growth cycles. Plant And Soil, 362, 345- 354. DOI: 10.1007/s11104-012-1291-7

Minguez, L; Devin, S; Molloy, DP; Guerold, F; Giamberini, L (2013) Occurrence of zebra mussel parasites: Modelling according to contamination in France and the USA. Environmental Pollution, 176, 261- 266. DOI: 10.1016/j.envpol.2013.01.031

Pauget, B; Gimbert, F; Coeurdassier, M; Crini, N; Peres, G; Faure, O; Douay, F; Hitmi, A; Beguiristain, T; Alaphilippe, A; Guernion, M; Houot, S; Legras, M; Vian, JF; Hedde, M; Bispo, A; Grand, C; de Vaufleury, A (2013) Ranking field site management priorities according to their metal transfer to snails. Ecological Indicators, 29, 445- 454. DOI: 10.1016/j.ecolind.2013.01.012

Perez, KFB; Charlatchka, R; Ferard, JF (2013) Assessment of the LuminoTox leachate phase assay as a complement to the LuminoTox solid phase assay: Effect of fine particles in natural sediments. Chemosphere, 90, 1310- 1315. DOI: 10.1016/j.chemosphere.2012.09.078

Poirel J., Joulian C., Leyval C., Billard P. (2013) Arsenite-induced changes in abundance and expression of arsenite transporter and arsenite oxidase genes of a soil microbial community. Res. Microbiol. 164(5):457-65.

Raous, S; Echevarria, G; Sterckeman, T; Hanna, K; Thomas, F; Martins, ES; Becquer, T (2013) Potentially toxic metals in ultramafic mining materials: Identification of the main bearing and reactive phases. Geoderma, 192, 111- 119. DOI: 10.1016/j.geoderma.2012.08.017

Rotureau E, 2013. Analysis of metal speciation dynamics in clay minerals dispersion by Stripping Chronopotentiometry techniques Colloids and Surfaces a: Physicochemical and Engineering Aspects. in press

Tlili, S; Minguez, L; Giamberini, L; Geffard, A; Boussetta, H; Mouneyrac, C (2013) Assessment of the health status of Donax trunculus from the Gulf of Tunis using integrative biomarker indices. Ecological Indicators, 32, 285- 293. DOI: 10.1016/j.ecolind.2013.04.003

Town, RM; Buffle, J; Duval, JFL; van Leeuwen, HP (2013) Chemodynamics of Soft Charged Nanoparticles in Aquatic Media: Fundamental Concepts. Journal Of Physical Chemistry A, 117, 7643-7654. DOI: 10.1021/jp4044368

Vellinger, C; Gismondi, E; Felten, V; Rousselle, P; Mehennaoui, K; Parant, M; Usseglio-Polatera, P (2013) Single and combined effects of cadmium and arsenate in Gammarus pulex (Crustacea, Amphipoda): Understanding the links between physiological and behavioural responses. Aquatic Toxicology, 140, 106- 116. DOI: 10.1016/j.aquatox.2013.05.010

Vignati, DAL; Secrieru, D; Bogatova, YI; Dominik, J; Cereghino, R; Berlinsky, NA; Oaie, G; Szobotka, S; Stanica, A (2013) Trace element contamination in the arms of the Danube Delta (Romania/Ukraine): Current state of knowledge and future needs. Journal Of Environmental Management, 125, 169- 178. DOI: 10.1016/j.jenvman.2013.04.007

Zegeye A., Billard P., Mustin C. (2013) Bioreduction of Sb-substituted goethite: A mechanism for Sb mobilisation and bio-availability? Goldschmidt conference, August 25-30, Florence, Italy.

Zelano, I; Sivry, Y; Quantin, C; Gelabert, A; Tharaud, M; Jouvin, D; Montarges-Pelletier, E; Garnier, J; Pichon, R; Nowak, S; Miska, S; Abollino, O; Benedetti, MF (2013) Colloids and suspended particulate matters influence on Ni availability in surface waters of impacted ultramafic systems in Brazil. Colloids And Surfaces A-Physicochemical And Engineering Aspects, 435, 36- 47. DOI: 10.1016/j.colsurfa.2013.02.051

2014

Cebron A., Arsene-Ploetze F., Bauda P., Bertin P.-N., Billard P., Carapito C., Devin S., Goulhen-Chollet F., Poirel J., Leyval C. (2014) Rapid Impact Of Phenanthrene And Arsenic On Bacterial Community Structure And Activities In Sand Batches.Microbial Ecology 67, 129-144. [Doi]

Clivot H., Charmasson F., Felten V., Boudot J.-P., Guérold F., Danger M. (2014) Interactive Effects Of Aluminium And Phosphorus On Microbial Leaf Litter Processing In Acidified Streams: A Microcosm Approach.Environmental Pollution 186, 67-74. [Doi]

Colas, F., Vigneron, A., Felten, V. & Devin, S. (2014)The contribution of a niche-based approach to ecological risk assessment: Using macroinvertebrate species under multiple stressors. Environ. Pollut. 185, 24–34 , 8.

Custos, J.-M., Moyne, C., Treillon, T. & Sterckeman, T. (2014) Contribution of Cd-EDTA complexes to cadmium uptake by maize: a modelling approach. Plant Soil 374, 497–512 (2014).

Duval, J. F. L. & Rotureau, E. (2014) Dynamics of metal uptake by charged soft biointerphases: impacts of depletion, internalisation, adsorption and excretion. Phys. Chem. Chem. Phys. 16, 7401–7416 (2014).

Etique M., Jorand F.-P.-A., Zegeye A., Gregoire B., Despas C., Ruby C. (2014) Abiotic Process For Fe(Ii) Oxidation And Green Rust Mineralization Driven By A Heterotrophic Nitrate Reducing Bacteria (Klebsiella Mobilis).Environmental Science & Technolog 48, 3742-3751. [Doi]

Ferrari, B. J. D., Vignati, D. A. L. & Dominik, J. (2014) Bioaccumulation kinetics and effects of sediment-bound contaminants on chironomids in deep waters: new insights using a low-disturbance in situ system. Environ. Technol. 35, 456–469 (2014).

Funck, J. A. et al. (2014) Behavioural and physiological responses of Gammarus fossarum (Crustacea Amphipoda) exposed to silver. Aquat. Toxicol. 142, 73–84 (2013).

Gal, F. et al. (2014) Study of the environmental variability of gaseous emanations over a CO2 injection pilot Application to the French Pyrenean foreland. Int. J. Greenh. Gas Control 21, 177–190 (2014).

Garaud M, Trapp J, Devin S, Cossu-Leguille C, Pain-Devin S, Felten V, Giamberini L (2014). Multibiomarker assessment of cerium dioxide nanoparticle (nCeO2) sublethal effects on two freshwater invertebrates, Dreissenapolymorpha and Gammarus roeseli. Aquatic Toxicology 158 (2014) 63–74

Garcon, M., Chauvel, C., France-Lanord, C., Limonta, M. & Garzanti, E. (2014) Which minerals control the Nd-Hf-Sr-Pb isotopic compositions of river sediments? Chem. Geol. 364, 42–55 (2014).

Gonzalez, V., Vignati, D. A. L., Leyval, C. & Giamberini, L. (2014) Environmental fate and ecotoxicity of lanthanides: Are they a uniform group beyond chemistry? Environment International 71, 148–157 (2014).

Huot, H. et al. (2014) A Technosol as archives of organic matter related to past industrial activities. Sci. Total Environ. 487, 389–398 (2014).

Kamel, N. et al. (2014) Effects of increasing temperatures on biomarker responses and accumulation of hazardous substances in rope mussels (Mytilus galioprovincialis) from Bizerte lagoon. Environ. Sci. Pollut. Res. 21, 6108–6123 (2014).

Montargès-Pelletier E., Duriez C., Ghanbaja J., Jeanneau L., Falkenberg G., Michot L.-J. (2014) Microscale Investigations Of The Fate Of Heavy Metals Associated To Iron-Bearing Particles In A Highly Polluted Stream.Environmental Science And Pollution Research 21, 2744-2760. [Doi]

Oulkadi, D. et al. (2014) Bioweathering of nontronite colloids in hybrid silica gel: implications for iron mobilization. J. Appl. Microbiol. 116, 325–334 (2014).

Pain-Devin S, C. Cossu-Leguille, A. Geffard,L. Giambérini, T. Jouenne,L. Minguez, B. Naudin, M. Parant, F. Rodius, P. Rousselle, K. Tarnowska, C. Daguin-Thiébaut, F. Viard, S. Devin. (2014) Towards a better understanding of biomarker response in field survey: A case study in eight populations of zebra mussels Aquatic Toxicology155: 52-61. DOI: 10.1016/j.aquatox.2014.06.008

Pellerin, J., Lévy, B., Caumon, G. & Botella, A. (2014)Automatic surface remeshing of 3D structural models at specified resolution: A method based on Voronoi diagrams. Computers & Geosciences 62, 103–116

Pey, B. et al. (2014) Technosol composition affects Lumbricus terrestris surface cast composition and production. Ecol. Eng. 67, 238–247 (2014).

Polyakov, P. D. & Duval, J. F. L. (2014) Speciation dynamics of metals in dispersion of nanoparticles with discrete distribution of charged binding sites. Phys. Chem. Chem. Phys. 16, 1999–2010 (2014).

Raous, S. et al. (2014) Potentially toxic metals in ultramafic mining materials: Identification of the main bearing and reactive phases. Geoderma 192, 111–119 (2013).

Rees, F., Simonnot, M. O. & Morel, J. (2014) L. Short-term effects of biochar on soil heavy metal mobility are controlled by intra-particle diffusion and soil pH increase. Eur. J. Soil Sci. 65, 149–161 (2014).

Rotureau, E. (2014) Analysis of metal speciation dynamics in clay minerals dispersion by stripping chronopotentiometry techniques. Colloid Surf. A-Physicochem. Eng. Asp. 441, 291–297 (2014).

Shi-Zhong, W. et al. (2014) A Fuzzy-based Methodology for an Aggregative Environmental Risk Assessment of Restored Soil. Pedosphere 24, 220–231 (2014). 10.

Funded research internships at masters level

(Masters funded by Labex RESSOURCES21 in blue)

Priority research actions	Name	First name	Supervisors	Laboratory	Title
PRA1	AOUA	Mariette	M. Cathelineau	Georessources	Les éléments en traces dans l'apatite (U, terres rares) comme marqueurs des conditions de formation,
PRA1	FORTUNATO	Jolanda	M. Cuney, F. Malartre	Georessources	Les calcretes de Trekkopje
PRA1	FROMAGEOT	Augustin	M. C. Boiron	Georessources	Or primaire vs or secondaire dans les gisements associés aux shear zones de St Elie (Guyane)
PRA1	**BELISSONT**	**Rémi**	**M. C. Boiron, B. Luais**	**Georessources**	**Traçage élémentaire et isotopique du Germanium dans les sulfures (LA ICP-MS et MC-ICPMS)**
PRA1	MAGOTT	Rémi	M. Cuney	Georessources	Etude des inclusions fluides et pétrologie d'échantillon du socle du Bassin de Thelon au Canada
PRA1	DROUET	Sylvain	M. Cuney	Georessources	Fractionnements de l'uranium à la limite de l'Archéen-Protérozoïque. L'exemple du Nord Québec
PRA1	**HARLAUX**	**Matthieu**	**M. Cathelineau**	**Georessources**	**Chronologie relative des transferts et des minéralisations en Ni de Nouvelle Calédonie**
PRA1	ESCARIO PEREZ	Sofia	M. C. Boiron	Georessources	Origine des minéralisations en fluorine de l'Est de la France
PRA1	GAILLARD	Nicolas	M. Cathelineau, M.C. Boiron	Georessources	Les concentrations en or et uranium dans les niveaux de calc-silicates du Nord de la Finlande (Rovaniemi)
PRA1	FONTAINE	Arnaud	A.S. André - Mayer	Georessources	Gold occurrence in the Kioka prospect, Burkina Faso
PRA5	**LAFRANCE**	**Noémie**	**R.Giot**	**GeoRessources**	**Interprétation d'essais de surcarottage avec prise en compte de l'anistropie plastique**
PRA5	MAILLOT	Jonathan	G. Caumon	GeoRessources	Simplification de réseaux de failles 3D sous contraintes
PRA5	CHAUVIN	Benjamin	G. Caumon	GeoRessources	Comparaison de Modèles structuraux pour le problème inverse
PRA3	MEHENNAOUI	Kahina	C. Cossu-Leguille & V. Felten	Liec	Effet des nanoparticules sur des réponses sub-individuelles et individuelles de Gammarus sp. (Crustacea Amphipoda): approche en mésocosme.
PRA3	ZANUTINI	Cyrielle	L. Giamberini	Liec	Effet de nanoparticules chez la moule d'eau douce Dreissena polymorpha. Utilisation d'une batterie de biomarqueurs (systèmes de défense, filtration, osmorégulation…)
PRA3	MARCHAL	Benoit	S. Pain-Devin	Liec	Effet de nanoparticules chez la moule d'eau douce Dreissena polymorpha. Etude des systèmes MXR.
PRA5	FRABOULET	Jean Gabriel	J. J. Royer	GeoRessources	3D modeling of a copper mine in Polish Kupferschiefer
PRA2	PERROTON	Arthur	L. Filippov	Anciennement LEM	Flottation des fines particules considérées comme des impuretés au sein de kaolins».
PRA2	ALLAIS	Bastien	L. Filippov	Anciennement LEM	Développement d'un procédé de flottation pour les sulfures hydrothermaux sous-marins
PRA2	GIGON	Joséphine	L. Filippov	Anciennement LEM	Caractérisation et valorisation des phosphates du Karatau
PRA2	LAPELOU-SAM-BOUMA	Crépin	L. Filippov	Anciennement LEM	Etude morphologique des associations minérales dans le faciès calcrête du gisement de Trekkopje en vue d'une séparation physique et/ou chimique.
PRA2	GENEVOIS	Nicolas	T. Sterckeman	LSE	Variation de l'hyperaccumulation du Cd pour différentes accessions de Noccaea caerulescens
PRA2	LUCISINE	Pierre	E. Benizri	LSE	Comparaison de l'efficience d'extraction du Ni entre couverts plurispécifique et monospécifique de plantes hyperaccumulatrices et caractérisation de la communauté bactérienne fonctionnelle
PRA2	MOUCHON	Liane Clarisse	G.Echevarria	LSE	Disponibilité du nickel dans les sols ultramafiques de Nouvelle Calédonie
PRA1	GUERGOUZ	Célia	O.Vanderhaeghe	Georessources	Étude géodynamique sur la chaine hercynienne pour la compréhension de la mise en place d›une minéralisation en Ni-Cu-PGE de la zone d›Ivrea en Italie.

PRA1	**ROMEO**	**Benjamin**	**M. CUNEY, C. MARIGNAC**	**Georessources**	**Etude de la minéralisation à quartz-wolframite de Puy-les-Vignes (Limousin)**
PRA1	MAWARABA	Keita	J. Cauzid	Georessources	Développement des capacités d'analyse par microthermométrie des inclusions fluides piégées dans les minéraux opaques
PRA1	**MASSEI**	**Fréderic**	**M. Cathelineau, G. Caumon, R. Mosser-Ruck**	**Georessources**	**Garnierite de Nouvelle Calédonie : étude de la substitution Ni-Mg dans les kérolites (talc-like) et les serpentines**
PRA1	FREMONT	François	O.Vanderhaeghe	Georessources	Minéralisations d'Uranium en Zambie
PRA1	FERRAINA	Clémént	A. Eglinger, A. Tarantola	Georessources	Etude pétrographique et géochimique des inclusions fluides de minéralisations uranifères, Copperbelt, Zambie.
PRA1	LEBRUN	Charline	M. Jebrak	Georessources et UQUAM	Étude de la minéralisation en antimoine et de sa distribution dans un gisement d'or orogénique : cas de la mine Lapa, ceinture de roches vertes de l'Abitibi
PRA1	SCHEFFER	Christophe	O.Vanderhaeghe, A. Tarantola	Georessources	Reconstruction des paléocirculations et géochimie des paléofluides dans la région de Lavrion (Grèce) : Implications géodynamiques et métallogéniques.
PRA5	VAUTRIN	Noémie	F. Golfier	Georessources	Modélisation à l'échelle du pore des processus biochimiques
PRA5	**CHRETIEN**	**Alexis**	**F. Golfier**	**Georessources**	**Modélisation du transport réactif en milieu fracturé - Mise en œuvre sous HYTEC**
PRA1	**GROULIER**	**Pierre-Arthur**	**A.S. André-Mayer, D. Ohnenstetter.**	**Georessources**	**Le système minéralisé en Nb-Ta-REE du gîte associé à l'intrusion alcaline de CREVIER (province du Grenville, Québec)**
PRA3	PETIT	Elisabeth	M. Parant, C. Cossu Leguille	LIEC	Impact cellaire d'un agent de contraste au Gadolinium sur des cellules humaines en culture
PRA3	SCHEFFER	Allisson	S. Pain-Devin	LIEC	Impact environnemental des nanoparticules manufacturées. Effet à l'échelle populationnelle sur Gammarus roeseli.
PRA3	URIARTE	Maxime	L. Giamberini	LIEC	Impact environnemental des nanoparticules manufacturées. Effet à l'échelle individuelle sur Gammarus roeseli et polex.
PRA3	BERTRAND	Carole	L. Giamberini	LIEC	Effet génotoxique de nanoparticules chez les bivalves
PRA2	NGUEGANG	Blandine	L. Filippov, B. Orberger	Georessources	Concentration des carbonates de Mn par floculation hydrophobique-magnétique'
PRA2	BOUTEILLER	Anne- Sophie	L. Filippov,	Georessources	Caractérisation des particules fines de minerais uranifères de type calcrête en vue d'un procédé de traitement minéralurgique
PRA2	STAINE	Marion	L. Filippov	Georessources	Développement des méthodes de flottation différentielle des fines de quartz à partir des résidus industriels (kaolins)
PRA1	BOUDOIRE	Guillaume	L. France	CRPG	Nouvelles contraintes sur lle système magmatique de l'Oldoinyo Lengaï (Tanzanie)
PRA1	**PELISSIER**	**Benjamin**	**M. Ohnenstetter**	**Georessources**	**Mobilité des éléments traces incompatibles, en particulier des terres rares, du faciès granulite aux conditions épithermales et supergènes**
PRA1	**MARTZ**	**Pierre**	**M. Cathelineau, M-C Boiron**	**Georessources**	**Distribution des porteurs de Terres rares dans les minerais à eudyalite- Application au gisement de Norra Karr**
PRA1	**JACQUINET**	**Damien**	**M. Cathelineau, L. Truche, F. Golfier**	**Georessources**	**Formation des minéralisations saprolitiques en Nouvelle Calédonie : minéralogie, séquence paragénétique et modélisation du transfert réactif**
PRA1	**LEGROS**	**Hélène**	**M. Cuney, J. Mercadier, C. Marignac**	**Georessources**	**Transition magmatique-hydrothermale dans les gisements géants de tungstène et Nb-Ta du SE de la Chine**
PRA1	**TEXIER**	**Clément**	**A. Tarantola, J. Cauzid**	**Georessources**	**Minéralisations BPGC de la Montagne Noire. Structure, parageneses, circulations fluides et datation**
PRA1	SCHNEIDER	Thomas	J. Cauzid	Georessources	Cathodoluminescence sur sphalérite

