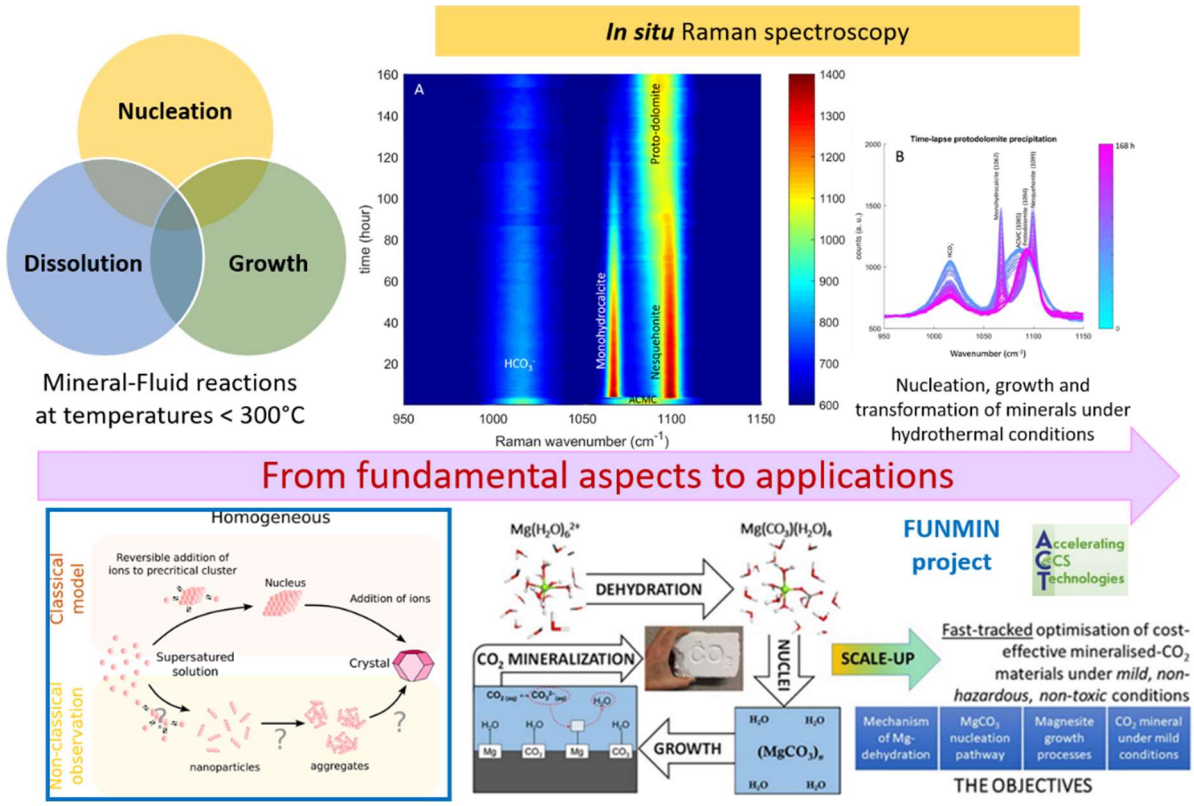
# Nucleation, growth and transformation of mineral phases under mild and hydrothermal conditions – from fundamental to applied concepts

## PREFACE

My **NAISSANCE** program was developed a new understanding and technologies to progress on the nucleation, growth and transformation of mineral phases widely used in the modern world such as carbonates, phosphates, iron oxyhydr(oxides), sulphates and clay minerals. All these minerals play also a crucial role in the global cycle of major and trace elements (including the so-called strategic elements) in the Earth and other telluric planets. In general, these secondary minerals are formed by aqueous alteration of primary minerals such as anhydrous silicates contained in rocks (e.g., peridotites, basalts, granites….) but their formation conditions are frequently ambiguous (e.g., serpentines and clays), poorly known (e.g., carbonates enriched with Earth rare elements, bastnäsite) or enigmatic (e.g., dolomite). In this perspective, the experimental tracking on the nucleation, growth and transformation of strategic minerals under mild and hydrothermal conditions remains a crucial challenge in Geosciences, Chemistry and Materials Science. **NAISSANCE** research program was consecrated to design and develop independent but complementary experimental setups to simulate a diversified panel of mineral-fluid reactions and then to provide insights on the following questions: (1) What is the reactivity of $CO_2$ during hydrothermal alteration of ultrabasic minerals? (2) What are the roles of amorphous and crystalline transient phases during the formation of minerals? and (3) What is the role of water dynamic in unsaturated gas-solid systems at low T (<0°C) and high T (>100°C)? In this general context, my **NAISSANCE** program has provided new insights on the carbonation and serpentinization processes with relevance in hydrothermal systems as well as $CO_2$ capture, utilisation and storage (CCUS). Moreover, the experimental and theoretical studies over the last decade have provided evidence for the existence of multi-step nucleation pathways and aggregation processes that take place during the early stages of mineral formation. These multi-step pathways contrast with the over-simplified traditional view described by the classical nucleation theory (CNT); based exclusively on the nucleation from oversaturated systems and behaviour of size of nucleated particles. Experimental tracking of these pathways by *in situ* Raman spectroscopy in real time, which is challenging because of the small size – in the nm range – and short lifetimes of some of the precursors and/or transient phases, was the core of my research activities in the last ten years and will continue for next five years as described in the **CLEANER** proposal, a simplified graphical abstract is also illustrated below.