PRA1	DE RUNZ	Thibault	A-S. André, J. Bonnet	Georessources	Distribution des éléments traces Ga Cd et In dans le VMS de La-Ronde: étude minéralogique, séquence paragénétique et dosage des éléments traces.
PRA1	TURLIN	François	A-S. André, O. Vanderhaeghe	Georessources	Contexte tectonique des minéralisations cuprifères du gisement de Lumwana dans la ceinture Pan-africaine du Lufilien, Zambie
PRA1	BARRE	Guillaume	L. Truche	Georessources	Thermo-réduction des sulfates dans les MVT: mesures cinétiques et isotopiques
PRA1	WALTERSPERGER	Quentin	J. Mercadier	Georessources	Détermination des compositions en éléments majeurs, en éléments traces et en métaux dans les fluides minéralisateurs des gisements d'uranium de type discordance des bassins du Thélon (Canada) et de Kombolgie (Australie)
PRA1	MOUSSA	Karim	M. Mamane, M. Cathelineau	Georessources	Processus d'altération autour de la faille d'Arlit dans la formation permienne de l'Izégouaude sur le secteur de Tamgak (bassin de Tim Mersoï, Niger)
PRA5	**DESCREUX**	**P.I**	**L. Scholtès A-J. Tinet**	**Georessources**	**Modélisation couplée des processus hydromécaniques à l'échelle des grains**
PRA3	NGUYEN	V-X	S. Devin, J. ANDREI	LIEC	Variabilité inter-populationnelle de la sensibilité de Gammares aux nanoparticules d'argent : approche biochimique et fonctionnelle
PRA3	KOEHLE-DIVO	Vanessa	L. Giamberini, C. Pagnout, C. Bojic, M. Garaud, D. Vignati	LIEC	Effets écotoxicologues de nanoparticules d'argent (Influence de la taille, et de l'enrobage)
PRA3	DAMMOUS	Mirella	Elise Rotureau, J.P Pinheiro	LIEC	Spéciation des métaux trivalents dans les eaux naturelles: Étude des interactions entre Indium et acides humiques
PRA3	MBA EKOMO	Vitalys	M. Fillela	LIEC	La calibration de différentes méthodes d'analyse de la matière organique dissoute dans des eaux naturelles ou des milieux d'exposition en écotoxicologie
PRA2	DURAND	Alexis	Emile Benizri	LSE	Bioremédiation associée à la phytoremédiation d'un sol naturellement riche en nickel
PRA3	TIMBO	Mamadou Boubacar	Sophie Leguedois	LSE	Analyse environnementale du procédé de construction de sol
PRA3	POTET	Marine	S Pain Devi/L Giamberini	LIEC	Effets de nanoparticules sur l'activité MXR chez Dreissena et Gammarus sp

Year	Priority research actions	Name	Origin	Supervisor	Laboratory	Title of the project
2012-2015	PRA1&PRA4	Rémi BELISSONT	France	Marie-Christine Boiron & Béatrice Luais	GeoRessources	Cycle du germanium et métaux stratégiques associés dans les concentrations métalliques : traçage élémentaire, isotopique et approche expérimentale
2012-2015	PRA5	Maxime FAIVRE	France	Fabrice Golfier & Richard Giot	GeoRessources	Modélisation du comportement hydrogéomécanique d'un réseau de failles sous l'effet des variations de l'état de contrainte
2012-2015	PRA3&PRA4	Jennifer ANDREI	France	Sandrine Pain Devin François Guerold	LIEC	Effets des nanoparticules manufacturés sur les invertébrés d'eau douce et leurs fonctions au sein des écosystèmes
2013-2016	PRA1&PRA4	Matthieu HARLAUX	France	Michel Cuney & Christian Marignac	GeoRessources	Les minéralisations tardi-orogéniques varisques à tungstène et métaux associés (Sn, Nb, Ta) : source des fluides et des métaux, relations avec les évènements tectonomagmatiques fini-carbonifères sur l'exemple de gisements du massif central français
2013-2016	PRA1&PRA4	Gaelle MOLLEX	France	Lydéric France	CRPG	Génèse, évolution et altération des magmas carbonatitiques Recrutement doctorante
2013-2014	PRA6	Damien PARRELLO	France	Christian Mustin	LIEC	Identification à l'aide de bactéries génétiquement modifiées émettant des signaux luminescents (biocenseurs) les paramètres biotiques et abiotiques induisant la mobilisation de métaux stratégiques comme le nickel (fin de thèse 3 mois)
2013-2016	PRA3	Emilie PERRAT	France	Marc Parant & Cossu-Leguille	LIEC	Impacts environnementaux des agents de contraste au Gadolinium : situation locale, approches cellulaires et in vivo
2014	PRA1&PRA5	Pablo MEJIA-HERRERA	Colombie	Jean-Jacques Royer	CRPG	Apport de la modélisation 3D et de la restauration structurale dans la compréhension de gisements de matières premières minérales (fin de thèse 6 mois)
2014-2017	PRA1&PRA5	Andrey MYAGKIY,	Russia	Fabrice Golfier, Laurent Truche	GeoRessources	Formation des minéralisation de Ni saprolitiques en Nouvelle Calédonie: Dynamique des transferts de métaux et modélisation couplée

Year	Priority research actions	Name	Origin	Supervisor	Laboratory	Title of the project	After LABEX
2012-2013	PRA4&PRA6	Asfaw ZEGEYE	Ethiopia	Dr Christian Mustin	LIEC	Bio-Reduction of Metal-substituted Iron Oxides: A Mechanism for Metal Remobilization?	CR2 - CNRS
2012-2013	PRA1	Marc ULRICH	France	Dr Michel Cathelineau	GeoRessources	Enrichissement en Scandium des profils latéritiques de Nouvelle-Calédonie : Origine, spéciation et potentiel d'exploitation.	MCF - Université de STRASBOURG
2012-2013	PRA4	Nicolas ESTRADE	France	Dr Chistophe Cloquet & Dr Thibault Sterckeman	CRPG	Optimizing nickel phytomining: use of isotope fractionation to better understand soil to hyperaccumulating plant transfers	Post-doctorat - CANADA
2012-2014	PRA3	Véronica GONZALES ANDRES	Spain	Pr Laure Giamberini & Dr Corinne Leyval	LIEC	Transfert et dissémination des éléments rares et des terres rares dans l'écosphère : mécanismes, impact écotoxicologique et stratégies de remédiation	Université d'Almeria
2014-2015	PRA2	Antony VAN DER ENT	Australia	Pr Jean Louis Morel & Pr Guillaume Echevarria	LSE	Agromining of Ni in tropical Ni mine environments	
2015	PRA4	Ritech MISHRA	Inde	Dr Marc Chaussidon	CRPG	Développements de mesures isotopiques de haute précision avec les sondes ioniques ims 1270 et ims 1280HR2. (fin de post-doc 6 mois)	NASA Johnson space centre, Houston
2015	PRA1	Afifé EL KORH	Switzerland	Dr Etienne Deloule, Dr Marie-Christine BOIRON, Dr Béatrice LUAIS	CRPG	Mobility of metallic trace elements in the Limousin ophiolite massifs : implication for fluid-rock interactions signatures and concentration processes- (fin de Post-doc 5 mois)	
2014-2015	PRA1/PRA4	Thoms BOULESTEIX	France	C FRANCE-LANORD/M. CATHELINEAU/P ROBERT	CRPG/GeoRessources	Laboratoire Potassium – Argon : datation des argiles dans les bassins sédimentaires et les systèmes hydrothermaux pour l'amélioration des modèles génétiques des ressources métalliques- Post-do	

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ENVIRONMENT – ECOTOXICOLOGY - ECOSYSTEMS

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The program is designed to focus on environmental management and engineering dealing with the following major aspects: global changes, diagnosis and management of environmental hazards, effects of anthropic disturbance on organisms, populations, community and ecosystems, biodiversity erosion, preventing and treatment of contaminent, aquatic resources management and protection of the natural environment.

SUSTAINABLE DEVELOPMENT ENGINEERING

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The course leads to train specialists able to manage the requirements about sustainable development, environmental processes and hazard control during the conception and use of industrial systems. The developed skills and knowledge are focusing on (i) regulation and standard regarding safety, occupational health and environmental issues, (ii) process engineering, (iii) methods and systems of monitoring and diagnosis, (iv) sustainable management and treatment of contaminated soils, (vi) ecological engineering of urban environments, ...

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MASTERS

GEOSCIENCES: PLANETS, RESOURCES, ENVIRONMENT

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The goal of this Master level education program is to train geoscientists with a wide scope of skills and knowledge with the ambition to face challenges of the 21st century ranging from fundamental questions (Solar system origin, Planetary and Earth differentiation, lithospheric transferts, soil formation...) to industrial and societal issues (mineral exploration, 3D-4D modeling, mining exploitation and mining project, ore processing, soil, water and environment management and sustainable development). Five specializations are proposed inside the Master program : 1. Earth, 2. Mineral resources, 3. Oil geosciences and reservoir engineering, 4. Soil, water and environment and 5. Water resources, management and development.

ERASMUS MUNDUS EMERALD

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The program is designed to focus on the following major aspects of georesources engineering: characterisation, processing, modelling and management. Focussing on industrial needs, bridge the gap between georesources and advanced processing technologies, give students an in-depth knowledge of mineral resources, co-products, and most recent and modern techniques of processing, promote an effective and environmentally friendly management, these are some of Emerald goals.

MINERAL RAW MATERIALS, RISK ENGINEERING AND MANAGEMENT

www.mines-nancy.univ-lorraine.fr
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Philippe MARION - philippe.marion@univ-lorraine.fr
Secretary: anne.galausiaux@univ-lorraine.fr

The course leads to a Professional Master's Degree in Mines and Quarries. It covers the whole of the mineral mining cycle, from the exploration of deposits to the processing and recycling of mining waste, along with mining methods. Strong emphasis is placed on human risk prevention and environmental protection. The course includes visits of industrial sites and fieldwork.

SUBTERRANEAN RESERVOIRS OF ENERGY: HYDRODYNAMICS - GEOLOGY – MODELLING

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Antonio PEREIRA - antonio.pereirauniv-lorraine.fr
Secretary : sandie.fantin@univ-lorraine.fr

The objective of this Master program is to train specialists for combined engineering and scientific careers in:
- Conventional energy resources: oil, gas, uranium leaching
- Non-conventional geoenergies: bitumen, shale gas, gas hydrates, coal-bed methane, geothermy; tight gas reservoirs
- Underground storages of natural gas, CO2, hydrogen, electricity
- Hydro-thermodynamics of exploitation of different types of subterranean energy reservoirs
- Engineering methods of predicting, controlling, modelling, and optimising recovery scenarios
- Simulation and 3D-modelling of reservoir geological structures and reservoir dynamic processes
- Reservoir geology and geophysics.

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INSTITUT MINES TÉLÉCOM :

Created in March 2012, the "Institut Mines-Télécom" is one of the leading Higher Education and Research strengths in engineering in France with 12,000 students and 1,700 PhD students. Mines Nancy is a strategic partner of the Institute and the National School of Geology is one of the associated schools involved in this public institution.
www.mines-telecom.fr

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— 3 year programme leading to a Master's degree in Engineering
— Project-based education
— From Semester 5: Introduction into Business Management and Economics, Foreign languages & cultures, Humanities, Project Management
— Minimum 9-month work experience and 8 weeks abroad (internships or studies) throughout the curriculum
— Last year « à la carte »: Possibility to study abroad (exchange or master's programmes) among others

> SCIENTIFIC DEPARTMENTS AND RESEARCH PARTNERSHIPS

Laboratories	Academic departements	Majors
Géoressources	Geo-engineering	Geo-engineering
Laboratoire Lorrain de Recherche en Informatique et ses Applications (LORIA)	Computer Science and IT	— Architecture of secure systems — Organization of information and process
Institut Elie Cartan	Applied Mathematics and Industrial Engineering	— Decision and Production Engineering — Mathematical Engineering
Laboratoire d'Energétique et de Mécanique Théorique Appliquée (LEMTA)	Energy : Production, Transformation	Energy : Production, Transformation
Institut Jean Lamour	• Materials • Environment, energy and Process Engineering	— Functional Materials — Structural Materials — Energy and Environmental Engineering of Industrial Systems

> EDUCATION
— Education in science and engineering,
— Electives (ex: science, humanity, ARTEM working groups),
— Education in languages and foreign cultures.

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ENSG
GEOSCIENCES & ENGINEERING

NANCY: A MAJOR SCIENTIFIC AND GEO-ENGINEERING CENTER IN FRANCE
Nancy is the centre of one of the largest and most dynamic scientific communities in geosciences and environmental sciences in France. ENSG is associated with 4 research laboratories involving about 500 people.

Domains of excellence are fluid mechanics, soil mechanics, rock mechanics, ore geology, ore processing, CO_2 sequestration and storage, geochemistry and cosmochemistry, geodynamics, applied mineralogy, 3&4D geomodeling, soil and environmental sciences, continental surface sciences. The CRPG is one of the few laboratories outside the U.S. accredited by NASA to receive extra-terrestrial samples.

A UNIQUE CONCEPT
For more than 100 years the ENSG (École National Supérieure de Géologie) has developped the concept of the Engineer-Geologist. We produce top level Masters graduates, with a unique combination of expertise in geosciences, maths and engineering sciences.

All students complete the first 3 semesters of basic geosciences and engineering sciences including 5 weeks of geological field work. During the final two semesters, students specialize in one of the following domains: petroleum geology, geomodeling, civil engineering and geotechnics, ore processing and mining geology, water supply, or environment. The last semester consists of a 6 month internship in companies. A high level in English is a mandatory requirement for graduation.

At present ENSG respects parity as out of 120 students graduate each year half of whom are women.

ALUMNI
ENSG Graduates have reached the highest levels in major national and international companies. Outstanding alumni include Thierry Pilenko (CEO Technip), Manoelle Lepoutre (Senior Vice President Top Executive careers and management at Total), Olivier Peyret (Vice President New Businesses at Schlumberger), Philippe Vecten (delegate CEO manganese division Eramet)...

ENSG Graduates with international research careers include Edouard Bard (Climatology, Académie des Sciences, Paris), Christophe Sotin (Planetology, Distinguished Visiting Scientist in JPL-Caltech), Nicolas Dauphas (Cosmochemistry, Univ. Chicago)...

The ENSG is also famous for being the birthplace of the Gocad geomodeling software, created by Pr. JL Mallet more than 20 years ago.

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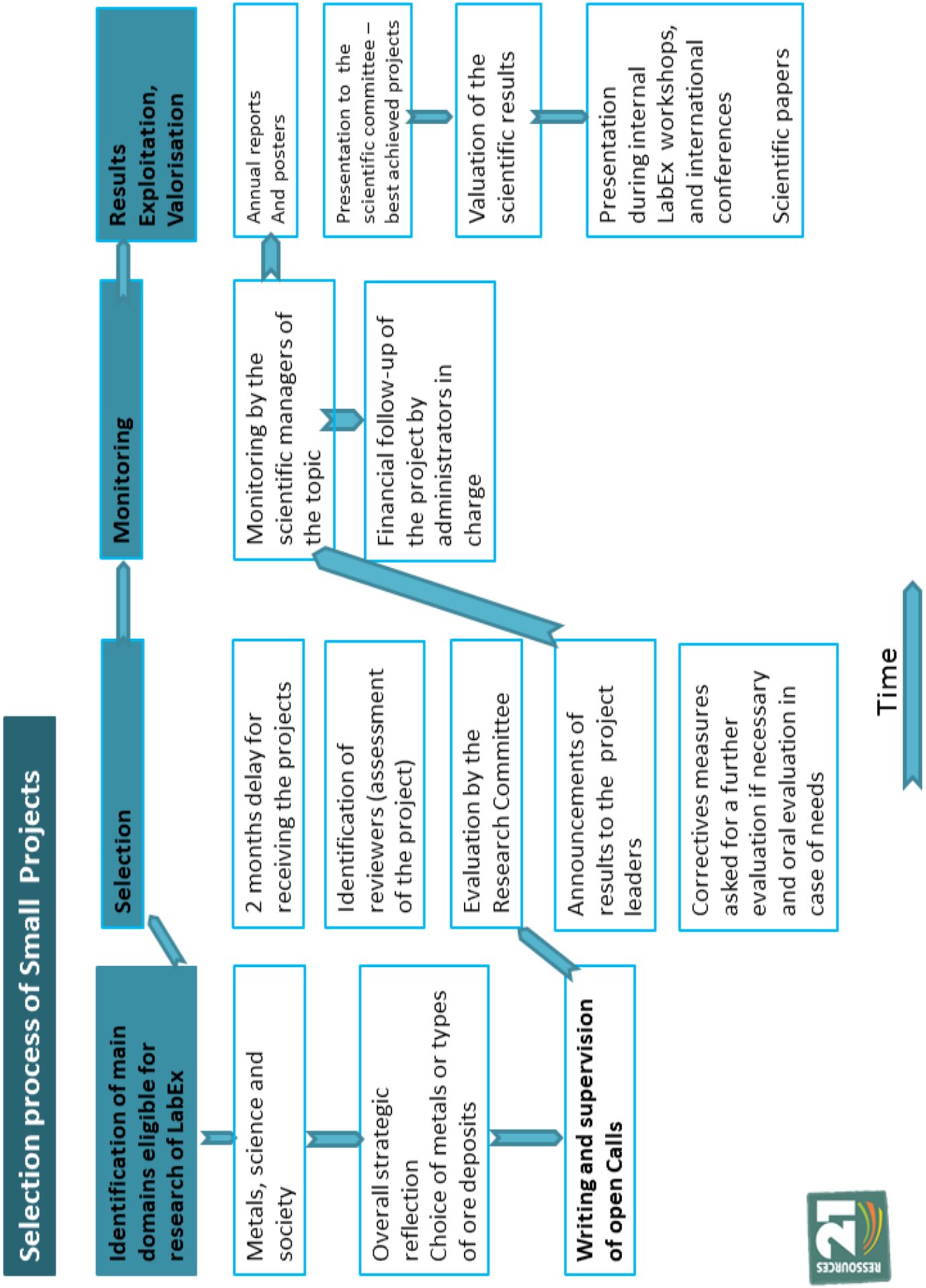
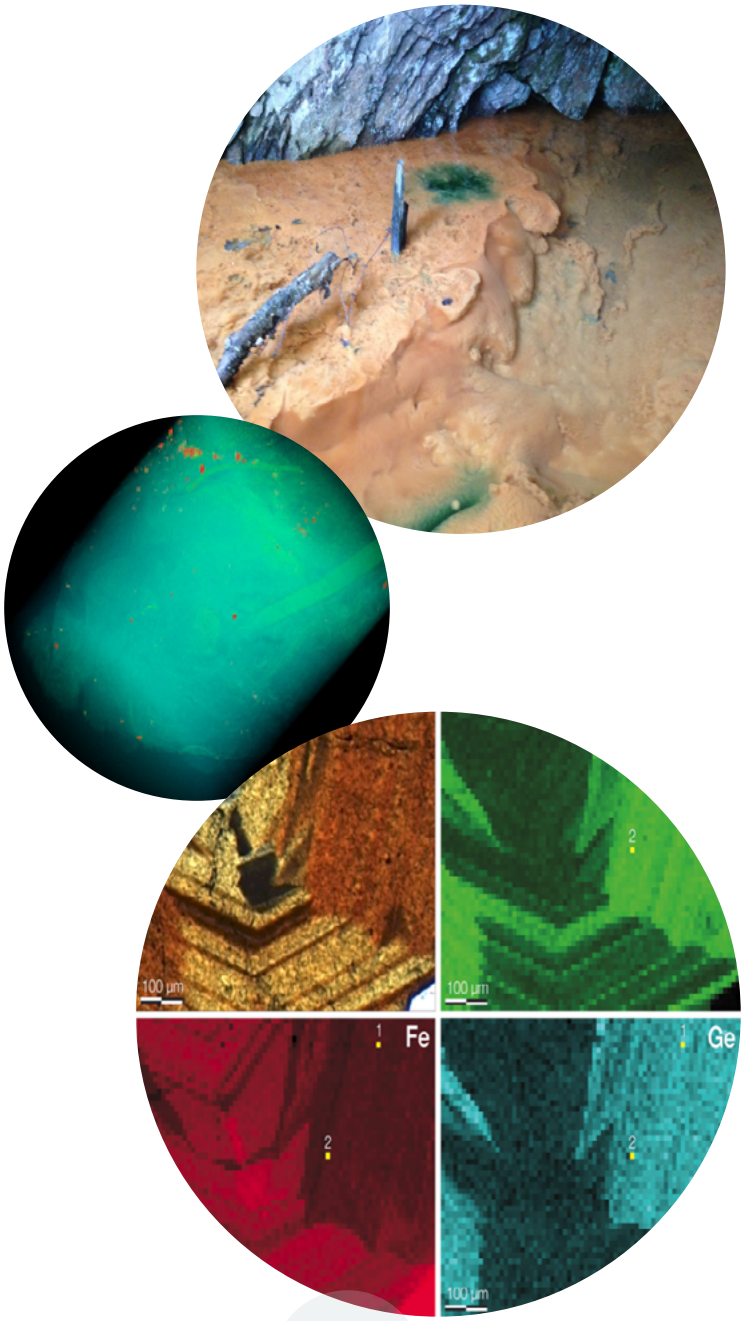
Procedure for short projects selection