The **NAISSANCE** program and strategy in the last ten years was in line with the various topics existing at the ISTerre that concerns mainly the geochemistry and mineralogy teams and the cross-disciplinary axis on the energy transition. Moreover, my leadership in various management and scientific tasks in geochemistry team (as scientific supervisor from 2017 to 2019), in ANR coro (2012-2015), three PhD French thesis (2011-2019), FUNMIN (European collaborative project from 2019 to 2023), CEMENTO project (PEPS-CNRS 2023), SOLUTION project (MITI-CNRS 2024-2025) and various graduated/ungraduated students (M2, M1, DUT, BUT, L2) have strongly contributed to my current perspective on the nucleation, growth and transformation of minerals. In this way, from 2019 a cross-disciplinary research group at ISTerre on the mineral nucleation was created during my leadership in

geochemistry team, with strong national and international collaborations as implemented from my arrival to Grenoble in 2006, working on the nucleation and growth processes of minerals and crystalline particles under mild and hydrothermal conditions; and processes preferentially monitored in real time by Raman spectroscopy and/or imagery (optical and atomic force microscopy). Obviously, the mass balance simulation by a classical nucleation approach and synchrotron measurements by time-resolved X-ray scattering/diffraction were also performed during of execution of my research program.




*Graphical abstract of **NAISSANCE** summarizing the chemical processes investigated under mild and hydrothermal conditions, with fundamental relevance on the classical and non-classical nucleation of crystals, and the application to magnesite synthesis concerning the carbon capture, utilisation and storage (CCUS). Strategically, I note that the research on the nucleation of minerals or crystal phases needs the monitoring in real-time; in this research program the Raman spectroscopy was suggested as a powerful technique (see below), but this program was open to other real-time techniques and also open to numerical simulations (preferentially in collaboration).*

## I. Context and positioning

The nucleation, growth and transformation of minerals and crystals are widely investigated in the modern world because their understanding concerns several societal applications in catalysis, water depollution, mineral processing, protein purification, drugs synthesis, concrete performance, cement hydration, etc. (e.g., 1-3). In natural media, the mineral formation and alteration play also a crucial role in the global cycle of major and trace elements (including the so-called strategic elements). In general, the so-called secondary minerals are formed by aqueous alteration of primary minerals such as anhydrous silicates contained in rocks (e.g., peridotites, basalts, granites….) but their formation conditions are frequently ambiguous (e.g., serpentines and clays), poorly known (e.g., carbonates enriched with Earth rare elements, bastnäsite) or enigmatic (e.g., dolomite). In this perspective, the experimental tracking on the nucleation, growth and transformation of strategic minerals under mild and hydrothermal conditions remains a crucial challenge in Geosciences, Chemistry and Materials Science. In this general picture, I am convinced that the nucleation, growth and dissolution of minerals are among the most fundamental global Earth processes in the first deep-kms, including the so-called critical zone, several natural and/or engineered fluid storage systems and formation of mineral deposits in hydrothermal fields (4-12). In these aqueous media, numerous experimental and theoretical studies in the past two decades have provided evidence for the existence of multi-step nucleation pathways and aggregation processes that take place during the early stages of mineral formation. These multi-step pathways contrast with the over-simplified traditional models described by the classical mineral nucleation theory, i.e. nucleation of seed entities (clusters or nuclei of nanometric size) followed by crystal growth via ion-by-ion or molecule-by-molecule (unit formula) attachment to existing nuclei (formalism exclusively based on the size notion). Herein, *real-time monitoring* of nucleation, growth and transformation of amorphous and/or crystalline particles with microscopic, spectroscopic, chromatographic, thermogravimetric or diffractometric analytic methods, and complementary, or independent, numerical simulations are being developed and are crucial to improve our understanding on these interfacial processes (13-20). In this way, **NAISSANCE** goals and strategy were focused on the formation of minerals under mild and hydrothermal conditions. All envisaged scenarios with relevance on the functioning of current hydrothermal settings as well as on the mineral processing for societal applications, including carbonates, phosphates, sulphates, clay minerals, iron oxides and mineral-mineral composites. In general, the nucleation of minerals in aqueous media is affected by a large set of parameters that are often difficult to study under *in situ* conditions such as i) the multi- and inter-dependency of nucleation-growth processes, ii) the nanometer size of interacting and nucleating particles, iii) the elevated particle-number concentration, iv) the high pressure-temperature-concentration conditions, v) the overlap between nucleation and growth processes, and vi) the role of precursors. The **NAISSANCE** program overcame several of these difficulties by using Raman spectroscopy in real-time and also complemented with other real-time monitoring sophisticated techniques. For example, the so-called non-classical crystallization pathway has been proposed to explain the nucleation of precursors (e.g. formation of stable clusters < 3nm) and the role of transient phases that can reduce significantly the interfacial energy during nucleation and growth processes of several minerals in aqueous solutions. The understanding of the kinetics and thermodynamic parameters governing non-classical crystallization pathway is still in its infancy and many reaction mechanisms and particle-aggregation-transformation processes remain enigmatic (21-26).

## II. Relevant results in the last 10 years

The **NAISSANCE** program was mainly focused on the time-resolved *in situ* measurements of the nucleation, growth and transformation of mineral phases under mild and hydrothermal conditions. Here, the monitoring in real-time by using adapted techniques was a crucial tool to obtain realistic and reproductible insights on the nucleation of minerals and crystals. In the las decade various experimental analytical techniques have been developed as summarized in Figure 1, but, other continue to be developed.