Scientific results posters (in french)



Les minéralisations tardi-varisques à métaux rares (W, Sn, Nb, Ta) : exemple du gisement de Puy-les-Vignes (Massif Central Français)

Matthieu Harlaux, Christian Marignac, Michel Cuney, Julien Mercadier

UMR 7359 GeoRessources, Equipe Ressources Minérales

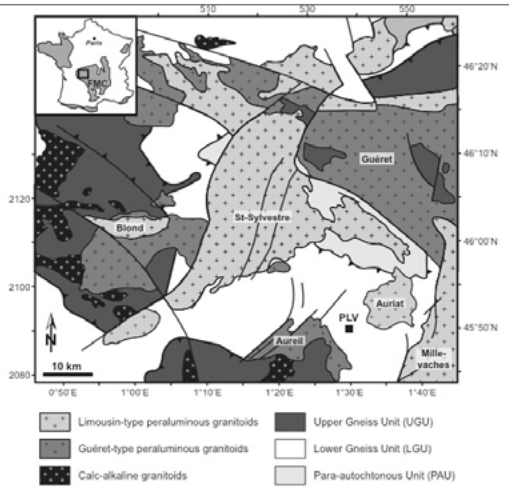
AXE Scientifique : 1

Enjeux scientifiques

Les métaux rares (W, Nb, Ta, Li, Be, Sn) font partie des « ressources minérales critiques » du XXI^{ème} siècle et représentent un enjeu majeur pour l'Europe en terme d'approvisionnement pour des applications de haute technologie. Le Massif Central Français, situé au cœur de la Chaîne Varisque ouest-européenne, possède un potentiel géologique de plus de 40 kt WO_3, essentiellement sous forme de wolframite $(Fe,Mn)WO_4$. Les objectifs de ce projet sont: 1) dater le ou les épisode(s) minéralisateur(s); 2) identifier la source des fluides et des métaux; 3) caractériser le comportement des métaux rares dans les minéraux accessoires. La compréhension des processus de concentration des métaux rares est un enjeu scientifique majeur, pour la conception de nouveaux modèles métallogéniques, avec des implications économiques potentielles en terme d'exploration minière dans le Massif Central Français.

Etat de l'art

Les minéralisations à métaux rares du Massif Central Français sont associées spatialement à des granites peralumineux mis en place au cours du Carbonifère vers la fin de l'orogenèse varisque. Elles se présentent sous forme de minéralisations disséminées dans des granites spécialisés et de systèmes filoniens périgranitiques à quartz-wolframite-cassitérite-sulfures. Situé dans le Limousin, au nord-ouest du Massif Central Français, le gisement de Puy-les-Vignes représente un cas original de minéralisation à tungstène associée à une structure de type «conduit bréchique», particulièrement intéressant pour comprendre la nature des fluides et la mobilité des métaux.



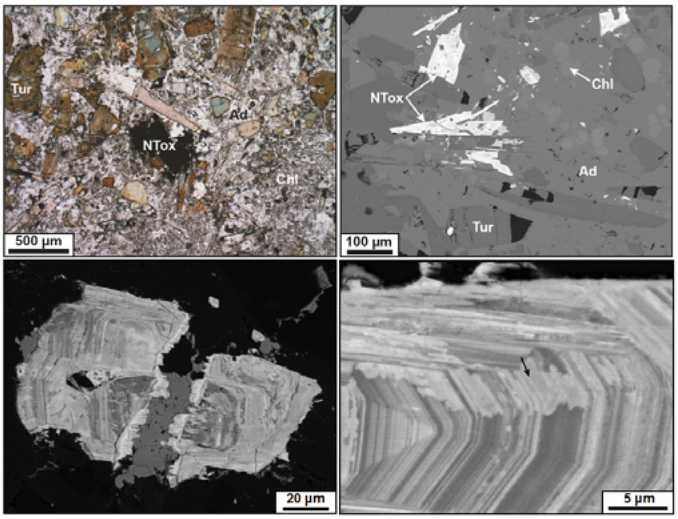
Contexte géologique du gisement de Puy-les-Vignes (PLV) dans le Massif Central Français (MCF)

Approche méthodologique

L'étude pétrographique des encaissants et des veines minéralisées par microscopie optique et électronique a permis d'établir la séquence paragénétique du gisement. Les analyses géochimiques ont été réalisées par microsonde électronique afin de mesurer les concentrations en métaux rares.

Résultats

Une nouvelle paragenèse hydrothermale tardive recoupant la minéralisation principale à tungstène a été mise en évidence. Elle est formée d'une association à zircon, rutile, xenotime et d'un nouveau type d'oxyde de Nb-Ti-Y-HREE-W-U dans une matrice d'adulaire, tourmaline et chlorite. L'étude minéralogique de ces oxydes montrent qu'ils se sont formés au cours d'une cristallisation polyphasée complexe. Ils possèdent des concentrations chimiques majeures en Nb, Ti, mineures en Fe, Y, HREE, W, U, Ca, avec des traces de Ta, Mn et Th.



Oxydes de Nb-Ti-Y-HREE-W-U (NTox) de Puy-les-Vignes, observés en microscopie optique et électronique à balayage.

L'étude cristallochimique montre que ces oxydes possèdent une formule structurale du type $A_{1-x}B_{2+x}(O,OH)_6$ avec A: Fe, Mn, Y, REE, Ca, U, Th, B: Nb, Ta, Ti, Si, W et x les vacances structurales. Ces oxydes sont interprétés comme une solution solide multiple entre des pôles purs rutile ($TiTi_2O_6$), columbite ($FeNb_2O_6$), euxenite (Y) ($YTiNbO_6$) et son équivalent théorique à tungstène ($FeTiWO_6$). Ces nouveaux oxydes, non décrits dans la littérature, sont des minéraux tardifs dans cette paragenèse hydrothermale caractérisée par une signature géochimique à P, Y, REE, Nb, Ti, Zr et U, typique du magmatisme peralcalin à métaux rares.

Bilan – Perspectives de développement

Ces résultats suggèrent la contribution d'un fluide hydrothermal peralcalin au cours de la formation du gisement de Puy-les-Vignes. Cette étude ouvre de nouvelles perspectives sur la contribution du magmatisme peralcalin dans la genèse des minéralisations à tungstène, ainsi que sur le potentiel économique de Puy-les-Vignes et de la région Limousin.

Les perspectives de ce travail sont:

-Déterminer l'âge de la minéralisation à tungstène via une datation directe de la wolframite (U-Pb TIMS).

-Analyser la concentration des éléments traces dans la wolframite par LA-ICP-MS.

-Identifier la source des fluides à partir de l'étude des inclusions fluides dans le quartz et la wolframite.

Conseil Scientifique du 23/01/2015
Rapport mi-parcours ANR du 2/06/2015

•Origine de l'horizon B1 des komatiites:
• approche expérimentale

Faure François (CRPG, Nancy), Montel Jean-Marc (GéoRessources, Nancy), Arndt Nick (IsTerre, Grenoble), Nicollet Christian (LMV, Clermont-Ferrand)

AXE Scientifique : 1

Enjeux scientifiques

Les coulées de komatiites sont connues pour contenir des amas sulfures présentant des concentrations en nickel, cuivre et PGE d'importance économique. L'origine des métaux semble sans conteste liée à la nature primitive des magmas. En revanche, l'origine du soufre est plus débattue. Cependant l'hypothèse communément admise correspond à l'assimilation par les coulées de leurs encaissants sédimentaires riches en sulfure. L'érosion thermique de l'encaissant sulfuré permettant d'enrichir artificiellement la lave en soufre au cours de son écoulement.

Etat de l'art

L'hypothèse d'érosion thermique du substratum a été modélisée en considérant une lave souvent surchauffée s'épanchant de manière turbulente (Williams et al., 2001). Cependant, les coulées de komatiites présentent une structuration en horizons bien définis qui laisse suggérer que le caractère surchauffé de la lave est peu vraisemblable. La compréhension de la formation des amas sulfurés porteurs de métaux passe donc nécessairement par une connaissance du mode de cristallisation des coulées dans leur ensemble.

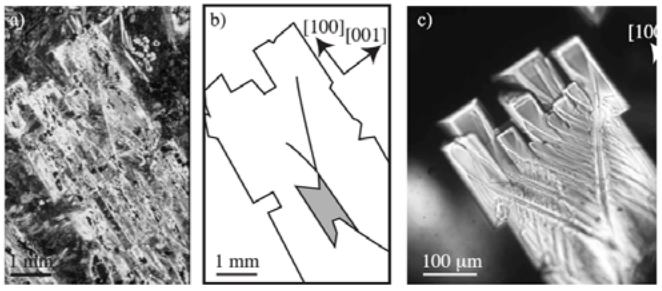
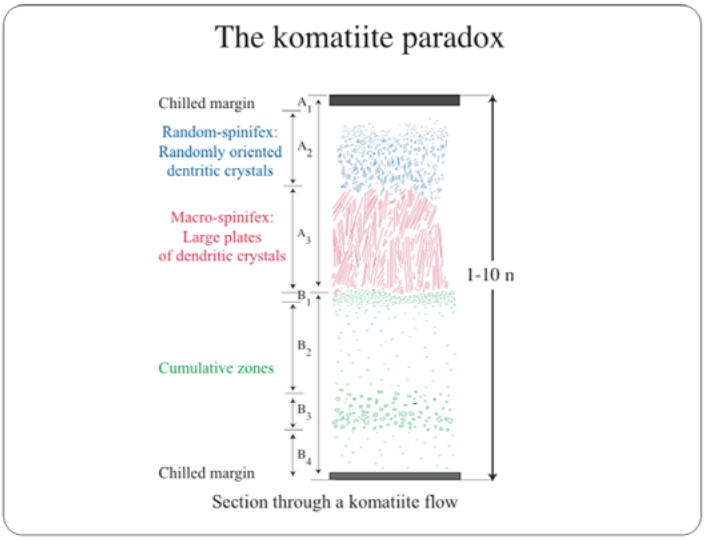


Figure montrant (a) la morphologie d'un cristal B1 naturel, (b) schéma interprétatif du cristal observé en a, (c) morphologie d'un cristal B1 reproduit expérimentalement.

Approche méthodologique

Les expériences de cristallisation dynamique ont été réalisées dans des fours verticaux haute température. Des compositions chimiques simplifiées (CMAS) ont été utilisées afin de s'affranchir, dans un premier temps, du problème du fer.
Les charges expérimentales ont ensuite été analysées avec différents types de microscopie (photonique et électronique) et analysées à la microsonde électronique.



Résultats

1) La morphologie particulière des olivines de l'horizon B1 a été reproduite expérimentalement en enchaînant un épisode de réchauffement après un épisode de refroidissement rapide.
2) Les cristaux B1 correspondent à des cristaux qui se sont formés initialement dans l'horizon A2 au toit de la coulée où la vitesse de refroidissement est rapide et ces cristaux ont sédimenté dans le cœur de la coulée plus chaud.
3) Un modèle global de cristallisation de la coulée de komatiite expliquant la formation des différents horizons texturaux est proposé.

Bilan – Perspectives de développement

Le modèle global de cristallisation impliquant une compétition entre l'avancé du front froid dans la coulée et la sédimentation des cristaux dendritiques d'olivines dans l'horizon A2 impose des contraintes sur l' épaisseur de l'horizon B1. Des mesures de terrain précises de l'horizon B1 de différentes coulées de komatiites devraient permettre d'affiner les paramètres de mise en place de ces coulées et de mieux comprendre la formation et la localisation des amas sulfurés associés. Cette partie pourrait être réalisée en collaboration avec Michel Houlé du Geological Survey of Canada.

Conseil Scientifique du 23/01/2015
Rapport mi-parcours ANR du 2/06/2015

Genèse et évolution des principaux gisements de Terres Rares: Les carbonatites (exemple de l'Oldoinyo Lengaï, Tanzanie)

AXE Scientifique : 1

• Gaelle Mollex1, Lydéric France1, Pete Burnard1, François Faure1, Sören Wilke2, Roman Botchanikov2

•1CRPG-CNRS, Nancy; 2 Institut für Mineralogie, Hannover, Allemagne

Enjeux scientifiques

ENJEUX: Les magmas carbonatitiques représentent les principaux gisements en Terres-Rares [REE] (+P, Nb…) □ *Métaux critiques*, et sont associés à un dégazage en CO2 important

PROBLEMATIQUE: Quelle est la genèse et l'évolution de ces magmas atypiques (laves non-silicatées, extrêmement fluides, très enrichies en métaux critiques), et comment s'altèrent-ils? **Quel est le mécanisme de concentration des REE, Nb-Ta, P?**
OBJECTIFS: Identifier les processus qui régissent la genèse et l'évolution des carbonatites (et magmas alcalins associés) pour pouvoir **identifier** et **quantifier** le(s) processus de concentration des REE dans ces magmas, ainsi que les phases porteuses.

Etat de l'art

• Deux modèles principaux existent aujourd'hui pour la genèse des magmas carbonatitiques: *fusion d'un manteau enrichi en carbone, *fusion d'un manteau classique associé à une différenciation extrême résultant en l'immiscibilité de magmas silicatés et carbonatitiques.

•• L'origine de la forte concentration en REE des produits finaux n'est pas identifiée (magma initial, processus dans la chambre magmatique, ou altération secondaire).

Résultats

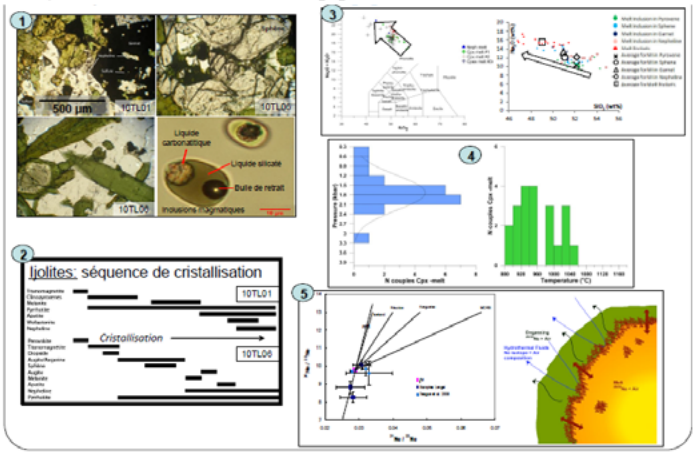
• L'étude pétrologique **[1]** et géochimique des enclaves a permis:

•*de caractériser les séquences de cristallisation **[2]**;

•*d'obtenir la composition des liquides ainsi que leur évolution au cours du processus de cristallisation fractionnée **[3]**;

•*de contraindre les paramètres physiques (profondeur de la chambre magmatique et température du magma) **[4]**;

•*de caractériser la composition isotopique (Ne, Ar) des magmas présents dans la chambre magmatique et d'identifier un composant de contamination **[5]**;



Approche méthodologique

• Le projet est basé sur une étude micro-pétrologique, géochimique et expérimentale d'échantillons provenant directement de la chambre magmatique du seul édifice carbonatitique actif aujourd'hui. Ces échantillons sont des roches plutoniques qui ont enregistré (via des inclusions de magma [IM] piégées dans les différents minéraux) les étapes successives de l'évolution chimique du magma depuis son extraction du manteau jusqu'à son enrichissement en REE.

•Une fois les séquences de cristallisation des minéraux reconstruites, des analyses in-situ (via EPMA, LA-ICP-MS, et SIMS) seront effectuées au sein des IM pour identifier les fractionnements chimiques mis en oeuvre à chaque étape.

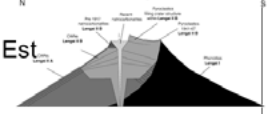
Contexte

• L'Oldoinyo Lengaï (OL; Tanzanie) est le seul volcan arbonatitique actif sur Terre;

• Plus de 500 occurrences de magmatisme carbonatitique au cours de l'histoire de la Terre;

• L'OL se situe sur la branche Est du Rift Est Africain; l'activité débute il y a 370ka

• Deux périodes principales: Lengaï I (phonolitique) et Lengaï II (néphélinitique)



Coupe et unités lithologiques de l'Oldoinyo Lengaï par Klaudius et al. (2006)

Discussion

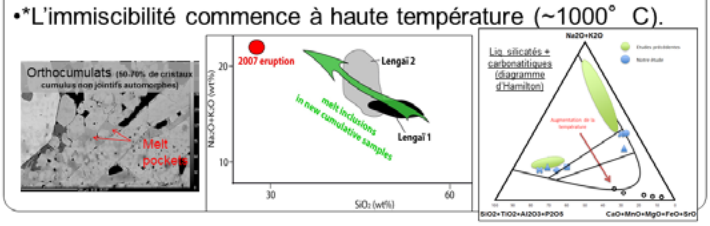
• Les résultats préliminaires permettent de mettre en évidence:

•*Ijolites formées à ~6 km de profondeur (barométrie);

•*la cristallisation d'un magma silicaté en bordure de chambre;

•*l'évolution de la composition du liquide au cours de la cristallisation observée couvre toute la gamme de variation connue pour l'Oldoinyo Lengaï;

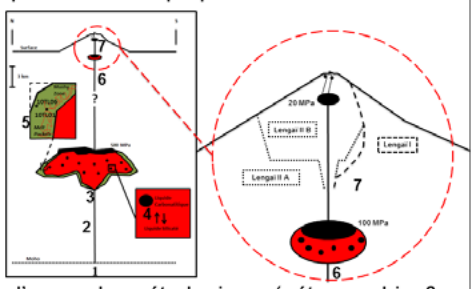
•*L'immiscibilité commence à haute température (~1000° C).



Bilan – Perspectives de développement

Les résultats obtenus permettent de proposer le modèle suivant:

- 1. Fusion partielle (0,3%) à partir d'un manteau type MORB
- 2. Différenciation du liquide silicaté
- 3. Arrivée d'un liquide silicaté de composition phonolitique
- 4. Immiscibilité déjà présente à 200 MPa et au-delà de 1000 ° C (**pôle calciocarbonatitique**)
- 5. Contamination en bordure de chambre
- 6. Cristallisation fractionnée (10TL01 et 10TL06)
- 7. Arrivée d'un liquide néphélinitique et immiscibilité continue (**pôle natrocarbonatitique**)
- 8. Migration du liquide carbonatitique + différentiation tardive



Le modèle établi avec l'approche pétrologique (pétrographie & géochimie) est actuellement complété par une approche expérimentale (IfM, Hanovre & ETH, Zürich).

Conseil Scientifique du 23/01/2015
Rapport mi-parcours ANR du 02/06/2015

Ge, éléments traces associés, et isotopes du Ge dans les sphalérites par LA-ICP-MS et MC-ICP-MS

Belissont R.[a,b], Boiron M.-C.[a] ,Luais B.[b], et Cathelineau M.[a]

[a] GeoRessources, CNRS-UMR 7359, Vandoeuvre-lès-Nancy
[b] Centre de Recherches Pétrographiques et Géochimiques, CNRS-UMR 7358, Vandoeuvre-lès-Nancy,

AXE Scientifique : 1

Enjeux scientifiques

Le germanium (Ge) est largement utilisé dans l'industrie des hautes technologies (semi-conducteurs, fibres optiques). La position dominante de la Chine sur ce marché (83% de la production mondiale en Ge), représente un risque pour l'économie de l'U.E. et motive une recherche accrue.

Les enjeux concernent la compréhension du cycle géologique du Ge et l'identification des facteurs contrôlant sa concentration dans les minéraux métalliques.

Etat de l'art

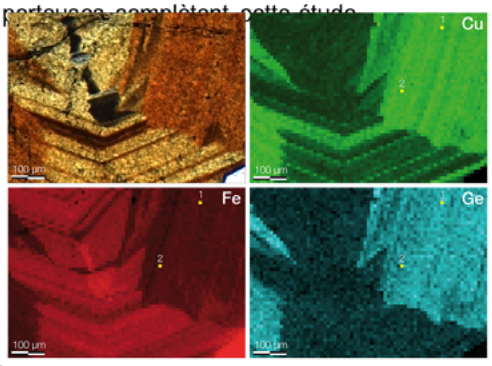
Une particularité intéressante du Ge est d'être un élément sidérophile, chalcophile, lithophile et organophile, si bien qu'il est présent dans un grand nombre de réservoirs terrestres.

Dans la croûte, les plus fortes teneurs en Ge sont reportées dans les charbons et certains sulfures. Les gîtes Zn-Cu hydrothermaux de basse température sont souvent enrichis en Ge. Dans la sphalérite (ZnS), le Ge peut atteindre notamment des teneurs de 2500 ppm en substitution du Zn.

Les minéraux porteurs de Ge (sphalérite, chalcopyrite, et sulfures de Ge) provenant de gisements de contextes géologiques variés font l'objet de cette étude: St-Salvy (France), Barrigão (Portugal) et Kipushi (R.D. Congo).

Approche méthodologique

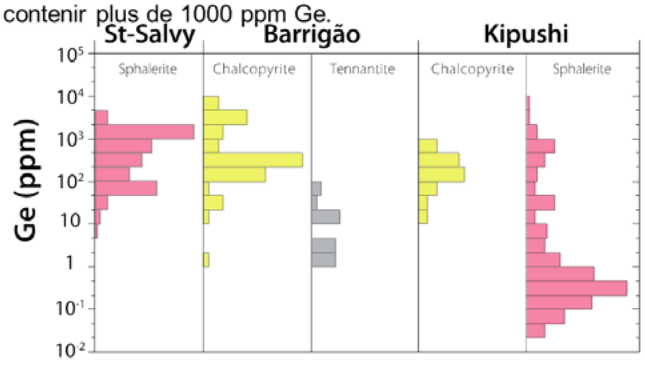
L'objectif de cette étude est de comprendre les mécanismes et les processus de concentration du Ge et des éléments mineurs/traces associés (e.g., Cu, In, Ga) dans les minéraux porteurs. L'étude pétrographique détaillée (microscopie, MEB, MSE) est ensuite approfondie par le couplage d'analyses *in situ* des traces par LA-ICP-MS (GeoRessources, Nancy), et d'analyses isotopiques globales (Ge, Fe) par MC-ICP-MS (CRPG, Nancy), et S par spectrométrie de masse en phase gazeuse (EA-IRMS, CRPG) sur des grains de sphalérite séparés. Des analyses de l'état d'oxydation du Ge par µ-XANES (synchrotron, ESRF Grenoble) dans les phases porteuses complètent cette étude.



Cartographie µ-XRF (synchrotron) Ge est enrichi dans les zonages sectoriels, corrélé à Cu.

Résultats

Les analyses élémentaires mettent en évidence un contrôle cristallographique de lincorporation du Ge dans les sphalérites de St-Salvy (Belissont et al., 2014). La chalcopyrite est le porteur principal de Ge dans le gisement de Barrigão (jusqu'à plus de 5000 ppm). Si les principaux porteurs de Ge sont les sulfures de Ge (rénierite, germanite, ≈6-7% Ge) dans le gisement de Kipushi, la sphalérite et la chalcopyrite peuvent contenir plus de 1000 ppm Ge.



Les analyses isotopiques du Ge dans la sphalérite de St-Salvy montrent une large gamme de composition isotopique ($\delta^{74}Ge_{NIST3120a}$ de -2,07 à +0,91‰) caractéristique d'une précipitation en système ouvert à partir de fluides hydrothermaux de basse température (<150° C). Des analyses isotopiques complémentaires (Fe, S, globales) permettent d'affiner le modèle de formation des minéralisations de sphalérite.

Trois échantillons de sphalérite de St-Salvy couvrant la gamme des concentrations et compositions isotopiques en Ge ont été étudiés par µ-XANES (Oct. 2013, ligne BM23). Les spectres µ-XANES montrent un état d'oxydation des métaux identique selon les deux types de zonations. Des états d'oxydation différents sont néanmoins observés entre les échantillons, corrélés aux concentrations en Ge et aux rapports isotopiques. Ces observations permettent d'apporter de nouvelles contraintes sur les mécanismes de substitutions et les conditions physico-chimiques du fractionnement isotopique de Ge (Belissont et al., *soumis à EPSL*).

Bilan – Perspectives de développement

Les résultats sur la sphalérite de St-Salvy ont été publiés à *GCA*[1] (Belissont et al., 2014). Les données µ-XANES obtenues au synchrotron font l'objet d'une seconde publication soumise à *EPSL*[2].

Une approche expérimentale complètera cette étude afin d'affiner notre compréhension des mécanismes d'incorporation du Ge dans la sphalérite ainsi que son fractionnement isotopique.

Un nouveau projet synchrotron a été soumis en septembre 2014 pour la poursuite de l'étude de l'incorporation du Ge dans les sulfures de Cu par µ-XANES/EXAFS (ESRF, Grenoble).

Références : [1] Belissont, R., Boiron, M-C., Luais, B., Cathelineau, M., 2014, LA-ICP-MS analyses of minor and trace elements and bulk Ge isotopes in zoned Ge-rich sphalerites from the Noailhac – St-Salvy deposit (France): Insights on incorporation mechanisms and ore deposition processes, *Geochimica et Cosmochimica Acta*, 126, 518-540, [2] Belissont, R., Munoz M., Boiron, M-C., Luais, B., Mathon O. Determination of the oxidation state of Ge, Cu and Fe in zoned Ge-rich sphalerite: Integrated µ-XRF mappings and µ-XANES study. *soumis EPSL*

Conseil Scientifique du 23/01/2015
Rapport mi-parcours ANR du 2/06/2015

Fluides et métallogénie des métaux de base (Pb-Zn-Cu-Ag-Au) autour des plutons syntectoniques du domaine égéen

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VANDERHAEGHE Olivier, GéoRessources
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PIK Raphaël, CRPG
France Lydéric, CRPG

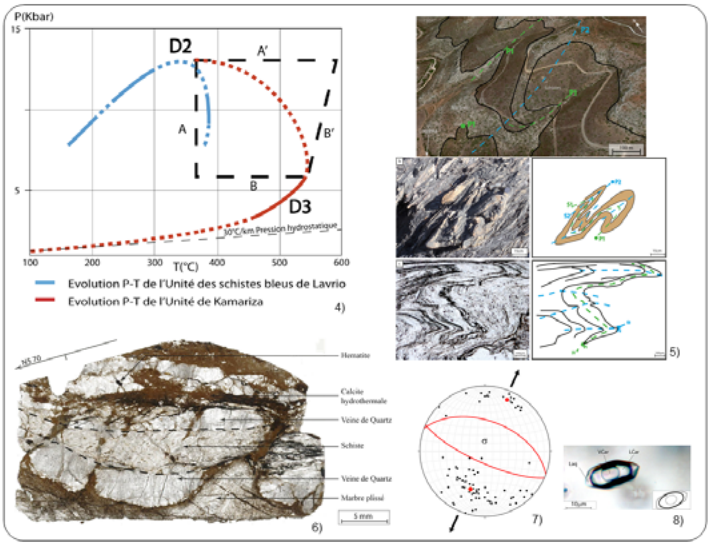
AXE Scientifique : 1

Enjeux scientifiques

L'objectif de ce projet est de comprendre la **genèse**, le **rôle** et l'**évolution** des **circulations fluides** à l'**échelle de la lithosphère** dans un contexte orogénique depuis la **subduction** jusqu'à l'**effondrement** de la chaine [1]. Cette étude vise à replacer chacun des **événements hydrothermaux** dans son **contexte géodynamique** et ainsi permettre de retracer l'histoire des formations des **gîtes métallifères**. La région du **Laurion** fut choisie d'une part par sa position stratégique au sein du complexe métamorphique égéen [2-3] et d'autre part par ses richesses métallifères.

Etat de l'art

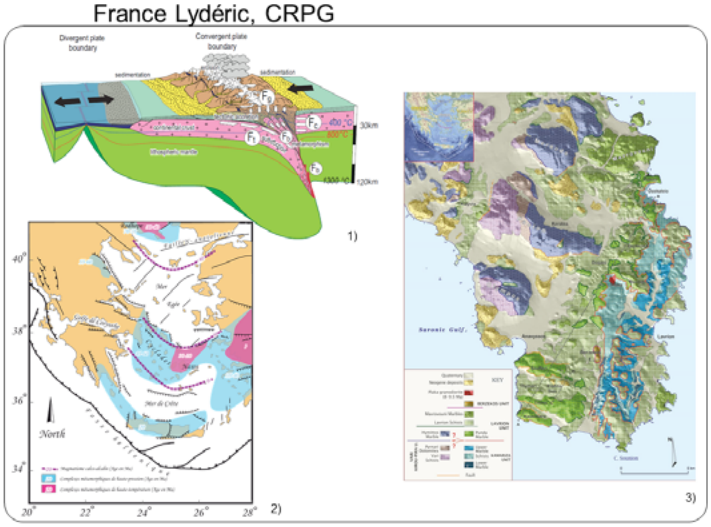
La région du **Laurion** (Grèce continentale) est localisée à l'intersection entre les **chevauchements** et **détachements syn-orogéniques** [2-3] responsables de l'empilement et de l'exhumation des nappes accommodant la formation du **Metamorphic Core Complex** égéen. Cette région a enregistré des degrés de **métamorphisme** distincts et diachrones allant des Schistes bleus aux Schistes verts, à la migmatisation et à la fusion de la croûte (Naxos). De nombreux événements minéralisateurs riches en **Pb-Zn-Fe-Cu-Ag-Au**, pour la plupart associés à l'**évolution tardi-orogènique**, liés à l'extension et l'effondrement de la chaine jalonnent la région .



Approche méthodologique

L'approche scientifique fut la suivante:
- **Cartographie** et **structure (terrain)**
- **Echantillonnage** systématique *(veines et encaissant)*
- **Pétrographie** *(microscopie optique, MEB)*
- **Géochimie** *(minéraux et roche totale)*
- **Inclusions fluides** *(pétrographie, microthermométrie, Raman)*

Afin de :
- Caractériser l'**évolution thermobarométrique** de la région
- Reconstruire l'**histoire tectonique** et **métamorphique** de chacune des unités lithotectoniques
- Définir les **contextes de mise en place** des roches magmatiques et sédimentaires associés
- Retracer l'**histoire des fluides** associés au **contexte géodynamique** et aux **événements minéralisateurs**



Résultats

Ces travaux ont permis de :

- Contraindre le **trajet *PT* des unités métamorphiques du Laurion**, avec une phase D2 (12,5 Kbar, 350 ° C – Schistes bleus) retromorphosée par une phase D3 (6 Kbar, 550 ° C – Schistes verts) [4]

- Identifier et corréler ces **phases de déformations** avec celles observées à toutes les échelles (P_n E-W transposés suivant un axe N-S, P_{n+1}) expliquant notamment les successions de marbres visibles dans la vallée de Megala Pefka [5]

- Identifier que les **schistes du Laurion** se sont déposés en contexte de **marge passive**, et les **schistes de Kamariza** en contexte de **marge active**

- Identifier que la **phase de mylonitisation** N-S liée à la formation du MCC au passage d'un régime ductile à fragile a engendré une **dissolution/décarbonatation** des bancs de marbres [6-7] accompagnée d'une genèse de **fluides aquo-carboniques** [8]. Il pourrait s'agir d'un événement associé à la première phase de **formation des minéralisations** identifiées comme étant du type remplacement de carbonates.

Bilan – Perspectives de développement

Ces données ont permis de recadrer l'**histoire géodynamique du Laurion**, sur lequel nous sommes désormais capable de relier les différents événements à l'**histoire des fluides et des minéralisations**. Deux publications sont en cours d'écriture.

Ces travaux ont fait l'objet de sujets de **Master** *(Ponthus 2012, Scheffer 2012/2013)* et sont à la base du lancement de la **thèse** de **C. Scheffer** sur la thématique des circulations fluides à l'échelle de la région du Laurion et de l'Eubée.

Très récemment l'équipe de ce projet a démarré une collaboration avec une équipe d'archéologues-spéléologues (D. Morin - Université de Toulouse et de Lorraine), l'IGME (A. Photiades) et l'Université d'Athènes (P. Voudouris) afin d'identifier les minéralisations exploitées durant l'antiquité dans le secteur du Laurion.

Conseil Scientifique du 23/01/2015
Rapport mi-parcours ANR du 2/06/2015

The role of thermochemical sulfate reduction in the origin of Mississippi Valley-type depositits

Truche Laurent, Barré Guillaume, Bazarkina Elena, Dubessy Jean

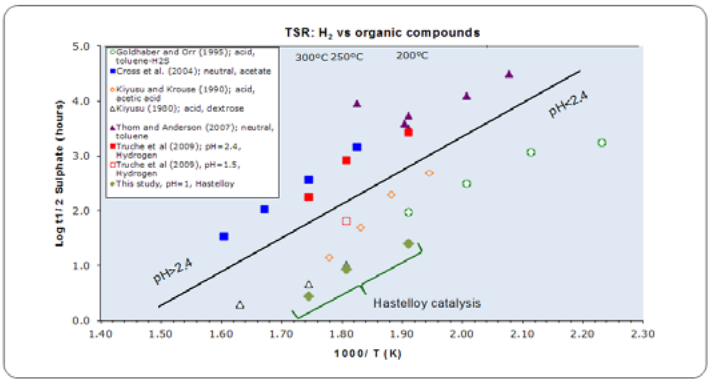
AXE Scientifique : 1

General Framework

Thermochemical sulfate reduction (TSR) refers to the abiological, thermally driven reduction of sulfate to hydrogen sulfide induced by natural electron donors such as hydrocarbons, ferrous iron found in rocks, carbon graphite, methane or hydrogen. The occurrences, and consequences of TSR have been documented in numerous geological observations from around the world at temperatures ranging from 100° to more than 400° C. TSR plays a crucial role in the deposition of metal sulfide ore bodies in both magmatic/hydrothermal) and sedimentary settings (MVT, VMS, black-shale-hosted Ni-Cu-Zn-Co ore deposits, Copperbelt).

Objectives

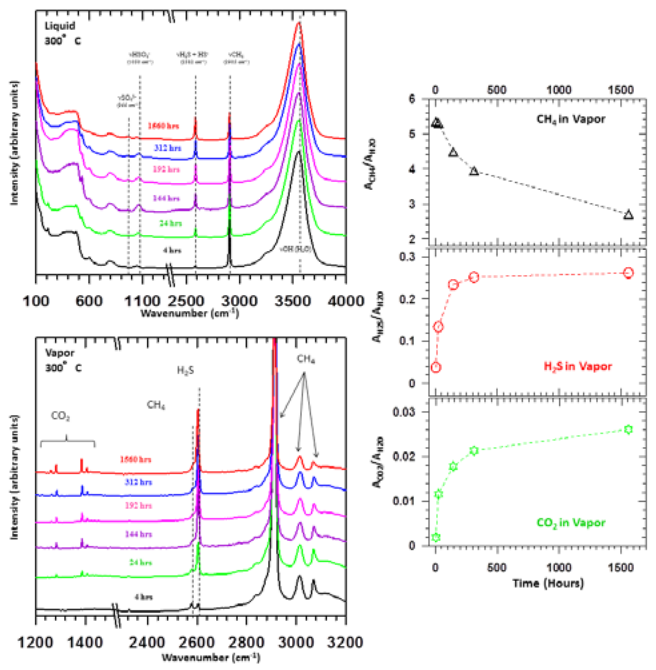
Although the process of TSR has been recognized for nearly fifty years, understanding the critical geologic and geochemical controls has been hindered by: i) the inability of experimentally reproduce TSR under laboratory conditions comparable to those deduced from field sites, ii) the lack of systematic evaluation of the reaction pathway. Understanding The mechanism of H_2S catalysis in sulfate reduction is necessary to constrain both the reaction kinetic, and the sulfur isotopic fractionation models derived from laboratory experiments, which can then be extrapolated to geologic conditions. Also, it is still controversial whether methane, the predominant component of natural gases, actually participates in natural TSR reactions. The aim of the present study is to evaluate TSR reaction rate and mechanism under geologicaly relevant conditions.



Methods

We employed in situ Raman spectroscopy in silica-glass capillary at temperature ranging from 25 to 350° C in order to measure the identity and stability of sulfur species involved during TSR experiments. We performed original kinetic experiments using methane as reducing agent, and we reproduced classical TSR experiments of Kiyosu and Krouse (1993), Goldhaber and Orr (1995), Cross et al. (2004), and Thom and Anderson (2007). Altogether, these experiments allow us to evaluate the effect of temperature, pH, and sulfur speciation on TSR reaction rate and mechanism.

$MgSO_4$-S-H_2O-CH_4



Results

In situ Raman spectra collected at high temperatures and pressures in the $MgSO_4$-S-H_2O-CH_4 and other S(+VI)-S(-II)-H_2O-CH_4 systems demonstrate that (1) the disproportionation of elemental sulfur occured at temperature above 200° C and produced sulfide and sulfate, and (2) sulfate, in the presence of sulfide, can be reduced by methane within few hundreds of hours at 300° C and few months at 200° C to produce CO_2 and H_2S. This is the lowest temperature at which TSR has been documented so far. TSR may proceed under low temperature conditions, such as those encountered in oil fields, or MVT deposits, provided sulfide is initially present. Methane can act as an efficient electron donor.

Perspectives

We would like to extend the parametric kinetic investigation, by varying pH, sulfate speciation, temperature, and the nature of the reducing agent. Sulfur speciation play a crucial role in TSR processes but it remain to be understood at T above 100° C. Our developed experimental technique will be useful to resolve sulfur speciation in complex system out of thermodynamic equilibrium.
Comparisons with natural system through fluid inclusion and sulfur isotopic analysis will be a major tack in 2014.

Conseil Scientifique du 23/01/2015
Rapport mi-parcours ANR du 2/06/2015

Développement d'un laboratoire pour la datation K-Ar

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C. France-Lanord-(CRPG),
P. Robert-(Otelo))

AXE Scientifique : 1

Enjeux scientifiques

La datation des évènements fluides et des phases de formation d'un gisement métallique est essentielle à la conceptualisation des modèles métallogéniques. Or dans les domaines de basse et moyenne température, seuls les minéraux de gangue sont le plus souvent utilisables car seuls porteurs d'éléments radiogéniques. Les argiles sont les phases les plus ubiquistes mais ne peuvent être datées en Ar-Ar, ce qui oblige à utiliser le système K-Ar. L'objectif est donc de redévelopper le K-Ar appliqué aux argiles, notamment les phases les plus fines.