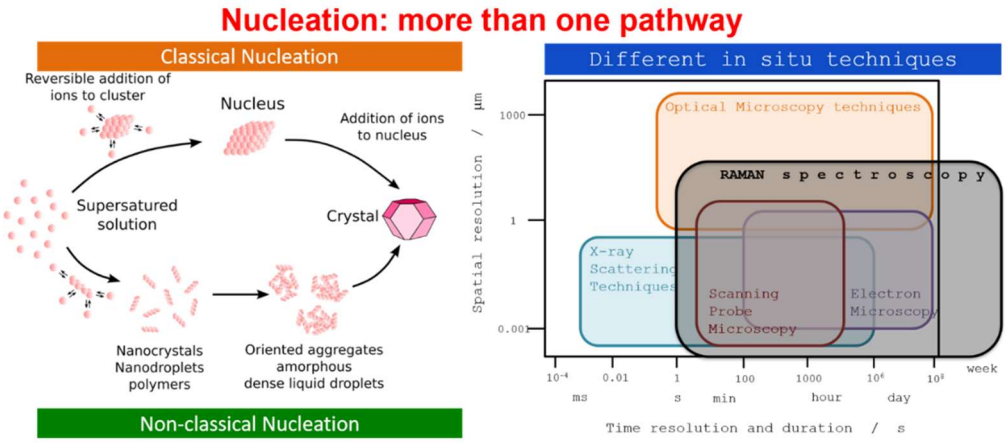



*Figure 1. Schematic description of classical an.d non-classical nucleation of a given crystal from a supersaturated solution (left part). Summary of more relevant analytical techniques used to monitor in real-time the nucleation of crystals (right part).*

In **NAISSANCE**, I designed and developed independent but complementary experimental setups, linked to thermodynamic and kinetics modelling, to simulate a diversified panel of mineral-fluid reactions as a function of temperature (up to 400°C) and pressure (up to 300bar). Herein, the most important experimental development (experimental setup) concerns the monitoring in real-time of nucleation and transformation of mineral phases by Raman spectroscopy.

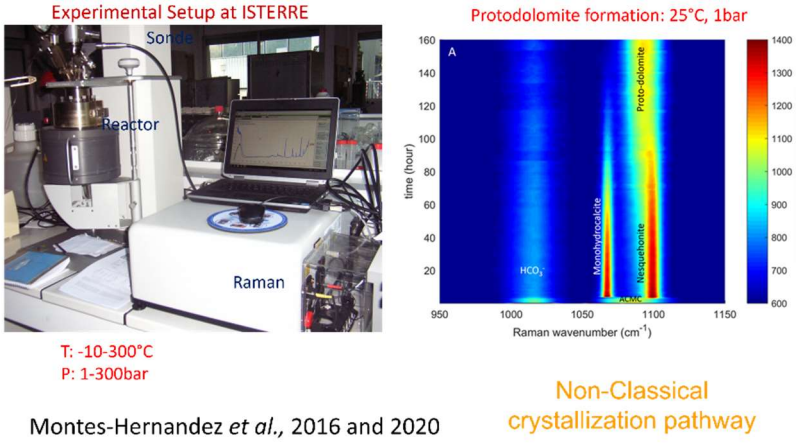



*Figure 2. Experimental setup at ISTerre for real-time monitoring of nucleation and transformation of mineral phases. Parts: Reactor with stirring, temperature and pressure controllers; Raman spectrometer with infrared laser (785nm) and probe (fiber optic); Computer with software for controlling and spectra acquisition and treatment.*

From 2016 to 2025, several international publications have been published including nucleation of carbonates, phosphates, sulphates and iron oxides (1-3, 7, 19, 26-32). See also the Synthetic Mineral Catalog in my web page: Synthetic Minerals Catalog | ISTerre

In the present report-document only, some relevant examples are briefly described:

With my collaborators, we have demonstrated that a triphasic reactor with Raman spectroscopy *in-situ* probe offers unique possibilities for investigating mineral precipitation from aqueous solutions and slurries, and in general for investigating the chemical reactions at solid-fluid interfaces with direct relevance for Earth systems and industrial applications. Data have produced high impact research results (1-3, 7, 19, 26-32). For example, since the 1970s, it has been shown that calcium phosphate crystals nucleate from one or several early amorphous calcium phosphate phases into several *in vivo* and *in vitro* systems. However, the precise chemical composition, structure, and transformation mechanism of these amorphous phases remain controversial. In a recent study, we characterize the reaction mechanism and kinetics of formation of two phosphate crystals, brushite and hydroxyapatite, by using *in situ* Raman spectroscopy in batch reactors from room T (≈25°C) to 90°C. Here, we identified new reactive pathways that characterize the formation of amorphous calcium phosphate phases and their transformation into brushite microcrystals or hydroxyapatite nanocrystals under abiotic conditions as illustrated in the Figure 3 (see also ref. (1)), relevant to a wide range of technological applications and natural environments.

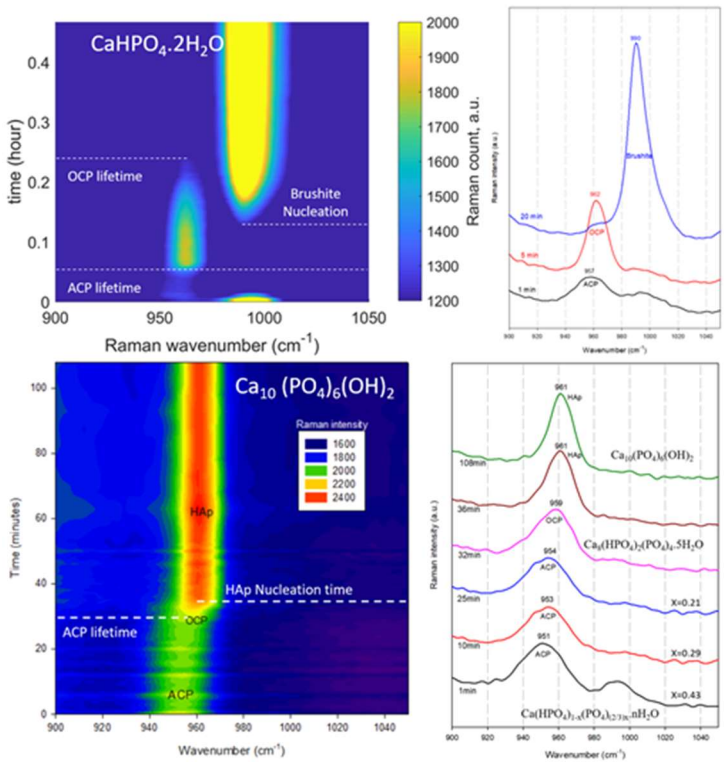



*Figure 3. The nucleation of brushite and hydroxyapatite was monitored in real-time by Raman spectroscopy at room T to 90°C. For both minerals, amorphous calcium phosphate ($Ca(HPO_4)_{1-x}(PO_4)_{(2/3)x}.nH_2O$) with different Raman signature was instantaneously formed in the initial solution. Then, the amorphous phases transform into OCP, a crystalline transient phase. Finally, OCP transforms into brushite micro-crystals or into hydroxyapatite nanocrystals.*

In another fundamental study, real-time Raman measurements have recently suggested that that primary dolomite, as initially defined ($Mg^{2+} + Ca^{2+} + 2CO_3^{2-} \rightarrow CaMg(CO_3)_2$), has a very low probability to form at room temperature in abiotic or biotic systems, i.e. through heterogeneous nucleation (pre-existence of reactive surfaces) and a non-classical nucleation mechanism is therefore expected (27). This study has demonstrated that transient amorphous or crystalline phases play a significant role in the formation of disordered dolomite. Such transient phases can decrease the energy barrier under ideal fluid chemistry conditions (e.g. direct transformation of ACMC into disordered dolomite), but they can also retard dolomite precipitation (e.g. via transient formation and concurrent dissolution of nesquehonite and monohydrocalcite) as demonstrated in Figure 4. The important consequence is that my experimental data have shown that proto-dolomite may form, but the mechanism is not direct and involves precursory minerals that nucleate, grow and then dissolve before proto-dolomite can form.

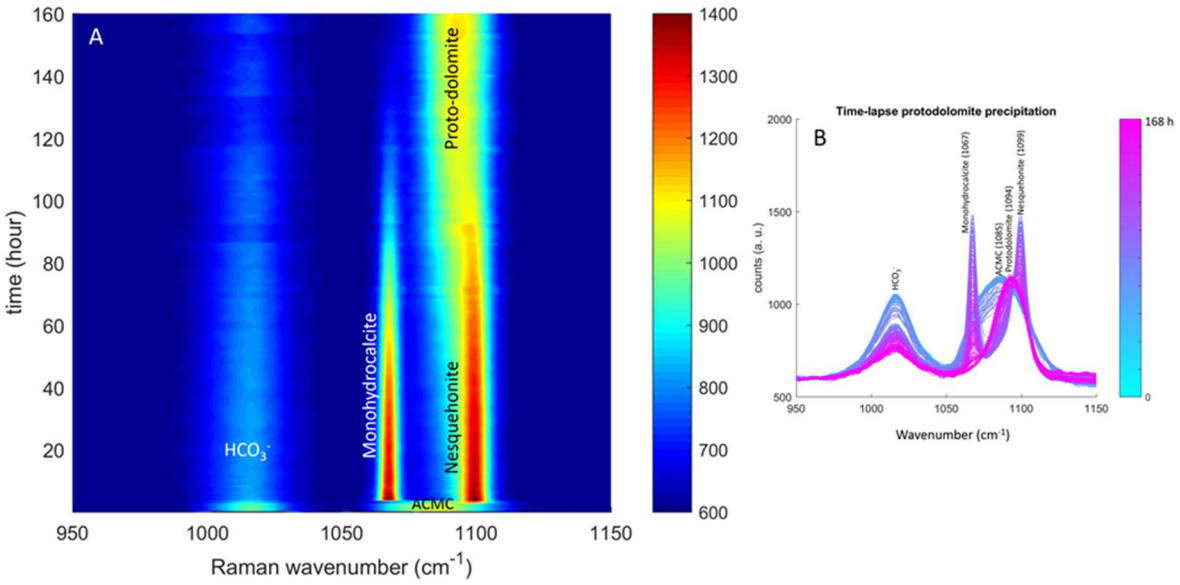



*Figure 4. Time-lapse Raman spectroscopy monitoring of the formation of disordered dolomite from ACMC. Left: evolution of the two transient phases, nesquehonite and monohydrocalcite. Right: time-lapse evolution of the main Raman peaks. Details and spectral movie in Montes-Hernandez et al. 2020 (27).*

The early formation of amorphous phases and their transformation into transient or stables crystalline phases is now considered to be a generalized reaction pathway during the formation of inorganic and biogenic carbonates and phosphates minerals in aqueous systems. This crystallization pathway agrees with Ostwald's rule, but transient phases are not necessary polymorphs; in fact, they can have different chemical compositions and different particle sizes. Moreover, thermodynamic and kinetic arguments should be considered in the crystallization tendency (1). In this way, multi-steps nucleation events may exist and combine various reaction mechanisms at the solid-fluid interfaces. Thus, solid-state transition, dissolution-recrystallization, self-assembly aggregation and partial dissolution-aggregation may operate together during nucleation and growth processes (e.g., 1-3, 7, 19, 26-32).