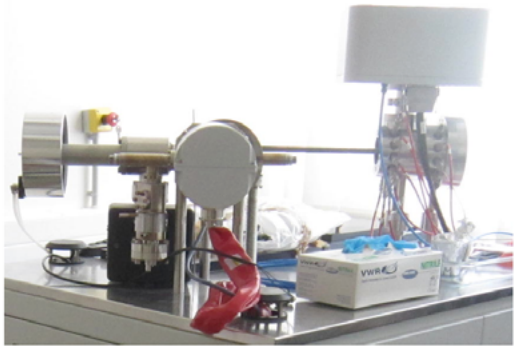
Etat de l'art

La méthode potassium - argon permet par application de l'équation géochronologique, de dater des événements de quelques milliards d'années à plusieurs milliers d'années, en dosant indépendamment K et Ar.
Elle devrait permettre de résoudre des problèmes de datation des minéraux de gangue (argiles en particulier) des minéralisations lorsque d'autres outils radiochronologiques ne sont pas applicables.

Vue du spectromètre Thermofischer Argus

Approche méthodologique

L'ensemble des équipements comprend :
- les méthodes d'extraction des argiles (dislocation des roches sans création de fines de phyllosilicates hérités et radiogéniques par cycle de cryogénie, extraction par décantation et ultracentrifugation)
- un dispositif d'extraction composé essentiellement d'un four ;
- un dispositif de calibrage (injection de traceur (spike)) ;
-un dispositif de purification qui piégera la presque totalité des composants du mélange issu du four
- un dispositif de mesure comprenant le spectromètre de masse et sa ligne d'introduction.



Vue du spectromètre Thermofischer Argus

Résultats

Le spectromètre Thermofisher Argus a été testé au CRPG et installé en mai 2013 à Georessources.

La ligne de gaz est en cours de conception et montage, montage qui sera finalisé en début 2015.

Les tests démarreront au second trimestre 2015.

Bilan – Perspectives de développement

La datation des argiles est du plus grand intérêt pour la compréhension des formations des gisements métalliques, mais aussi pour l'étude des modes de remplissage des bassins et l'identification des sources des éléments détritiques (muscovite, feldspaths) en combinaison avec d'autres approches géochronologiques.
Une collaboration a été amorcée entre Ressources 21 , le CSIRO et l'Université de Western Australia (H. Zwingmann) autour de la datation des évènements fluides par la datation des argiles. H. Zwingmann devrait séjourner à Nancy dès que la ligne préparative sera connectée au spectromètre.

Conseil Scientifique du 23/01/2015
Rapport mi-parcours ANR du 2/06/2015

Séjour scientifique de 6 mois au Québec pour conforter la position stratégique de l'UL dans le domaine du génie minier

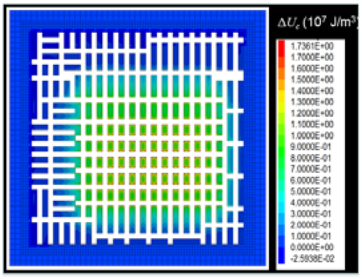
Yann Gunzburger, GéoRessources

AXES Scientifiques : 1

Enjeux scientifiques et état de l'art

Alors que la France a longtemps bénéficié d'une expertise reconnue dans le domaine de l'exploitation minière, la fermeture des dernières mines, accompagnée des départs en retraite de ceux qui les ont connues en activité, a entraîné une érosion progressive des compétences nationales en génie minier, tant sur le plan de la formation, que de la recherche et de l'ingénierie. En parallèle, l'intérêt pour les ressources naturelles s'est beaucoup focalisé ces dernières années sur l'amont (processus concentrateurs, prospection) et sur l'aval du cycle minier (traitement des minerais, environnement minier, après-mine), délaissant ainsi les aspects liés aux techniques d'exploitation elles-mêmes, au sujet desquelles d'importants défis techniques se présentent pourtant du fait des conditions de plus en plus difficiles d'accès aux gisements (grandes fosses, mines profondes sujettes aux « coups de terrain », réduction des risques, etc.).

Chevalement de la mine Niobec (Saguenay), où plusieurs coups de terrain ont été enregistrés.



Calcul par modélisation numérique de l'énergie élastique emmagasinée dans les piliers et le terrain d'une mine lors de son développement. Ce calcul permet de déterminer la probabilité de survenue des coups de terrains, notamment dans les angles des piliers où on observe une forte densité d'énergie.

Résultats scientifiques

Mon séjour m'a permis de développer les outils de modélisation nécessaires à l'estimation de la sismicité induite à partir du code commercial FLAC-3D. Ce travail a été valorisé par deux communications dans des congrès internationaux (World Mining Congress à Montréal et Congrès Eurock en Pologne). Un article pour une revue internationale est également en cours de finalisation. Ce travail se poursuivra sous la forme de collaborations entre GéoRessources et l'Université McGill, l'Université Laval et l'Université du Québec à Chicoutimi (réponses conjointes à des appels d'offres, co-encadrement projeté de masters et de doctorats, etc.).

Approche méthodologique

L'objectif de ma mission de 6 mois au Québec, était de :
- de dresser un portrait et de faire un diagnostic de la vitalité du génie minier dans cette grande province minière (formation, recherche, ingénierie, activité économique),
- d'établir ou renouer des collaborations avec des établissements universitaires québécois pour favoriser l'émergence d'actions de recherche communes et promouvoir les échanges d'étudiants et d'enseignants,
- de prendre des contacts auprès des grandes entreprises minières présentes au Québec afin de favoriser les collaborations en R&D et de faciliter le placement des étudiants lorrains en stage et dans leur premier emploi.

En parallèle, j'ai développé un projet de recherche portant sur l'utilisation de la modélisation numérique pour la prévention des coups de terrain (« rockbursts ») dans les mines profondes en roches dures, qui sont des manifestations de la libération brutale de l'énergie élastique emmagasinée dans les zones de concentration des contraintes induites par l'exploitation. La prévention de ce phénomène nécessite la connaissance de l'état de contrainte initial, de ses hétérogénéités locales (associées par exemple aux zones de faille), de son évolution lors de la progression des fronts de taille et le suivi en continu de la sismicité induite. La modélisation numérique est un outil incontournable pour y parvenir.

Bilan – Perspectives de développement

Le Québec apparaît très naturellement comme un partenaire de premier ordre pour le Labex Ressources21 dans le domaine du génie minier, tant sur le plan de la recherche que de la formation et de l'ingénierie. Toutefois, étant donné le dynamisme local existant actuellement au Québec, la mise en place de nouvelles collaborations nécessitera soit de s'appuyer sur des universités québécoises « sœurs », soit de viser des domaines de compétence spécifiques à notre université : modélisation numérique avancée, acquisition et traitement en continu de données (notion d'observatoire), intégration de la dimension humaine (risques et plus largement toute forme de plurisdisciplinarité faisant une place aux SHS), etc. Cela ne pourra aboutir sans un renforcement des actions de communication et d'affichage de nos domaines d'excellence (présence dans des forums, séjours scientifiques réguliers, visite d'étudiants en stages, etc.). Il apparaît donc souhaitable de maintenir, voire de renforcer, l'affiche de Ressources21 (et plus largement du secteur des géosciences de l'Université de Lorraine) aux forums du PDAC (à Toronto en mars) et de Québec Mines (à Québec en novembre), entre autres.

Conseil Scientifique du 23/01/2015
Rapport mi-parcours ANR du 2/06/2015

Modélisation du comportement hydrogéo-mécanique d'un réseau de failles sous l'effet des variations de l'état de contrainte

Faivre Maxime, Giot Richard, Golfier Fabrice,

GeoRessources (Université de Lorraine – ENSG/CNRS/CREGU), UMR 7359, Vandœuvre-lès-Nancy

AXE Scientifique : 5

Enjeux scientifiques

Les gisements métallifères liés aux enrichissements supergènes (*e.g.*, pour le nickel) sous l'effet de l'altération et du lessivage des roches ultramafiques, sont fortement contraints par le développement et l'extension du réseau de fractures préexistant.

En effet, ces fractures jouent un rôle essentiel dans le processus de minéralisation à la fois en facilitant le lessivage de la péridodite mais aussi en contribuant à la concentration du nickel par l'augmentation de l'espace poral et la remobilisation des phases minérales nickélifères préalablement formées.

L'influence de ces processus de fracturation active sur la formation des gisements et le couplage avec la circulation des fluides restent toutefois encore très mal compris.

Etat de l'art

Une des principales difficultés dans la modélisation du couplage entre dynamique de fracture et écoulement provient du constant remaillage nécessaire afin de tenir compte de l'évolution de la géométrie du réservoir. L'originalité de la méthode des éléments finis étendue (XFEM) (Belytschko & Black 1999), utilisée dans le cadre de cette thèse est d'intégrer directement la discontinuité (la fracture) dans l'écriture de l'équation aux nœuds (méthode « *Level Set* », cf. Fig. 1). Autrement dit, il n'est plus nécessaire de mailler ces objets géologiques allégeant ainsi considérablement les temps de calcul.

L'approximation des champs de déplacement et de pression est rendue discontinue par l'ajout de degrés de liberté supplémentaires (cf. Fig. 1) définis au voisinage de la fracture.

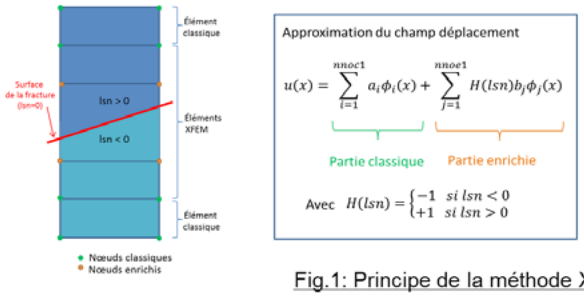


Fig.1: Principe de la méthode XFEM

Approche méthodologique

Le but est d'introduire un nouveau type d'éléments finis hydromécaniques (pour le cas saturé) dans le code de calcul *Code_Aster* (EDF). L'originalité de cet élément est multiple car il peut (Fig. 2) :

- prendre en compte la discontinuité des champs de déplacement et de pression au voisinage de la fracture,
- décrire la circulation du fluide dans la fracture,
- autoriser les échanges de fluides fracture/massif,
- prendre en compte la fermeture ou l'ouverture des parois de la fracture (utilisation d'un modèle de zone cohésive)

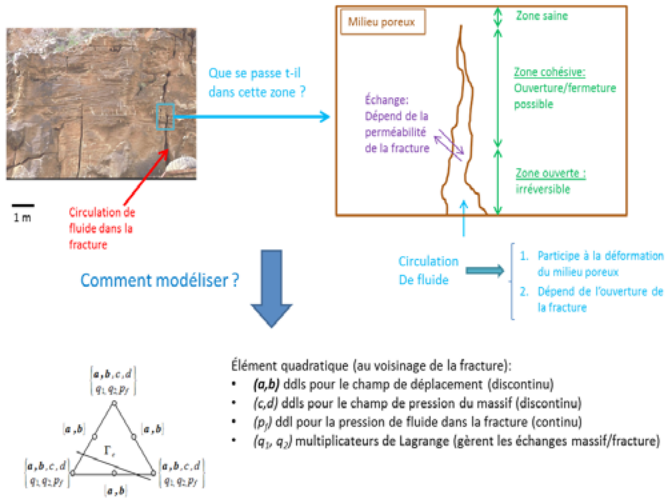


Fig.2: Du massif réel au modèle éléments finis

Résultats

Les premiers développements ont conduit à la validation:

- de la prise en compte des *level-sets* dans le modèle,
- du sous-découpage en sous-éléments pour l'intégration numérique de part et d'autre de la fracture,
- de la discontinuité du champ de déplacement au voisinage de la fracture.

La validation est menée (pour une pression de pore nulle) sur un barreau de roche traversé par une fracture où est appliquée une force répartie sur les deux lèvres. Les résultats sont cohérents avec la théorie (Fig. 3)

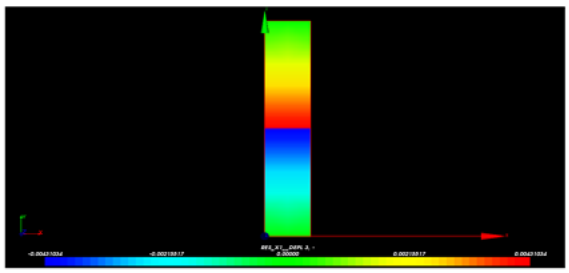


Fig.3: Discontinuité du champ de déplacement

Bilan – Perspectives de développement

La suite des travaux de thèse consistera à coupler les développements de l'élément HM-XFEM avec la modélisation de l'écoulement et du transport d'espèces chimiques au sein du massif fracturé. Le couplage réactif sera réalisé de manière séquentielle par le biais d'un code géochimique. Les applications futures dépassent bien évidemment le cadre du Labex et devraient conduire par exemple à une meilleure compréhension des processus d'endommagement autour des ouvrages souterrains (stockage de déchet…) ou au développement des méthodes de fracturation hydraulique (thèse IFPEN, GeoRessources –LAMSID, 2013-2016).

Conseil Scientifique du 23/01/2015
Rapport mi-parcours ANR du 2/06/2015

Paramètres de déformation dans la prédiction des zones endommagées favorables aux minéralisations aurifères situées loin des failles

Pablo Mejía-Herrera, Jean-Jacques Royer

GeoRessources - Gocad Research Group

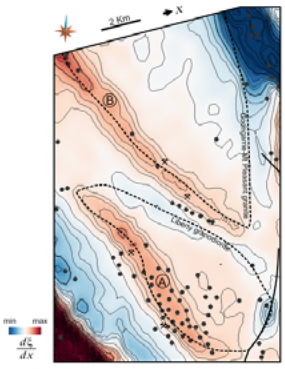
AXE Scientifique : 5

Enjeux scientifiques

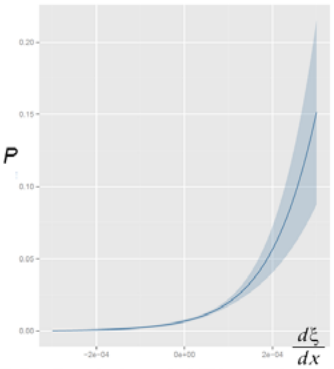
Les coûts élevés de l'exploration et les risques économiques des investissements limitent le développement des projets miniers. C'est pourquoi il est primordiale de développer les méthodes prédictives pour identifier les zones minéralisées susceptibles d'être mises en exploitation. Ce projet vise à mettre au point des méthodes d'estimation et de prévision des ressources minérales stratégiques d'une région à partir de critères structuraux liés au processus de minéralisation et obtenus lors de la restauration.

Etat de l'art

La reconstitution de la géométrie d'une région dans son état initial est appelée *restauration*. Elle peut être utilisée pour : valider un modèle géologique, estimer les déformations et leurs directions, éventuellement les contraintes et pour prédire les zones de fracturation (Groshong, 2006). La restauration a été utilisée pour quantifier la déformation du district de Mount Pleasant, Australie, et mettre en évidence des zones fractures situées loin des systèmes de failles, et susceptibles d'être minéralisées en Or (Moir et al., 2003).



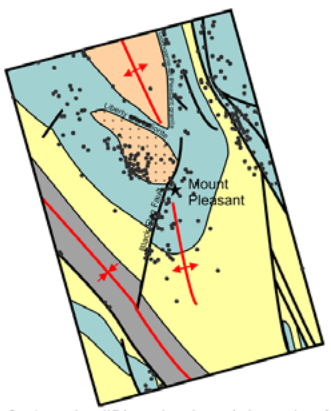
Gradient de ξ qui met en évidence les zones endommagées pendant la déformation



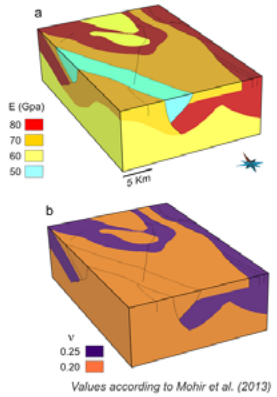
Estimation de la probabilité par régression logistique, de trouver une zone aurifère en utilisant le gradient de ξ.

Approche méthodologique

La démarche repose sur le développement de modèles structuraux 3D régionaux comprenant le gisement à étudier, l'évolution tectonique de la région, les principaux évènements tectoniques, le changement des conditions physico-chimiques et des propriétés mécaniques de l'encaissant. Le but est d'introduire dans le modèle rhéologique tous les phénomènes et processus géologiques observés ou supposés dans le gîte, même s'ils ne sont pas liés d'une façon évidente aux minéralisations. L'endommagement de la roche a été calculé selon le modèle de Lyakhosvsky et al. (1997) pour estimer les zones favorables aux minéralisations.



Carte simplifiée de la région de Mount Pleasant, Australie, montrant la distribution de l'Or. Ce travaux sont focalisés sur la concentration de l'Or située loin des systèmes de failles.



Modèle structural du district minier du Mont Pleasant avec propriétés élastiques des différentes unités de roches. **a**. Module de Young. **b**. Coefficient de Poisson.

Résultats

La restauration 3D fournit à la fois le comportement structural et mécanique de la zone d'étude, en simulant la déformation qui se produit au cours de l'épisode de raccourcissement impliqué dans le processus de minéralisation. Les résultats obtenus sont conformes aux endommagements et aux systèmes aurifères éloignés des failles du district de Mount Pleasant. Les paramètres de déformation intègrent l'hétérogénéité des roches et la configuration géométrique des unités avec le phénomène d'endommagement et la fracturation qui contrôlent la distribution d'Or dans la région. Avec l'utilisation du champ de déformation obtenu lors de la restauration, il est possible de définir les domaines potentiels de perméabilité élevée liée aux zones fracturées et favorables aux systèmes hydrothermaux. La méthodologie ici montrée est applicable aux métaux stratégiques qui ont une genèse similaire.

Bilan – Perspectives de développement

Les résultats obtenus montrent que la méthodologie utilisée donne un outil pour prévoir les zones minéralisées. Cette méthodologie n'est pas seulement applicable pour des minéralisations aurifères mais aussi aux gisements où l'endommagement et la fracturation de la roche ont joué un rôle primordial dans la formation de la minéralisation. Ce projet pourrait initier une collaboration étroite entre les laboratoires du Labex 21 (mécanique de roche, analyse pétrographique et de géomodélisation) pour dresser une carte des zones potentielles des différents gisements en Europe. De contacts ont été prit avec *Mira Geoscience* (Canada) pour exploiter ces résultats commercialement afin de définir une stratégie de recherche des zones minéralisées.

Conseil Scientifique du 23/01/2015

Rapport mi-parcours ANR du 2/06/2015

Enrichissement en scandium des profils latéritiques de Nouvelle calédonie

Marc Ulrich - Michel Cathelineau - Marie-Christine Boiron
(GeoRessources)

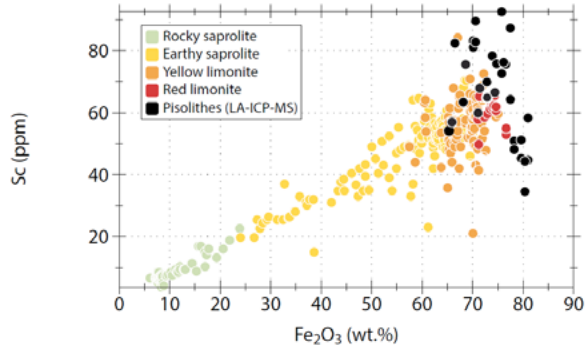
AXE Scientifique : 1/4

Enjeux scientifiques

Le projet se proposait de répondre aux questions suivantes :
- quelles sont les formations potentiellement hôtes des teneurs les plus élevées en Sc et REE
- quelles sont les teneurs en Sc et REE des roches mères et des roches issues de leur d'altération ?
- quelle est la variabilité verticale et horizontale du Sc et des REE dans les profils latéritiques ?
-l'analyse localisée du Sc est elle possible ?