In this case, the classical nucleation theory (CNT), the most common theoretical formalism to quantify nucleation and growth rates (including variation of particles size distribution) cannot be applied successfully in its original formalism as proposed in the last three decades by various

research groups (e.g. 21-24). These groups have proposed an alternative conceptual approach called non-classical nucleation and crystallization pathways (including pre-nucleation clusters). However, in the absence of a theoretical quantitative formalism to describe the so-called non-classical nucleation, the improved and/or adapted classical nucleation theory is still considered the best engineering tool to simulate the non-classical nucleation of crystals and amorphous particles (e.g., 25, 33).

In this way, I will associate numerical simulations to the experiments on the formation of diversified minerals. The numerical code Nanokin (34) can be regarded as an efficient option, as recently reported in two studies (33, 35). For example, this approach of mineral precipitation has successfully been used in a combined theoretical/experimental study of calcite formation by portlandite carbonation (33). It allowed us to decipher the various steps in the mineral transformation, behavior of crystal size distribution (Figure 5) and the evolution of the composition of the aqueous solution. The comparison between experiment and simulation was shown to put strong constraints on simulation parameters (surface energies, nucleation rate, growth law), and also provided information on in situ conditions (e.g. pH and element concentration and speciation).

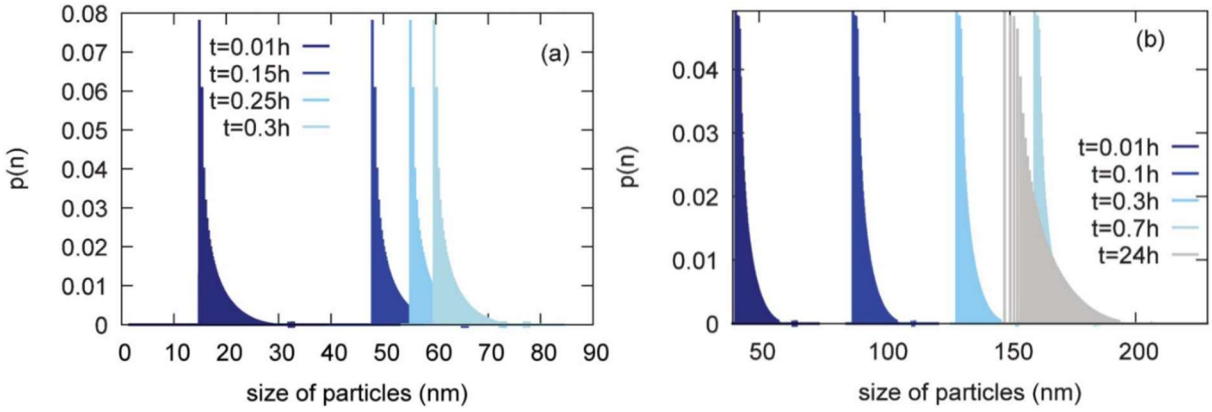



*Figure 5. a) Calcite crystal size distribution functions during precipitation of this mineral at T=30 °C, taken at four different times: t = 0.01, 0.1, 0.2 and 0.3 h. b) Calcite crystal size distribution functions during precipitation at T=90 °C taken at four different times: t = 0.01, 0.1, 0.3, 0.7 and 24 h (Fritz et al., 2013, ref. (33)).*

This approach has a strong interest for both geochemical and chemical engineering communities. It was also helped to understand the competition between different mineral phases that may form simultaneously in the solution. The thermodynamic and kinetics simulations can then predict the time evolution of the particle sizes and their composition profiles, with continuous feedback effects between the predicted evolutions of the aqueous solution and that of the population of forming particles.

In summary, time-resolved Raman spectroscopy measurements combined with X-ray scattering/diffraction and numerical simulations have provided basic knowledge on the classical and non-classical crystallization pathways in homogeneous or heterogeneous systems. In the continuity of these above results, the existence and persistence of precursory transient phases during nucleation processes and the role of organic matter in bio-mimetic systems will be investigated for several strategic minerals such as carbonates, phosphates, iron oxides and clay minerals in the coming years. The main outcomes of the continuity of **NAISSANCE** program and strategy for the next years will be i) the production of unique laboratory

experimental data, ii) the development of new kinetics and reaction mechanisms of mineral precipitation that accounts for the existence of transient phases, iii) the optimization of chemical reactions that can be used to reuse and store $CO_2$ permanently in minerals (**CLEANER** CNRS project). The implications of this collaborative project comply with fundamental aspects in Materials science, Chemical Engineering and geochemistry topics, actively investigated in France, Europe and through the world.

## III. Summary of activities related to research work

Since my arrival as postdoc in 2006 and my recruitment in CNRS in 2009, I have built a cross-disciplinary research group in Grenoble, with national and international collaborations, on topics related to nucleation, growth and transformation processes of minerals and I have also pioneered several studies on the $CO_2$ capture, utilisation and storage (CCUS). My experimental research is then relevant to fundamental processes in the Earth systems such as mineral formation in hydrothermal systems, $CO_2$ impact in drink water aquifers, gas-solid carbonation in soils and aerosols widespread in cold natural environments; and societal applications such as Ca looping process, synthesis of nanostructured minerals and composites, $CO_2$ capture by alkaline sorbents and particularly direct or indirect transformation of $CO_2$ gas to solidified added-value carbonates such as magnesite and dolomite (permanent $CO_2$ storage) and/or production of very-high used carbonates such as nano-calcite, siderite, eitelite, hydromagnesite, etc.