Etat de l'art

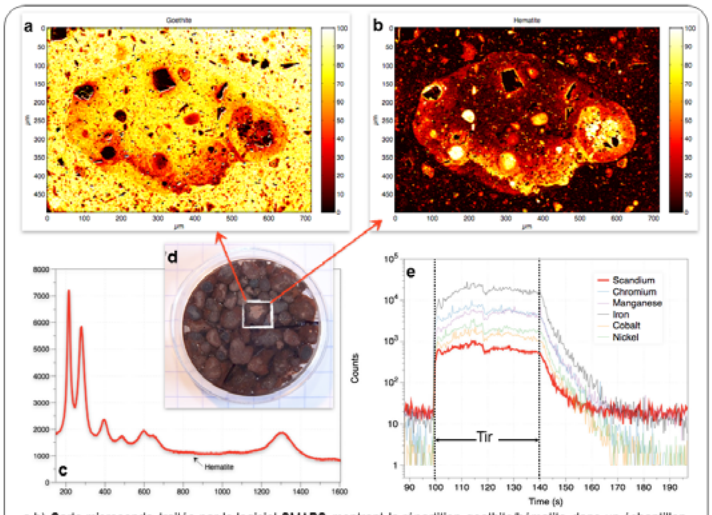
Si la Nouvelle-Calédonie est un des grands producteurs de Ni et Co (avec des réserves non négligeables en Cr), des études antérieures ont montré l'existence de minéralisations significatives en métaux du groupe de platine (Traore et al.,2008), et des enrichissements en Sc (Audet, 2008). Ces métaux sont valorisables en sous-produits de l'exploitation du Ni. Le cycle du scandium est par contre très mal connu, mis à part le fait qu'il est présent dans les pyroxènes, et qu'il s'enrichit avec le fer dans les sol résiduels (latérite).



Evolution de la concentration en Sc dans les niveaux latéritiques de Nouvelle-Calédonie (Ulrich et al., en prep.)

Approche méthodologique

La calibration de l'analyse localisée du Sc a été réalisée en ablation laser-ICP-MS en utilisant des verres NIST et de standards confectionnés à partir de poudres analysées au SARM. En amont des analyses *in situ* par LA-ICP-MS, les espèces minéralogiques en présence dans les pisolites ont été identifiées par spectroscopie raman (dissociation goethite/hématite), par microscope optique à balayage (MEB) et par cartographie chimique par microsonde électronique. Ces approches diverses ont également permis d'acquérir de nombreuses données sur la répartition et la concentration d'autres éléments d'intérêts économiques tels le Ni, Co, Mn ou Cr.



a,b) Carte microsonde traités par le logiciel SMAPS montrant la répartition goethite/hématite dans un échantillon de pisolite; c) Analyse Raman d'une hématite; d) échantillon de pisolite; e) Exemple de tir d'ablation Laser

Résultats

Les analyses géochimiques de profils développés sur différents protolithes ont été réalisées au CRPG. Les travaux ont permis d'identifier certains niveaux d'enrichissements dans les profils d'altération, en particulier l'horizon de saprolite fine (latérite jaune des mineurs) et l'horizon de transition. Les teneurs identifiées dans les profils varient de 7 ppm dans le protore harzburgitique à 75 ppm dans les latérites jaunes.

L'analyse quantitative du scandium à l'échelle d'une dizaine de microns a été calibrée jusqu'au ppm à Géoressources ce qui a permis de faire des progrès importants dans la localisation du scandium au sein des hétérogénéités des faciès de limonite rouge et des pisolites des cuirasses.

Bilan – Perspectives de développement

Ce projet a permis de déboucher sur la rédaction d'un projet multipartenaires (GéoRessources, EOST-IPG Strasbourg, ISTerre-Grenoble, BRGM, SGNC, CEREGE) soumis à l'appel d'offre du CNRT Nouvelle-Calédonie dans la thématique Nickel & son Environnement, en Novembre 2013.

Il a également permis de développer l'intérêt des industriels travaillant en Nouvelle Calédonie, notamment Koniambo SA.

Fractionnement isotopique et transfert du Ni dans les systèmes sol - plantes hyperaccumulatrices

Nicolas Estrade[1], Christophe Cloquet[1], Tenghaobo Deng[1,2,3], Guillaume Echevarria[2], Thibault Sterckeman[2], Jean Louis Morel[2]
[1]CRPG, [2]LSE, [3]SYSU

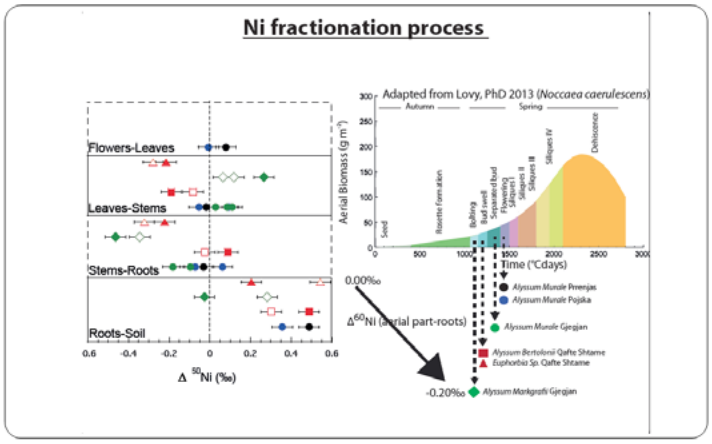
AXE Scientifique : 3/4

Enjeux scientifiques

- Comprendre les mécanismes du transfert sol-plante du Ni.
- Développer une méthode de mesure à haute précision du fractionnement isotopique du Ni.
- Évaluer le fractionnement de Ni dans des systèmes sol ultramafique - plantes métallophytes.

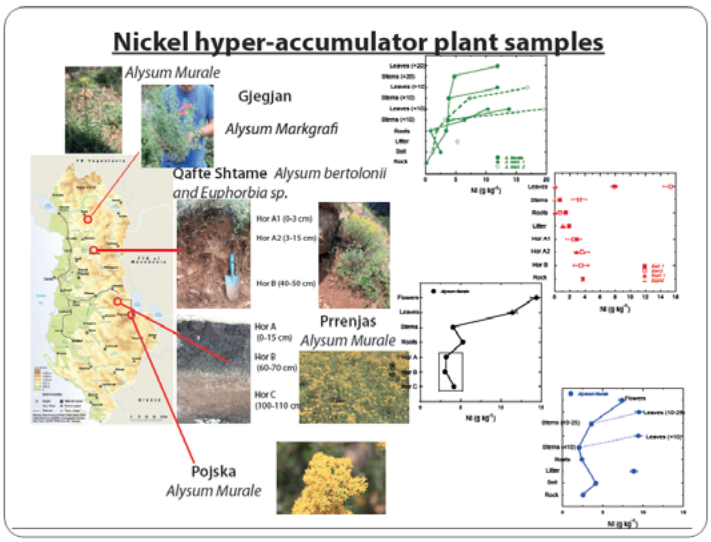
Etat de l'art

- Très peu de données sur la composition des isotopes stables du Ni et des processus de fractionnement associés en milieu continental.
- Lacunes sur la compréhension de la dynamique du Ni dans les plantes.
- Absence de méthode de dosage des isotopes stables du Ni au niveau du LabEx.



Approche méthodologique

- Echantillonnage *in situ* des roches mères, horizons des sols (dont litière et sol rhizosphérique) et organes végétaux (racines, tiges, feuilles et fleurs) sur quatre sites en Albanie.
- Calibration d'un double traceur isotopique.
- Mise en place d'un procédé de purification du Ni.
- Développement de la mesure à haute précision des isotopes du Ni dans les matrices environnementales.



Résultats

- Fractionnement des isotopes du Ni dans les sols et dans différentes parties de la plante. L'amplitude de la variation totale mesurée est d'environ 1,2 ‰.
- Composition isotopique des roches variant de 0 à 0,3 ‰.
- Dans la plante, l'enrichissement en isotopes légers serai dû au faible flux de translocation. L'absence de fractionnement dans les parties aériennes de certaines plantes, des hyperaccumulateurs à un stade phénologique avancé, serait la conséquence d'un très fort flux de translocation.
- Dans les sols, l'enrichissement en isotopes légers serait proportionnel à l'intensité de l'altération des minéraux primaires. Des mesures de fractionnement du Ni échangeable en cours devraient apporter des informations sur les mécanismes de fractionnement dans les sols et dans les racines.

Bilan – Perspectives de développement

- Obtention des premières données au niveau international sur la composition isotopique du Ni dans les systèmes sol-plante.
- Mise au point d'une nouvelle méthode d'analyse isotopique utilisable dans le cadre de futurs projets.
- Renforcement des collaborations avec l'Albanie (Université Agricole de Tirana) et avec la Chine (Université Sun Yat-sen de Canton).
- Présentation des travaux à deux conférences internationales.
- Article dans une revue à comité de lecture international en cours de rédaction.

Conseil Scientifique du 23/01/2015
Rapport mi-parcours ANR du 2/06/2015

ECOSCOPE

Plate-forme expérimentale en écologie et écotoxicologie des milieux aquatiques pour Observer – Expérimenter – Modéliser en conditions semi réelles

AXE Scientifique : 3

Simon Devin, Mickaël Danger, Christophe Pagnout, Florence Maunoury-Danger, Sandrine Pain-Devin, Philippe Wagner et Vincent Felten

Laboratoire Interdisciplinaire des Environnements Continentaux

Enjeux scientifiques

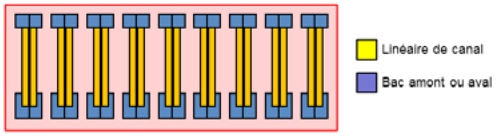
L'enjeu scientifique majeur de l'ECOSCOPE est de développer une approche d'évaluation des risques environnementaux sur du long terme (> 3 mois), à des concentrations réalistes du point de vue de l'environnement.

Ce dispositif permettra :
De simuler et renforcer les synergies en matière de physico-chimie, d'écotoxicologie et d'écologie :
- en intégrant la variabilité de l'environnement naturel comme paramètre d'interprétation des résultats
- en permettant l'étude du comportement des contaminants dans le milieu naturel

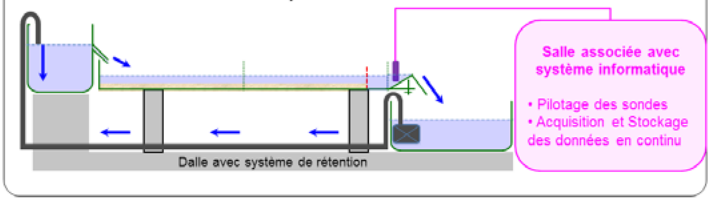
D'affiner notre compréhension des interactions entre le fonctionnement des écosystèmes et les effets liés aux contaminations

De participer à la mise en place de nouvelles méthodes d'évaluation du risque et au développement d'outils de diagnostic prévisionnels pertinents

Vue aérienne de la plate-forme ECOSCOPE



Vue latérale d'un canal expérimental



Description technique

- **18 canaux** de 3,5m de long et de section 0,25 x 0,25
- Doublure en bâche EPDM changée à chaque expérience
- Contrôle de la nature du substrat et de la mosaïque d'habitat
- **Réservoir amont** : Introduction toxique, brassage, régulation °C
- **Divisible** en 3 sous-parties de 0.95 m
- Circulation d'eau en **circuit fermé**: pompe immergée à débit réglable
- **Equipés en sondes** T°, pH, O_2, conductivité

- **Coût total** : 225 k€ HT ; Financement LABEX : 110 k€ HT

Etat d'avancement

L'ensemble des équipements est acheté (financement ZAM, CNRS, UL)

La dalle est achevée depuis octobre (financement LABEX, ZAM)

Il reste à monter les canaux, mettre en place les sondes et aménager la salle de pilotage de la plate-forme

L'ECOSCOPE devrait être complètement opérationnel pour la fin du printemps 2014

Les canaux ont déjà été testés dans une approche de dérivation de ruisseau *in situ*

Bilan – Perspectives de développement

Potentiel fédérateur important, à l'échelle de la Zone Atelier Moselle notamment
→Projet ANR ICARE déposé cette année
→ Autres projets à construire

Vitrine scientifique :
- Dispositif rare à l'échelle nationale
- Utilisation dans le cadre de la Fête de la Science
- Manifestation d'intérêt par Hydréos
- Excellence des filières d'enseignement : dispositif utilisé en TP

Projet réalisé par des entreprises lorraines :
Design des canaux par ISMA (57) et PSV (88), Instrumentation par CTB Choffel (88), BTP par ARDIZIO et SNEF (57)

Conseil Scientifique du 23/01/2015
Rapport mi-parcours ANR du 2/06/2015

Biodisponibilité, transfert, localisation cellulaire des nanoparticules manufacturées métalliques chez les invertébrés d'eau douce

Laure GIAMBERINI

Laboratoire Interdisciplinaire des Environnements Continentaux (LIEC), Metz, France

AXE Scientifique : 3

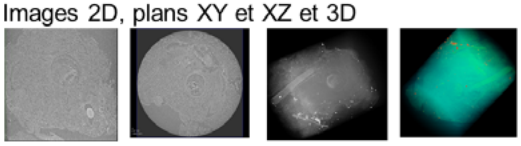
Enjeux scientifiques

La compréhension des mécanismes réactionnels aux interfaces eau/sédiments/organismes vivants reste un verrou dans le domaine de l'écotoxicité des NPs. Ces mécanismes réactionnels conditionnent la spéciation et la biodisponibilité de ces composés et leur écotoxicité.
L'étude de la biodisponibilité des NPs et du transfert des barrières biologiques, des mécanismes d'accumulation et de détoxication est essentielle à la compréhension des mécanismes écotoxiques et des risques associés.

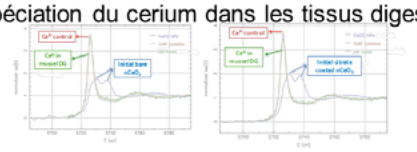
Etat de l'art

Alors que les études de toxicologie concernant les NPs progressent de façon remarquable, l'avancée des connaissances sur la dangerosité des nanoparticules est retardée par le caractère multiparamétrique des études qui doivent considérer pour chaque matériau constitutif des nanoparticules, leur taille, leur forme, leurs charges de surface, leur solubilité réductive ou oxydante liée à des propriétés de surface différentes des mêmes minéraux micrométriques
Dans l'environnement les NPs subissent des modifications de leurs caractéristiques susceptibles de moduler leur biodisponibilité et donc leur toxicité vis-à-vis des organismes vivants qui sont différentes de celle des polluants classiques.

Reconstruction des échantillons de glandes digestives de moules zébrées exposées à des NPs de CeO2
Images 2D, plans XY et XZ et 3D

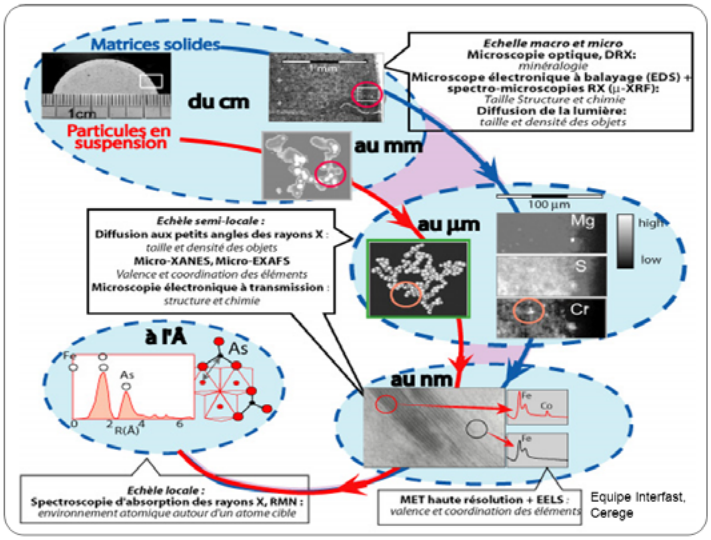


Spéciation du cerium dans les tissus digestifs



Approche méthodologique

Développer et partager les connaissances sur la bio-disponibilité/transfert des Nps au sein d'une chaine trophique complexe en prenant en compte l'exposition telle qu'elle est développée au CEREGE via la modélisation des phénomènes d'agrégation-dispersion-sédimentation
Développer des compétences en identification et localisation des nanoparticules métalliques dans les organismes en utilisant les outils de tomographie X en nano et micro résolution.
Spéciation du Ce mesurée par XAS X ray absorption spectroscopy , Ce L3-edge sur ligne FAME Synchroton à l'ESRF – (M Auffan, M Tella & O Proux)



Résultats

•Mise en place des protocoles de préparations des échantillons biologiques pour les observations et analyses en microfluorescence et micro – nano tomographie de RX.

• Nous avons obtenu les premiers résultats de reconstruction en 3D sur des échantillons de glandes digestives de moules zébrées exposées 21 jours à des NPs de CeO2 non enrobées indiquant la présence d 'éléments métalliques dans les tissus confirmée également par les analyses menées sur les mêmes échantillons en rayonnement synchrotron par A. Auffan.

•Réduction Quasi-totale de Ce^{IV} en Ce^{III} pour les 2 types de $nCeO_2$ dans les glandes digestives de bivalves indiquant les interactions redox entre $nCeO_2$ et les composés biologiques

Bilan – Perspectives de développement

La collaboration menée dans le cadre de la mobilité au Cerege apportera des connaissances originales, novatrices et indispensables dans le domaine de l'écotoxicologie des NPs et de l'évaluation de leur risque environnemental.
Elle me permettra de développer des:
• Compétences dans le domaine de l'exposition aux NPs qui pourront être partagées au LIEC
•Compétences sur une approche plus globale des risques liés aux nanotechnologies grâce à l'utilisation d'outils comme la nano et micro-tomographie X
•Renforcement des liens entre les Labex Ressources 21 et SERENADE par exemple sur les problèmes liés au recyclage des déchets contenant des nanomatériaux qui pourraient impacter la qualité des ressources

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Conseil Scientifique du 23/01/2015
Rapport mi-parcours ANR du 2/06/2015

Effets des nanoparticules manufacturées sur les invertébrés d'eau douce et leurs fonctions au sein des écosystèmes. Approche intégrée chez le gammare.

AXE Scientifique : 3

Andreï Jennifer, Pain-Devin Sandrine, Felten Vincent, Giambérini Laure & Guérold François

LIEC, CNRS-UMR 7360, Université de Lorraine, rue du Général Delestraint, 57070 Metz, France

Enjeux scientifiques

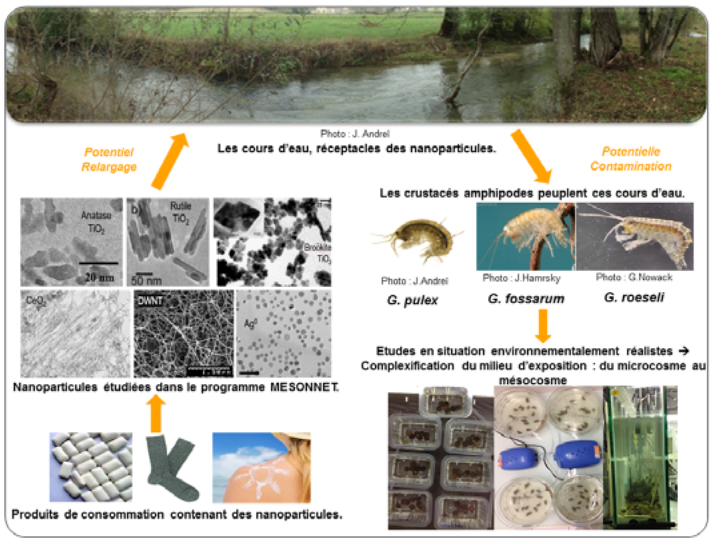
Malgré une dispersion dans l'environnement indétectable et des conséquences encore inconnues, les nanoparticules sont produites et couramment utilisées dans la vie quotidienne.

Au sein du Labex, les travaux de thèse développés ici visent à évaluer les impacts écologiques de ces nanoparticules manufacturées désormais potentiellement présentes dans les cours d'eau. Ces recherches poseront les bases de scénarios d'effets généraux liés à ces composés, pour permettre une évaluation du risque et une politique de gestion adaptée.

Etat de l'art

Les nanoparticules sont des amas de quelques milliers d'atomes possédant des propriétés inédites et étonnantes. Elles peuvent par exemple être utilisées comme agent blanchissant ($nTiO_2$) ou comme bactéricide (nAg).

Intégrateurs de l'état de l'écosystème qu'ils colonisent, les gammares sont de bons bioindicateurs de la qualité d'un cours d'eau et occupent une position clé dans son fonctionnement. L'analyse d'impact s'établit par l'étude de biomarqueurs cellulaires, physiologiques et comportementaux.



Résultats

Comportement des nanoparticules dans nos conditions :

- [nAg 60nm] 5µg/l = PAS de dissolution après 24 et 72h
- [nAg 10nm] 5µg/l = 15,8% après 24h et 10% après 72h

→ *Une toxicité liée à l'argent dissous pourrait s'ajouter à celle éventuelle de la forme nanoparticulaire.*

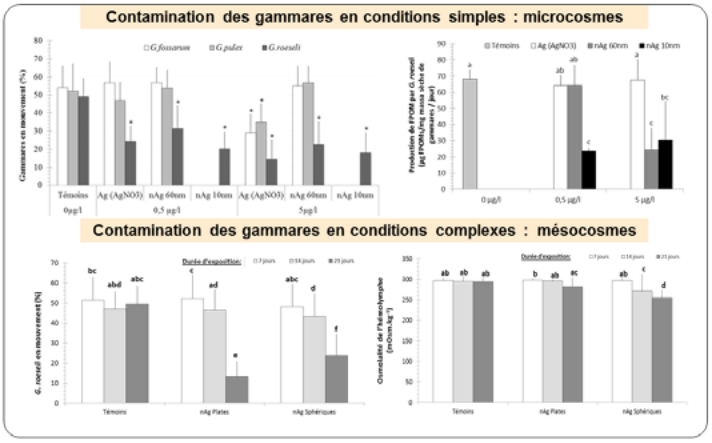
Contamination des gammares en microcosmes (nAg) :

- Locomotion des gammares :
  G. fossarum et *G. pulex* : PAS d'impact des nAg
  G. roeseli : ↘ de 17 à 31%
- Dégradation de la litière:
  G. roeseli : ↘ significative de la production de Fines Particules de Matière Organique (FPOM)

→ *G. roeseli est l'espèce la plus sensible, les nAg ont des impacts aux niveaux individuels et fonctionnels.*

Contamination en mésocosmes : nAg, $nTiO_2$, NTC):

→ *Effets dépendants du type, de la taille et de la forme des nanoparticules (nAg : effets similaires aux microcosmes).*



Approche méthodologique

Appartenant au programme international MESONNET, cette thèse a pour objectif d'évaluer les impacts des nanoparticules sur le crustacé *Gammarus sp.*

Un premier travail d'optimisation a permis d'élaborer un protocole standard pour s'approcher au mieux de conditions de contamination environnementalement réalistes. Le comportement des nanoparticules dans ces conditions a été évalué (adsorption, agrégation à la matière organique, dissolution). Ces pré-requis ont permis d'élaborer des études d'impacts sur plusieurs types de nanoparticules, plusieurs espèces et populations de gammares. Ces expériences d'exposition ont permis d'évaluer les effets de ces composés à plus ou moins long terme, dans des situations simples ou complexes par l'études de biomarqueurs cellulaires à fonctionnels.

Bilan – Perspectives de développement

Après des études déjà réalisées sur *Gammarus sp.* et l'argent dissous (Thèse J. Arce Funck), ce projet a permis d'approfondir et de compléter nos connaissances sur les risques liés à une contamination argentique. L'étude d'impacts à des échelles d'intégration supérieures présente une forte originalité dans le « monde des nanoparticules » plutôt habitué à évaluer leurs effets au travers de la mortalité des organismes.

Ce projet permet donc de considérer et d'accroître l'aspect écologique des questionnements actuels, de rajouter du « éco » dans écotoxicologie et ainsi d'avoir une meilleure vision des risques globaux que peuvent induire les nanoparticules.

C'est dans ce sens que sont développés l'ensemble des travaux afin de répondre aux objectifs du Labex ainsi que des laboratoires d'OTELo.

Conseil Scientifique du 23/01/2015
Rapport mi-parcours ANR du 2/06/2015

Uptake and intracellular distribution of Cr(III) in freshwater algae

VIGNATI Davide A.L., AHARCHAOU Imad, BATTAGLIA Eric, FORTIN Claude

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Institut National de la Recherche Scientifique (INRS), Québec, Canada

AXE Scientifique : 3

Enjeux scientifiques

- Quelle est l'influence du pH sur le taux d'internalisation du chrome trivalent -Cr(III)- par les algues d'eau douce ?
- Quelle est la distribution intracellulaire du Cr dans les cellules algales?
-Les formes Cr(III) et Cr(VI) ont-elles les mêmes cibles intracellulaires et les mêmes mécanismes de détoxication?

Cette action s'insère dans le projet LabexR21 sur « Ni et éléments associés ». L'étude du Cr(III) peut servir de modèle pour les terres rares qui sont aussi une priorité du LabexR21.

Etat de l'art

Parmi les formes chimiques dominantes dans les eaux de surface, le Cr(III) est considéré moins toxique que le Cr(VI). Toutefois, des études menées par notre groupe de travail et par d'autres équipes suggèrent que la toxicité du Cr(III) serait sous-estimée. De ce fait, la biodisponibilité du Cr(III) doit être mieux étudiée. Aucune connaissance définitive n'existe sur les formes chimiques qui contrôlent l'accumulation du Cr(III) par les algues, ni sur la distribution intracellulaire du Cr dans des algues exposées au Cr(III) et au Cr(VI).

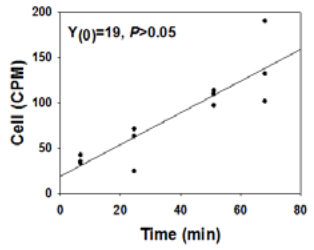


Figure 1. Activité du 51-Cr(III) mesuré à l'intérieure de cellules de *Chlamydomonas reinhardtii* en fonction du temps d'exposition.

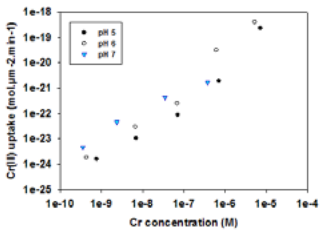
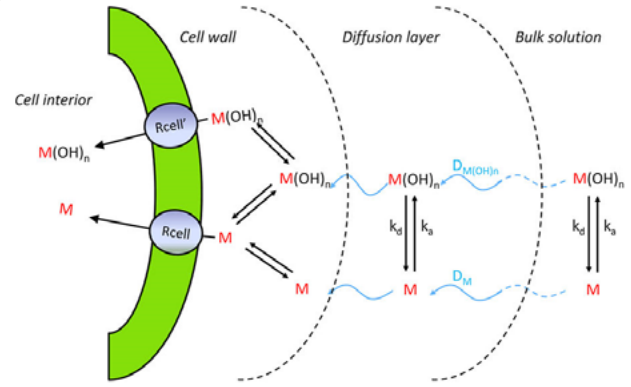


Figure 2. Taux d'internalisation du 51-Cr(III) par *Chlamydomonas reinhardtii* en fonction de la concentration et du pH.

Approche méthodologique

Objectif 1: Taux d'internalisation du Cr(III) par l'algue *Chlamydomonas reinhardtii*. Les algues sont exposées au ^{51}Cr(III) pendant 60 minutes. Les expériences sont effectuées à pH 5, 6 et 7 afin de déterminer l'influence du pH sur l'internalisation du Cr(III). Le Cr internalisé est mesuré par comptage gamma.

Objectif 2 (en cours): Distribution intracellulaire du Cr dans C. *reinhardtii*. Les algues sont exposées au Cr(III) et au Cr(VI) pendant 72 heures. La distribution parmi les différentes structures cellulaires est déterminée par centrifugation différentielle et analyse des fractions par comptage gamma.



Aperçu des mécanismes régissant le transport des éléments traces à travers les membranes cellulaires. M, métal; D, coefficient de diffusion ; R, récepteur cellulaire. La paroi cellulaire (cell wall) est représentée par la distance entre la membrane plasmique (en vert) et la 1ère ligne pointillée (d'après Crémazy et al, 2013, Environ. Sci. Technol., 47: 2408).

Résultats

Les expériences menées jusqu'au 25 octobre 2014 montrent que:

-L'internalisation du Cr(III) est linéaire entre 5 et 70 minutes. Ceci indique que le flux d'internalisation est constant et qu'il n'y a pas d'excrétion significative du Cr(III) (Fig. 1)

-La concentration du Cr(III) adsorbée sur les parois atteint très rapidement un équilibre. Ceci indique que l'adsorption du Cr(III) sur les algues est très rapide et confirme que c'est une cinétique d'internalisation et non d'adsorption qui est mise en évidence en Fig. 1.

-Le taux d'internalisation du Cr(III) augmente proportionnellement à sa concentration (de 1nM à 10 µM) pour un pH donné. Ceci peut s'expliquer par des changements dans la spéciation du Cr(III), par une compétition entre le Cr(III) et les protons H+ pour les sites d'internalisation et/ou par une modulation de la cinétique d'internalisation (Fig. 2).

Bilan – Perspectives de développement

Ces travaux ont permis de poursuivre la collaboration entre le LIEC et l'INRS (invitation de C. Fortin par le LabexR21 en juin-août 2013). Pour le LIEC, ils s'intègrent dans les travaux de thèse de M. Imad Aharchaou sur la biodisponibilité du Cr(III).

La deuxième partie de l'étude (localisation intracellulaire du Cr – objectif 2) permettra d'identifier les sites d'accumulation du Cr à l'intérieur des cellules exposées au Cr(III) ou au Cr(VI).

Les laboratoires partenaires étudient la possibilité d'une proposition de projet dans le cadre du partenariat stratégique en matière d'enseignement et recherche du Conseil Franco-Québécois de coopération universitaire.

Conseil Scientifique du 23/01/2015
Rapport mi-parcours ANR du 2/06/2015

Ecotoxicité des Terres Rares : Etude comparative à l'aide d'une batterie de biotests

Veronica Gonzalez[1], Davide.A.L. Vignati[1], Corinne Leyval[2], Laure Giamberini[1]

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AXE Scientifique : 3

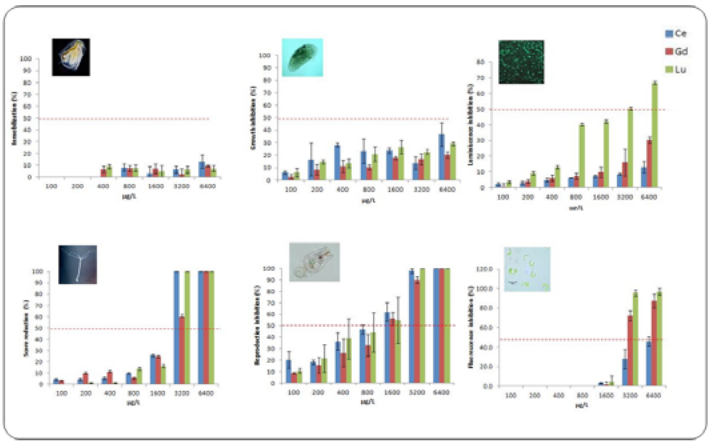
Enjeux scientifiques

Les Terres Rares (REEs) sont un groupe d'éléments présentant un large spectre d'applications dans différents secteurs industriels mais pour lesquels peu **d'informations écotoxicologiques** sont disponibles
Les objectifs sont de (1) **synthétiser les données** écotoxicologiques actuellement **disponibles** sur les REEs afin d'identifier les verrous et les besoins de recherches et (2) vérifier si l'écotoxicité des REEs varie d'une manière **prédictible** selon leur caractéristiques chimiques et selon quelle tendance.

Etat de l'art

On considère que **l'homogénéité chimique** des REEs prédit **leur toxicité** mais actuellement **aucun consensus n'**existe au regard des variations de leur écotoxicologie

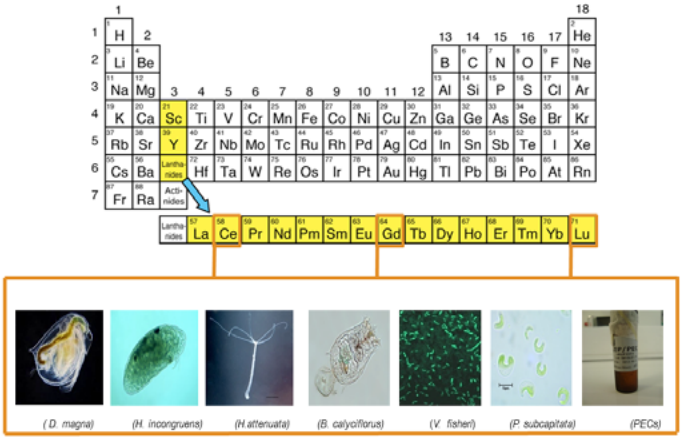
Les données écotoxicologiques concernant les REEs sont rares et hétérogènes. Dans les écosystèmes aquatiques , les études comparatives sont rares. Dans les sols, la plupart des études se focalisent sur les mélanges d'éléments.



Approche méthodologique

1. Revue bibliographique Les informations écotoxicologiques concernant les éléments les moins étudiés ont été collectées. Les jeux de données disponibles dans la littérature ont été analysés.

2. Partie expérimentale : 3 REEs ont été sélectionnés : un léger [Cerium (Ce)], un lourd [Lutetium (Lu)], et un intermédaire [Gadolinium (Gd)]. The toxicité de ces 3 métaux a été évaluée sur plusieurs espèces aquatiques modèles.



Résultats

1. En comparaison avec celle des autres métaux, l'écotoxicité des REEs reste encore à étudier. Les manques de données concernent l'écotoxicité des éléments lourds dans les systèmes aquatiques et les effets individuels dans les sols.

2. Les Crustacés (*D. magna* & *H. incongruens*) sont les moins sensibles et les rotifères (*B. calicyflorus*) et cnidaires (*H. attenuata*) sont les plus sensibles à l'exposition des REEs.

3. L'écotoxicité des REEs augmente significativement avec le numéro atomique uniquement chez les bactéries (*V. fisheri*) et les algues (*P. subcapitata*).

4. Des études écotoxicologiques des REEs supplémentaires sont nécessaires avant d'établir un schéma général de toxicité.

Bilan – Perspectives de développement

Grâce aux connaissances et compétences ainsi développées, le LIEC émarge dans de nouveaux programmes et s'enrichit de nouvelles collaborations :
- Réseau Lorlux : Lorraine – Luxembourg
- COST: European cooperation in science and technology
- Workshop on Mineral-Microbe interactions in concentration and fractionation of rare earth elements (Contact Simon Gregory, British Geological Survey ,Nottingham UK)

Les travaux menés dans le cadre de ce contrat post doctoral constituent un premier socle de connaissances sur l'écotoxicité des lanthanides permettant au laboratoire de répondre à un appel d'offre de l'ANR 2013 dans ce domaine de compétence en collaboration avec le Centre de recherche public Gabriel Lippmann

Conseil Scientifique du 20/01/2014
Rapport annuel mi-parcours ANR du 2/06/20

Impacts cellulaires de l'acide gadotérique (agent de contraste gadoliné) sur des cellules humaines en culture (HEK 293T)

PARANT Marc , PETIT Elisabeth, SOHM Bénédicte, BATTAGLIA Eric
Laboratoire Interdisciplinaire des Environnements Continentaux - LIEC

AXE Scientifique : 3

Enjeux scientifiques

Quels sont les risques biologiques associés à l'utilisation de terres rares dans des produits manufacturés ?
L'impact biologique de tels composés dans leurs conditions d'accumulation environnementale nécessite-t-il la prise en compte de précautions lors de leur utilisation ?
Ces questions s'appliquent aujourd'hui aux agents de contraste gadolinés qui après plus de 20 ans d'utilisation dans le secteur du diagnostic médical sans aucun contrôle de leurs rejets sont aujourd'hui présents dans les différents compartiments aquatiques de l'environnement y compris parfois l'eau du robinet.

Etat de l'art

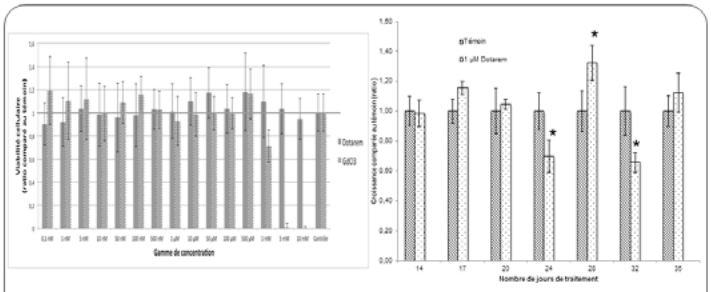
Depuis 1988, date du début de leur utilisation, les agents de contraste gadolinés ont été optimisés afin de gagner en efficacité et en sécurité pour le patient lors de l'examen de diagnostic médical.

Aucune approche ne s'est cependant intéressée à l'action de ces composés sur la matière vivante dans les conditions qui résultent de leur rejets dans l'environnement, conditions très différentes de l'examen en concentration et en biodisponibilité.

Cycle du gadolinium dans l'environnement suite à son utilisation dans le milieu médical

Approche méthodologique

L'objectif de ce projet est de suivre l'impact de l'agent de contraste le plus retrouvé dans l'environnement (acide gadotérique, - DOTAREM®) sur la matière vivante.
Pour ce faire nous avons suivi l'impact de ce composé sur la croissance de cellules humaines en culture (HEK 293T) en nous intéressant particulièrement aux concentrations présentes dans l'environnement dans le cas d'exposition aigües ou chroniques.
Afin d'évaluer des variations concernant la progression du cycle cellulaire pendant la croissance, une approche par cytométrie en flux a été menée sur les situations les plus intéressantes.
Une étude de bioaccumulation du gadolinium sur ces cellules a été menée après 48 h d'exposition à l'acide gadotérique 1µM.



Impact du Dotarem® et du chlorure de gadolinium sur la viabilité des cellules HEK293T après 48h d'exposition
Les cellules HEK293T ont été ensemencées dans des plaques 96 puits à hauteur de 2500 cellules par puits. Après 24h d'incubation, elles sont exposées durant deux jours à différentes concentrations de Dotarem® ou de $GdCl_3$. La mesure de viabilité est réalisée par un test MTT. Cette expérience a été répétée trois fois. La viabilité moyenne par rapport au témoin est présentée ci-dessus.

Suivi de croissance cellulaire durant une exposition chronique à 1 µM de Dotarem®
La quantité des cellules HEK293T a été évaluée par comptage après chaque repiquage au cours d'une exposition chronique à 1 µM de Dotarem®. La quantité moyenne de cellule est rapportée à celle du témoin. Aucune variation n'est observée avant 14 jours. * : différence significative par rapport au témoin (test t de Student, $p<0,05$, n=3)

Résultats

Pas de modification significative de la croissance des cellules HEK 293T (par comptage, test MTT ou test d'induction de prolifération) en présence d'acide gadotérique quelque soit la concentration utilisée en situation d'exposition aigüe.