### (a) Supervision/co-supervision of students

***PhD. Students***: I have supervised or co-supervised the PhD thesis of eight students. **Romain Lafay** (doctoral fellowship from the University Grenoble Alpes, 8 publications, thesis defended October, 3rd, 2013) worked on the partition of trace elements during nucleation and growth processes of serpentines and competitive serpentinization and carbonation of ultrabasic rocks (San Carlos olivine model). **Alexandre Garenne** (doctoral fellowship from ANR Spring, thesis defended December 10th, 2014, 4 publications) conducted research on the hydration and carbonation processes at the surface of Mars and asteroids. I have supervised this work in collaboration with Pierre Beck. **Ayumi Koishi (**doctoral fellowship from the University Grenoble Alpes, thesis defended October, 30th 2017, 2 publications) worked on the amorphous calcium carbonate (ACC) precursors to crystalline calcium carbonate phases in a context of biomineralization. She has obtained relevant insights on the water dynamic in partially stabilized amorphous phases by using synchrotron and conventional tools. I have co-supervised this work with A. Fernandez-Martinez.

In collaboration with the Natural Water Treatment Laboratory, Water Research and Technologies Center (CERTE) in Tunisia, **Afef Hamdouni** (PhD student in Tunisia, thesis defended in April 2017) has carried out a 6-months research internship in Grenoble under my supervision and published one article on the trapping of Fe(II) as oxyhydroxide during carbonation process. In the same way, **Sabah Hajji** (PhD student in Tunisia, thesis defended in July 2018) has finished a 6 months research internship on the sequestration of toxic ions in/on nanostructured iron minerals, published in one high-level international publication.

Before covid19 pandemic situation. I have hosted two other foreign PhD students at ISTerre (geochemistry group) for 6 months to deepen their research work on the calcium-silicates

nucleation under hydrothermal conditions (Xonotlite model) (**Felipe Marti Montava**, grant from Spain) and on the removal of ferrous iron contained in groundwater from Niger: Experimental approach (**Mamane Imrana Chaibou Ousmane**, grant from "Campus France" program). I have also hosted **Marthe Guren** in 2019 (PhD student, University of Oslo) for a short research stay that led to an article published in Chemical Geology on the sequestration of chromium on calcite.

More recently, in 2024, **Recardo Khumalo** (PhD student from South Africa) was hosted at ISTerre for two months in order to perform nucleation experiments on the aragonite (calcium carbonate polymorph). This with direct implications on the treatment of acid mine drainage (AMD) by urea hydrolysis (Carbonates recovery and Irrigation water use). Here, one publication was redacted by Recardo and final revision is being performed by co-authors.

***Master, Bachelor and ungraduated students***: I have supervised 23 B.S, M.S and ungraduated students and various of them are co-authors of articles written using the main results of their research.

1. **Nicolas Concha-Lozano:** Incorporation/sequestration of oxyanions during nucleation and growth of calcite crystals (G. Montes-Hernandez, N. Concha-Lozano, F. Renard, E. Quirico, Removal of oxyanions from synthetic wastewater via carbonation process of calcium hydroxide: Applied and fundamental aspects. *Journal of Hazardous Materials 166* (2009) 788-795).

2. **Alexandre Garenne:** Gas-solid carbonation of alkaline minerals under cold planetary conditions (A. Garenne, G. Montes-Hernandez, P. Beck, B. Schmitt, O. Brissaud, A. Pommerol. Gas-solid carbonation as a possible source of carbonates in cold planetary environments. *Planetary and Space Science 76* (2013) 28-41).

3. **Mahaut Di Girolamo**: AgNPs precipitation on textile fibers and their oxidative dissolution using flow-through reactors (G. Montes-Hernandez, M. Di Girolamo, G. Sarret, S. Bureau, A. Fernandez-Martinez, C. Lelong, E. Eymard Vernain. In situ Formation of Silver Nanoparticles (Ag-NPs) onto Textile Fibers. *ACS Omega* 6 (2021) 1316-1327.

4. **Mamadou Bah**: Séquestration minérale directe et indirecte du $CO_2$ : Approches expérimentales et apport des ligands organiques. (Stay in 2018). This experimental study has demonstrated how magnesite (strategic mineral to storage permanently $CO_2$) can be precipitated at moderate temperature (G. Montes-Hernandez, M. Bah, F. Renard. Mechanism of the formation of engineered magnesite: A useful mineral to mitigate $CO_2$ industrial emissions. *Journal of $CO_2$ Utilization 35* (2020) 272-276).

5. **Alix Tordo**: Removal of As from water using nanostructured minerals, some results were incorporated in an important recent study on the removal of As(III) and Cr(VI) from water (S. Hajji, G. Montes-Hernandez, G. Sarret, A. Tordo, G. Morin, G. Ona-Nguema, S. Bureau, T. Turki, M. Nzoughi. Arsenite and chromate sequestration onto ferrihydrite, siderite and goethite nanostructured minerals: Isotherms from flow-through reactor experiments and XAS measurements. *Journal of Hazardous Materials* 362 (2019) 358-367).

6. **Manon Revol**: Removal of Ag, Cr and As from water using nano-magnetite, hematite, pyrrhotite and hydroxyapatite using flow-through experiments.

7. **Clara Vernier**: Removal of amoxicillin from water using nano-magnetite, adsorption and Fenton reaction experiments using flow-through reactors. Some of obtained results were integrated in a recent paper on the removal of antibiotics from water (G. Montes-Hernandez, L. Feugueur, C. Vernier, A. Van Driessche, F. Renard. Efficient Removal of Antibiotics from Water via Aqueous Portlandite Carbonation. *Journal of Water Process Engineering* 51 (2023) 103466.)

8. **Michelle Garcia Salinas**: Precipitation of Mg-Fe-Ca Carbonates from indirect carbonation of peridotite and steel slag waste (confidential report).