En situation d'exposition chronique (1µM d'acide gadotérique, culture de cellules HEK 293T sur 35 jours), la croissance n'est pas significativement modifiée mais laisse apparaître une plus grande variabilité au-delà de 20 jours de culture, suggérant des conditions perturbantes pour les cellules. Aucune modification des phases G0/G1 ou G2/M n'a été observée par cytométrie en flux lors des situations perturbées.

Une faible fraction de gadolinium est retrouvée dans la fraction cellulaire après 48h d'exposition à 1 µM d'acide gadotérique. Ces résultats suggèrent soit une fixation membranaire forte, soit une internalisation de l'acide gadotérique ou de l'ion gadolinium seul et méritent d'être approfondis.

Bilan – Perspectives de développement

Ces premiers travaux ont permis d'établir des collaborations avec :
-L'Institut de Chimie Moléculaire de Reims - ICMR – UMR 7312 – Groupe Chimie de Coordination – Pr Françoise Chuburu,
- Le laboratoire d'hydrologie de Nancy ANSES (J-F Munoz – -J-S Py)
- Le Centre de Recherche Gabriel Lippmann Luxembourg (C. Guignard).

Les collaborations se développent aujourd'hui sous la forme :
- d'une thèse initiée en Octobre 2013 (co-financement Région Lorraine – Ressources 21)
- d'un projet EC2CO soumis en septembre 2013
- d'une participation au développement d'un projet ANR piloté par D. Vignati.

Conseil Scientifique du 23/01/2015
Rapport mi-parcours ANR du 2/06/2015

Evaluation de la présence des agents de contraste au Gadolinium sur le secteur lorrain.

Perrat Emilie, Cossu-Leguille Carole et Parant Marc (LIEC)

Rosin Christophe (ANSES)

AXE Scientifique : 3

Enjeux scientifiques

Le Gadolinium est un métal stratégique qui permet la création d' agents de contraste (AC-Gd) employés en IRM, c'est donc une technologie à haute valeur ajoutée. En France, il n'y a pas d'études sur le devenir de ces AC-Gd dans l'environnement ni pour comprendre leur impact sur les organismes aquatiques?

Cette étude s'inscrit dans les objectifs principaux du LabEx R21 d'un point de vue scientifique (réalité en Lorraine et impact écotoxicologique), et socio-économique par l'effet qu'elle aura au niveau des propositions d'amélioration concernant le retraitement dans les stations d'épuration (STEP).

Etat de l'art

Lors d'un examen IRM et suite à une injection d'AC-Gd, leur concentration décroit dans les urines de plus de 98% le lendemain de l'examen (Kummerer et *al.*, 2000). Ces AC-Gd se retrouvent alors dans les STEP. Telgman et *al.* (2012) et Birka et *al.* (2013) ont montré qu'environ 80% du gadolinium retrouvé en rivière est présent sous la forme d'AC-Gd employé en IRM. Notamment, ceux de type macrocycliques (Dotarem® et Gadovist®) à cause de leurs stabilités cinétique et thermodynamique (Port et *al.*, 2008 ; Bonnet 2006).

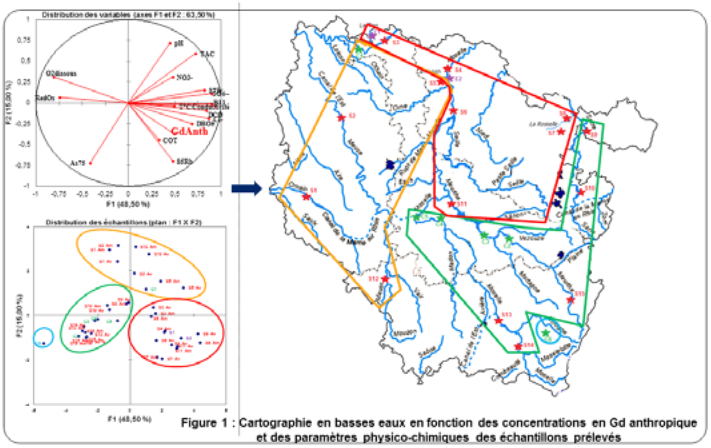


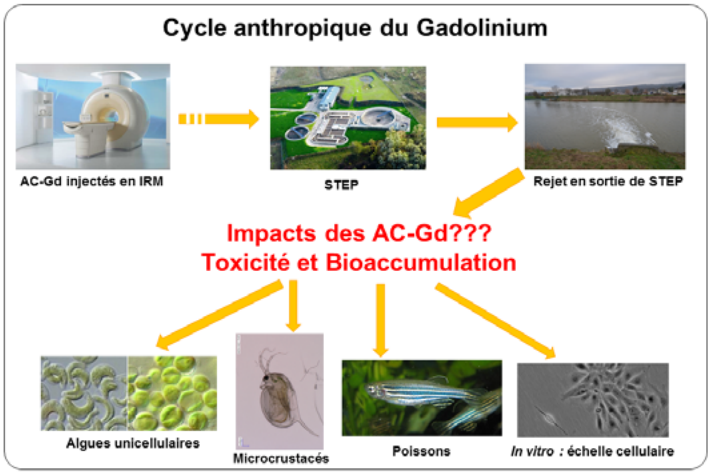
Figure 1 : Cartographie en basses eaux en fonction des concentrations en Gd anthropique et des paramètres physico-chimiques des échantillons prélevés

Approche méthodologique

Cette étude permettra de connaître les niveaux de contamination aux AC-Gd en Lorraine aux périodes de hautes et basses eaux.

Pour cela nous avons mis en place une campagne d'échantillonnage lors de ces deux périodes (Juillet 2014 et Janvier 2015). Les analyses des échantillons sont réalisées par ICP-MS (quantification des terres rares et du Gd anthropique) et par l'étude des paramètres physico-chimiques.

A partir des concentrations réelles en Gd observées, nous établirons une gamme de concentrations qui nous servira de référence pour effectuer les tests écotoxicologiques sur différents organismes aquatiques : algues, daphnies, poissons.



Résultats

Un premier screening des concentrations en terres rares dans les cours d'eau lorrains (février 2014) a montré la présence du Gd et la part en Gd qui est d'origine anthropique.

Nous avons réalisé une cinétique de prélèvements qui à confirmé les résultats observés dans la littérature, c'est-à-dire que **les concentrations en Gd sont en moyenne plus élevées le matin du mardi au vendredi**.

Lors de la campagne en Basses eaux (juillet 2014) :

- **[Gd]total > 100ng/L pour la moitié des échantillons prélevés sur l'ensemble des sites.**
- L'ACP des sites effectuée pour les différents paramètres testés et les **concentrations en *Gd anthropique*** montre une **séparation géographique en 4 groupes : site de référence (0 ng/L), sites les plus « propres » (~33 ng/L), sites intermédiaires (~52 ng/L) et sites les plus « sales » (~85 ng/L)** (Cf figure 1).

Les concentrations en *Gd total* à la sortie de la STEP de Nancy **varient de quelques dizaines de ng/L à plus de 10 µg/L** sur 2 semaines de prélèvements réguliers et **la part du *Gd anthropique*** représente entre 81 et 99% du *Gd total*.

Bilan – Perspectives de développement

Ces travaux ont permis de collaborer avec le laboratoire d'Hydrologie de l'ANSES à Nancy et l'Institut de Chimie Moléculaire de Reims (ICMR).

Nous avons montré que les sites dits les plus « sales » sont situés autour des 2 principales villes de Lorraine (Nancy et Metz). Ceci sera vérifié par la campagne d'échantillonnage de janvier 2015.

D'autre part, nous avons déterminé une gamme de concentrations en Gd environnementalement réaliste pour les tests écotoxicologiques (de quelques ng/L à 10µg/L).

Les résultats seront présentés sous forme de publications scientifiques. Nous proposerons aussi une communication lors du meeting de la SETAC qui aura lieu en Mai 2015 à Barcelone.

Conseil Scientifique du 23/01/2015
Rapport mi-parcours ANR du 2/06/2015

LIEC
GéoRessources
LSE
GRESE

Analyse de la mobilité de l'antimoine en contexte minier

Bauda Pascale[1], Masfaraud Jean-François[1], Montarges Emmanuelle[1], Zegeyé Asfaw[1], Aran Delphine[1], Muller Serge[1], Mustin Christian[1], Billard Patrick[1], Filella Montserrat[1], Boiron Marie-Christine[2], Echevarria Guillaume[3], Brill Hubert[4], Grybos Malgorzata[4], 1 LIEC UMR 7360 CNRS-UL, 2 Géoressources UMR 7359 CNRS UL , 3LSE UMR 1120 INRA-UL, 4 GRESE Université de Limoges .

AXE Scientifique : 3

Enjeux scientifiques

Objectif scientifique : Identifier les facteurs contrôlant la mobilité de l'antimoine en contexte minier.
Enjeux : exploitation propre et durable, valorisation des déchets
Projet fédérateur inter – actions prioritaires

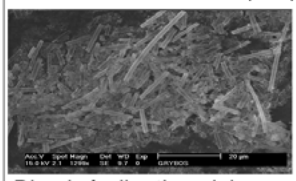
Apport du projet de recherche:
Identification d'un site atelier adapté pour étudier in situ la mobilité de l'antimoine, validation de méthodologies pour caractériser au laboratoire la mobilité de l 'antimoine et ses facteurs de contrôle.

Etat de l'art

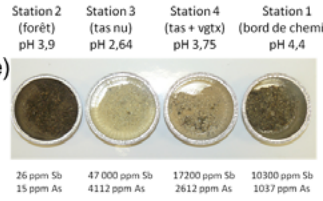
L'antimoine est un métal stratégique dont l'utilisation industrielle est croissante. Les concentrations environnementales en antimoine résultant des activités minières excèdent fréquemment les seuils autorisés (Filella et al., 2009 a, c Reimann et al., 2010). Les états d'oxydation III et V sont les plus fréquents, Sb(V) serait plus mobile dans les sols neutres ou alcalins (Johnson et al., 2005), Sb(III) serait adsorbé sur les oxyhydroxydes de fer et de manganèse (Wilson et al., 2010). L'influence des microorganismes sur la mobilité de l'antimoine n'est pas connue.

Bioaccumulation d'antimoine dans les plantes sur le site de la mine de Goesdorf (Lux)

plantes Goesdorf	feuilles Sb (ppm)	racines Sb (ppm)
Genet	64,38	35,00
Pissenlit	28,38	7,88
Rumex	56,00	32,00
Séneçon	92,38	50,50
Dactyle	21,63	46,63



Biominéralisation riche en As à l'exhaure de mine (la Bessade)



Contamination des sols sur le site de la Bessade district de Brioude-Massiac

Approche méthodologique

Etudes in situ : 2 campagnes de terrain ont été réalisées sur d'anciennes mines de Sb
-Goesdorf (Luxembourg) : identification d'un végétal modèle
-La Bessade (Cantal) : identification d'un site atelier accessible avec une problématique de mobilité de l'antimoine et d'arsenic

Développement méthodologigue: construction des dispositifs utilisant des membranes de Donnan (DMT) anioniques pour le suivi temporel des espèces oxyanions libres en solution
Des modèles minéraux porteurs de Sb de type goethite et ferrihydrite ont été envisagés pour des études in-vitro.

Résultats

-Des plantes rudérales pionnières tolérantes a Sb ont été identifiées sur des déchets miniers, leur capacité à accumuler Sb a été définie. Le Séneçon est proposé comme modèle.

- Le site de la Bessade (district de Brioude Massiac) est proposé comme site atelier accessible, une problématique de mobilité de l'antimoine et dans une moindre mesure de l'arsenic y a été identifiée.

- Une empreinte génétique des communautés microbiennes adaptées à Sb a été réalisée sur l'ensemble des prélèvements solides sur le site de la Bessade. La structure de ces communautés est influencée par le niveau de contamination leur identification par pyroséquençage est en cours.

- Bilan – Perspectives de développement

Ce premier projet d'un an sur la mobilité de l'antimoine a permi de fédérer 3 laboratoires et 3 actions prioritaires du labex, ainsi qu'à l'échelle nationale des collègues du GRESE et à l'échelle internationale Montserrat Filella (université de Genève, en mobilité pour 6 mois au LIEC). Des analyses sont encore en cours d'acquisition sur La Bessade et sur le modèle minéral de synthèse, toutefois les résultats acquis sur le site de La Bessade vont permettre de décrire une situation de mobilité de l'antimoine et de l'arsenic valorisable en terme de publication.

Les acquis du projet permettent d'envisager sur la mobilité de Sb en contexte minier la construction d'un projet plus ambitieux dans le cadre de l'année antimoine du Labex.

Conseil Scientifique du 23/01/2015
Rapport mi-parcours ANR du 2/06/2015

Impact de la bioréduction d'oxyde de fer substitués sur la re-mobilisation des métaux

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Introduction

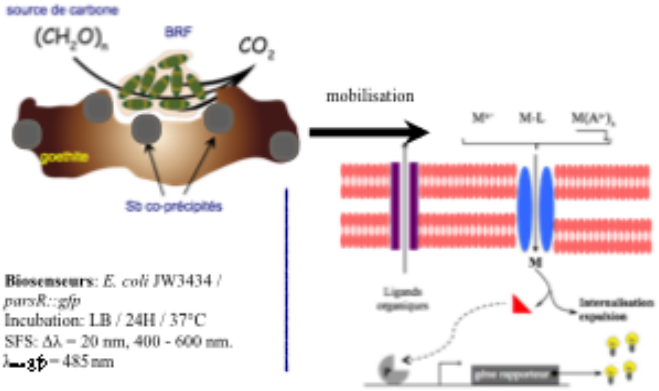
L'antimoine est le neuvième métal le plus exploité à des fins industrielles (alliages anti-friction, des plombs de chasse, ignifugeant…) même s'il est considéré comme un polluant prioritaire (5 µg L^{-1} pour la potabilité de l'eau, EUC, 1998). Dans l'environnement, l'antimoine interagit avec les oxydes de fer soit en se sorbant à la surface du minéral soit en se substituant au fer dans la structure cristalline ce qui pourrait réduire sa mobilité. Toutefois, des études approfondies sont encore nécessaires avant de considérer le piégeage de l'antimoine par les oxydes de fer comme un moyen pérenne de mobilisation de l'antimoine.

L'objectif de ce travail était de synthétiser des geothites substituées par l'antimoine et d'étudier la mobilisation biochimique du métal inclus dans le minéral et d'en évaluer la fraction biodisponible *via* l'utilisation de biosenseurs bactériens.

Approche méthodologique

Synthèses de goethite-Sb: (1) co-précipitation de $Fe(NO_3)_3$, ($K[Sb(OH)_6]$) par du KOH et vieillissement à 70°C pendant 72H -> **goeth-Fh-Sb**. (2) co-précipitation de ($FeSO_4$) ($K[Sb(OH)_6]$) par du NaOH et oxydation à l'air -> **goeth-Gr-Sb**.

Bioréduction: une bactéries respirant le Fe^{III} (BRF) en anaérobie *S. oneidensis* (10^8 CFU mL^{-1}). Source électrons = méthanoate; milieu de culture = MOPS; pH = 7.2; T°C = 28.



Biosenseurs: *E. coli* JW3434 / *parsR::gfp*
Incubation: LB / 24H / 37°C
SFS: Δλ = 20 nm, 400 - 600 nm.
λ_{em} *gfp* = 485 nm

Spectre de Fluorescence Synchrone (SFS)

↧

Traitement du Signal

$$\overbrace{S(a,\lambda)}^{SFS\ Data} = \sum_{i=1}^{n} \underbrace{a_i}_{\substack{promotor \\ response}} \circ \underbrace{s_i(\lambda)}_{\substack{reporter \\ spectra}}$$

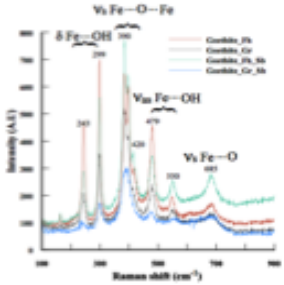
$$a = [Sb^{III\ or\ V}]$$

Résultats

Caractérisation des minéraux synthétisés

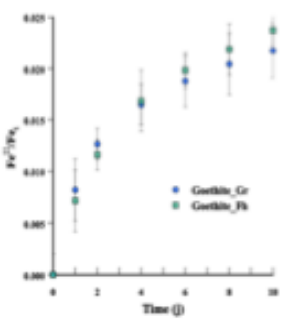
Echantillions	Longueur de cohérence (Å)	Substitution (%)
goeth_Fh	444	
goeth_Fh_Sb	360	0.5
goeth_Gr	341	
goeth_Gr_Sb	235	3.5

Les longueurs de cohérence ont été déterminées à partir de l'élargissement des pics de DRX en utilisant la formule de Sherrer. Les % de substitutions sont déterminés par ICP-MS après dissolution à l'acide (HCl 6M)

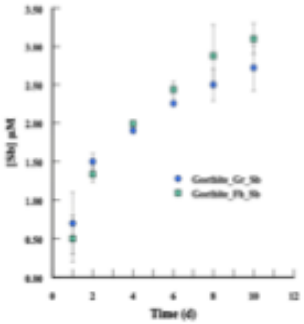


Spectre Raman de goethites synthétisées. Les bandes sont caractéristiques d'une goethite et l'élargissement des bandes est dû à la substitution du Fe^{III} structural par Sb

Bioréduction des goethites substituées



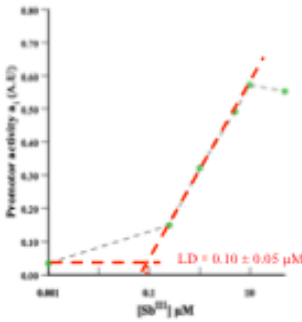
Bioréduction des goethites substituées par S. oneidensis au cours du temps

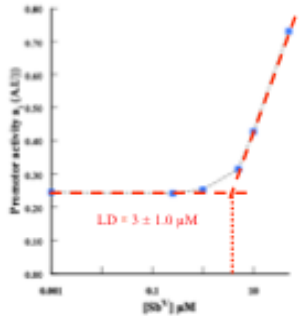


Mobilisation de l'antimoine en solution au cours de la bioréduction des goethites substituées.

- faible vitesse et faible taux de réduction des geothites substituées -> Fe^{II} structural peu labile malgré les substitutions.
- même profil et faible mobilisation (= 3 µM) de l'antimoine quelque soit le % de substitution après 10 jours de réduction.

Biodisponibilité de l'antimoine





Réponse dose dépendante du promoteur pArs en fonction de la spéciation de l'antimoine; à gauche en présence de l'antimonite (Sb^{III}) et à droite en présence de l'antimonate (Sb^{V}).
LD = limite de dosabilité ou de détection. Les concentrations sont exprimées en µM et reportées en échelle logarithmique, gamme [0-50µM].

- promoteur *pArs* sensible à l'antimonite et à l'antimonate (Sb^{III} et Sb^{V}).
- limite de dosabilité ou de détection = 0.10 µM et 3 µM pour Sb^{III} et Sb^{V}.
- promoteur *pArs* plus sensible à la forme trivalente.

Perspectives

- Conception de biosensurs actifs (*i.e.* ferri-réducteur et anaérobie): *S. putrefaciens / oneidensis pArs::ecfbfp* pour étudier *in situ* la mobilisation de l'antimoine lors de la bioréduction d'oxydes de fer substitués.
- Développer de biosenseurs pour la détection multiples de mélanges de métaux stratégiques Ni / Co / As en utilisant plusieurs protéines fluorescentes.
- Tester et valider la robustesse des biosensurs dans le cadre de contaminations minières poly-métalliques.

Conseil Scientifique du 20/01/2014
Rapport annuel ANR du 20/01/2014

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