9. **Elodie David**: Optical real-time monitoring of salts and calcite crystallization from drying drops (topic in developing)

10. **Noémie Bouchard**: Capture of carbon dioxide using NaOH and lysine compounds (confidential report).

11. **Lola Feugueur**: Removal of As(III) and antibiotics from groundwater using nanominerals and mineral nanoparticles. These results were shared in important publication on the antibiotic removal from water mainly concerning the work of two bachelor students (G. Montes-Hernandez, L. Feugueur, C. Vernier, A. Van Driessche, F. Renard. Efficient Removal of Antibiotics from Water via Aqueous Portlandite Carbonation. *Journal of Water Process Engineering* 51 (2023) 103466.)

12. **Sarah Clavier (M2R, Lille)** Indirect carbonation of peridotite and use of synthesized Fe-magnesite as partial substitute of cement in concrete fabrication. The obtained data have completed the results obtained in the las five years and achieved in an international publication (Montes-Hernandez, German, Weiss, Jérôme, Clavier, Sarah, Cordonnier, Benoit, Tafforeau, Paul, Plé, Olivier, Renard, Francois. Fabrication of iron-magnesite under mild conditions through indirect carbonation of peridotite and its utilization as partial substitute for cement in concrete. *Industrial & Engineering Chemistry Research* (2025) 64, 13770-13783.)

13. **Amethys Brioudes (BUT, Grenoble)** Optical real-time monitoring of calcite crystallization on hydrophilic and hydrophobic surfaces (topic in developing)

14. **Jules Collion (BUT, Grenoble)** Removal of ammonium and nitrate ions from water: real-time monitoring by using an multi-ionic probe (topic in developing)

15. **Suzanne Marchaland Le Bihan (L2, Paris)** Optical real-time monitoring of CO2 capture from air using µ-drops of NaOH and $Ca(OH)_2$ solutions (topic in developing).

16. **Sihem Rmili (invited PhD student from Tunisia)** Mineral and 2D elemental caracterization of natural samples from Bahloul and Fahdène formations in Tunisia and experimental Pb-Zn mineralization (PbS and ZnS formation) under hydrothermal conditions using batch mini-reactors). Several natural samples were characterized by infrared spectroscopy, electron microscopy and electron probe microanalyzer and thermogravimetry analyses.

17. **Lucas Bernard (BUT, Grenoble)** pH probe calibration at high T (90-150°C) and pH monitored in real-time during protodolomite formation at 90 and 150°C.

18. **Laureline Ronzière (BUT, Grenoble)** Fe-magnesite fabrication from indirecte carbonation of ultrabasic rocks. Support to SOLUTION project (MITI-CNRS 2024-2025)

19. **Mia Joubaud (BUT, Grenoble)** Cr(VI) removal from water by using synthetic mineral-mineral composites.

20. **Gabriel Montes (BUT, Lyon)** Monohydrocalcite (CaCO3.H2O) to aragonite (CaCO3) polymorph transformation: Kinetics and activation energy from Raman spectroscopy. The obtained results were shared in the Goldschmidt geochemistry congress in Prague and one international publication is in redaction.

21. **Clement Imbert (BUT, Grenoble)** Influence of Mg-Ca-Fe carbonates on the cement hydration: $CO_2$ storage as carbonate minerals in concrete. Very interesting preliminary results were obtained.

22. **Maëlle Bredeka (BUT, Grenoble)** Silver nanoparticles (AgNPs) nucleation/insertion in/on textile fibers and mineral surfaces: Reactivity tests using flowthrough reactors.

23. **Shaza Islim (M2R, Lille)** Portlandite synthesis and its utilization as precipitating agent for toxic oxyanions (As, Se, Cr) in polluted water. Very interesting results than allows the redaction of one international publication.

**(b) Projects management and lab infrastructure**

Since 2009, I have participated as principal investigator (PI) or co-PI to national and international projects. In chronological order: SOLUTION MITI-CNRS (2024-2025), CEMENTO PEPS-INSIS CNRS (2023), European ACT program FUNMIN (2019-2023), Labex SERENADE (2016-2017), ANR Coro (2012-2015), ANR Spring (2011-2014), Labex OSUG@2020 (2012), and other local or ISTerre internal (BQR) projects), I have built a state-of-the-art moderate-temperature (up to 400°C) and moderate-pressure (up to 400bar) geochemistry experimental room (about 40$m^2$) in Grenoble through the acquisition of various experimental setups that include:

1. A cryogenic reaction cell for *in situ* infrared measurement (investment: **10 kEuros**): Project in collaboration with Pierre Beck, Olivier Brissaud et Bernard Schmitt at Institut d'Astrophysique et Planétologie de Grenoble (IPAG).

2. A hydrothermal autoclave made of titanium with pH sensor (investment: **35 kEuros**): Collaboration with CiNAM center. Coro ANR project in collaboration with Daniel Vielzeuf Group.

3. A hydrothermal autoclave made of Hastelloy C22 for *in situ* Raman measurements (investment: **40 kEuros**): Collaboration between the Fault Mechanics and Mineralogy teams at ISTerre, Labex OSUG project: PIs: German Montes-Hernandez and François Renard.

4. A hydrothermal autoclave made of Hastelloy for simple mineral alteration and mineral hydrothermal syntheses (investment: **11 kEuros**). Project in collaboration with Alexander Van Driesche, ISTerre.

5. A multi-oven for hydrothermal experiments (investment: **10 kEuros**). Project in collaboration with Emilie Janots, ISTerre.

6. Several percolation reactors made of plastic for breakthrough curves and isotherms of pollutants, including pump and connectors (investment: **20 kEuros**). EU FUNMIN and SOLUTION MITI-CNRS projects.

7. Small equipment's (peristaltic pumps, flow-through reactors, analytical balance, Imacimus pH and multi-ions probes, etc.) (investment: **30 kEuros**). EU FUNMIN and CEMENTO PEPS-CNRS projects.

8. A Raman spectrometer (RXN Kaiser Optical Systems) with optical fiber probe (investment: **90 kEuros + 30 kEuros (**replacement of laser and maintenance): Collaboration between Faults Mechanics, Geochemistry and Mineralogy groups at ISTerre (GDF-SUEZ industry grant and Labex Serenade). PIs: German Montes-Hernandez and François Renard.

9. A Raman fiber optic probe and optical dispositive for Raman measurements onto powdered samples (investment: **20 kEuros**). FUNMIN project.

My experimental room is also equipped of 4 tubular hydrothermal reactors with three independent zones of heating for hydrothermal experiments up to 400°C and 400bar inherited from (J. P. Gratier) and reaction cells connected to one inverted optical microscope for nucleation experiments at room T inherited from (F. Renard) as illustrated in some compilated photos below.

*Figure 6. Experimental room for mineral-fluid interactions under mild and hydrothermal condition (up to 400°C and 500bar). All these experimental setups are also summarized in some slides available in my page web (https://www.isterre.fr/IMG/pdf/experimental-setups-in-my-lab.pdf )*

In the past years, using this equipment, I have pioneered a new experimental technique: the time-lapse measurements of the nucleation and growth of crystals and non-crystalline mineral particles (e.g., carbonates, phosphates, sulfates, iron oxyhydroxides and oxides, etc.) under hydrothermal conditions (See Figure 2). This laboratory technique is the only one so far with enough time resolution and precision to identify the formation and dissolution of chemical species and solid phases during a series of interacting hydrothermal reactions. These results have been disseminated in various international publications (1-3, 7, 19, 26-32 in this document).

**(c) Synergetic activities**

- Elected member of lab board at ISTerre (from 2025 to 2029)

**1.** From January **2017** to November **2019 (**3 years in total**)**, I was the leader of the Geochemistry team at ISTerre. This multi-disciplinary group (from mantle rocks to biosphere) federated 10 researchers, 4 engineers, 3 invited researchers and 10-15 students (B. Sc., M. Sc. and PhD). This is a very important management task in France because the team leader prepares decisions and proposes scientific directions concerning the allocation of PhD thesis grants, the definition of scientific priorities, and the allocations of local grant projects. As team leader, I was responsible of the technical staff (responsible of annual interview imposed by professional organization) and I coordinated the preparation of the national assessment of the group (HCERES). In this way, my most important task was the preparation and supervision of summary (five past years) and prospective document for the next five years program for HCERES assessment. I coordinated and wrote the general items of five-year summary (2015-2019) and project and strategy for the next five years (2020-2024). See: https://www.isterre.fr/IMG/pdf/e2_geochimie_self_assessment_document_vague_a_rech_ur_.pdf

2. From January 2016 to December 2016, I was elected as active member of the department board at ISTerre.

3. With Mexican colleagues, I participated to the creation to an informative French network "Red de Talentos, Capitulo Francia" for Mexicans working or studying in France. This network, supported by the Mexican embassy in France, facilitates the scientific and business exchange between France and Mexico.

4. From January 2015 to December 2018, I co-supervised the "axe transverse" on the geo-resources at ISTerre, a cross-disciplinary action at the department scale. The goal was firstly to identify potential cross-disciplinary local activities concerning geo-resources in order to create new collaborations and develop new research topics relevant for both basic research and societal needs. I coordinated various activities such as seminars and workshops during these three years.

5. In 2014, I organized one scientific session on the reactivity of $CO_2$ in aquifers during RST French meeting at Pau.

**(d) Teaching activities**

As Mexican citizen and ex-fellow of CONACYT in Mexico, I contribute annually with 8-16h teaching in the Master of Chemical Engineering in the University in Tlaxcala (UAT), Mexico. I am teaching:

- Synthesis and applications of nanostructured minerals
- Wastewater and solid waste treatment and management
- Removal of toxic ions and organic molecules from water
- Storage, mineralization, capture and reactivity of $CO_2$

**(e) International and national collaborations**

I participated to the European consortium on $CO_2$ mineralization via the project FUNMIN (2019-2023), as PI in France various actions (e.g., student formation, annual report for ADEM French agency, seminars, etc.) and preparation of one international publication. I have also developed several collaborations on the processes of crystal growth, that have allowed me to publish in the best journals of Earth Science and Chemical Engineering such as Chemical Geology, Geochimica et Cosmochimica Acta, Environmental Science & Technology, Chemical Engineering Journal, Journal of Hazardous Materials, etc. These publications involve international collaborations with co-authors in, e.g., Germany, Spain, Switzerland, The Netherlands, Norway, England, USA and Tunisia.

**(f) Peer-review of publications, projects and thesis**

1. I am reviewing around 15 publications every year in the domains of Earth Sciences and Inorganic Chemistry, including articles in the best journals in these domains such as Angewandte Chemie, Advanced Materials, Small, Environmental Science & Technology, Geochimica et Cosmochimica Acta, Journal of Hazardous Materials, Water Research, Journal of Colloid & Interface Sciences, Chemical Geology, Langmuir, Applied Clay Science, Journal of Crystal Growth, Crystal Growth & Design, Chemical Engineering Journal, Energy & Fuels, American Mineralogist, others.

2. I have reviewed four research projects (ANR and European)

3. I was invited to participate to evaluation committees of two Ph. D. thesis (Spain and France), one committee Ph.D. thesis (France) and four M. Sc. thesis (France).

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