RÉPUBLIQUE FRANÇAISE
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# InnovTech

## TECHNOLOGIES INNOVANTES ET DURABLES

FRANCE 2030

PROGRAMME DE RECHERCHE

*SOUS-SOL*

## *Résumé du projet en français (Non Confidentiel – 4000 caractères maximum, espaces inclus)*

Le sous-sol, défini comme l'enveloppe géologique supérieure de la surface terrestre sous le sol, est le siège d'activités qui sont essentielles pour relever les défis mondiaux. L'un des principaux objectifs du PEPR SousSol est de définir les conditions d'un usage durable et optimal du sous-sol. Cela implique non seulement d'améliorer les connaissances géologiques du sous-sol et de travailler sur les enjeux technico-économiques, sociaux et environnementaux de son utilisation, mais aussi de développer des approches scientifiques interdisciplinaires innovantes pour favoriser son exploitation. InnovTech contribuera à cet objectif en relevant les principaux défis scientifiques liés au traitement durable des ressources minérales, de la caractérisation à la métallurgie extractive en passant par la minéralurgie.

Ces grands défis scientifiques seront relevés dans InnovTech en améliorant les méthodes existantes et en développant des technologies innovantes. Pour atteindre cet objectif, les travaux qui seront réalisés dans InnovTech incluront le développement de méthodes automatisées, générique, *in situ* et en ligne de caractérisation d'un minerai et le développement de solutions intégrées pour traiter des ressources plus complexes, variables, polymétalliques et non conventionnelles, tout en réduisant la consommation d'énergie, de produits chimiques et d'eau, ainsi que la production de déchets. Pour ce faire, des approches innovantes seront mises en œuvre, notamment l'utilisation de l'intelligence artificielle et du *machine learning*, ce qui a rarement été fait auparavant. Ces approches incluront également la modélisation DEM et CFD, les simulations atomistiques fondées sur la chimie quantique et la modélisation thermo-cinétique. Elles seront combinées à des travaux expérimentaux originaux et de haut niveau afin de mieux comprendre les phénomènes qui interviennent dans les processus, ce qui permettra de concevoir des modèles pertinents et précis. Ces approches permettront d'améliorer la compréhension des mécanismes impliqués dans les processus de traitement, ce qui est essentiel pour le développement de méthodes innovantes. Ainsi, InnovTech sera pionnier dans le domaine de la caractérisation des minerais, du traitement des minéraux et de la métallurgie extractive.

Au-delà des objectifs scientifiques, InnovTech vise également à fédérer la communauté scientifique travaillant sur le traitement des minerais. Il rassemblera des communautés scientifiques travaillant dans divers domaines des géosciences, de la chimie, du génie des procédés et de l'informatique avec un lien fort avec le PC6 et donc avec la communauté scientifique travaillant dans l'évaluation environnementale des procédés.

Globalement, InnovTech soutient des projets scientifiques à faible TRL (1-4), permettant (1) d'augmenter le niveau de compétences de la communauté scientifique française dans le domaine de l'extraction, (2) de fédérer les différents groupes scientifiques sur des projets communs axés sur la problématique minière, et (3) de les placer au premier plan au niveau européen et international en ce qui concerne l'exploitation durable des matières premières primaires.

## *Résumé du projet en anglais (Non Confidentiel – 4000 caractères maximum, espaces inclus)*

The subsurface, defined as the upper geological envelope of the Earth's surface below the ground, is a physical environment for activities and resources extraction that are essential to meet global challenges. One of the main objectives of the PEPR SousSol is to define the conditions for sustainable and optimal use of the subsurface. This involves not only improving the geological knowledge of the subsurface and the technical-economic, social, and environmental aspects of its use but also developing innovative interdisciplinary scientific approaches for favouring its exploitation. InnovTech will contribute to this objective by addressing the main scientific challenges related to the sustainable processing of mineral resources, from the characterization stage to the extractive metallurgy phase and including the mineral processing steps.

These major scientific challenges will be addressed in InnovTech by improving state-of-the-art methods and by developing innovative technologies. To reach this objective, works in InnovTech will include the development of automated, *in situ*, and on-line ore characterization methods that are not ore-specific and the development of integrated solutions to process more complex, variable, polymetallic, and unconventional resources, while reducing the energy, chemicals, water consumption and waste production. For that, disruptive approaches will be implemented, including in particular the use of artificial intelligence/machine learning, which has rarely been performed before. These approaches will also include DEM and CFD modeling, quantum-chemistry-based atomistic simulations, and thermo-kinetic modelling. They will be combined with original and high-level experimental works in order to get more insights into the phenomena acting in the processes, allowing then to design the most relevant and accurate models. These approaches will allow improving the understanding of the mechanisms involved in the treatment processes, which is essential for the development of innovative methods. Hence, InnovTech will be pioneer in the field of ore characterization, mineral processing, and extractive metallurgy.

Beyond the scientific objectives, InnovTech also aims at federating the scientific community working on ore processing. It will put together scientific communities working in various fields of geosciences, of process engineering and of computer sciences with a strong link with PC6 and then with the scientific community working in the environmental assessment of processes.

On overall, InnovTech supports scientific projects at low TRL (1-4), allowing (1) to increase the level of skills of the French scientific community in the extraction domain, (2) to federate the various scientific groups on common projects focused on mining problematics, and (3) to put them at the forefront at the European and international level regarding the sustainable exploitation of primary raw materials.

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# 1. Context, objectives, and previous achievements

## 1.1. Context, objectives, and innovative features of the project

The subsurface, defined as the upper geological envelope of the Earth's surface below the ground, is a physical environment for activities and resources extraction that are essential to meet global challenges. In particular, it is a reservoir of raw materials which potential for energy and environmental transitions is not fully characterized and recognized. One of the main objectives of the PEPR SousSol is to define the conditions for sustainable and optimal use of the subsurface. This involves not only improving the geological knowledge of the subsurface and the technical-economic, social, environmental, and regulatory aspects of its use but also developing innovative interdisciplinary scientific approaches for favouring its exploitation. InnovTech contributes to this objective by addressing the main scientific challenges related to the sustainable processing of mineral resources, which are listed below:

- **Development of automated, *in situ*, and on-line ore characterization methods that are not ore-specific**. Characterisation is the starting point of any extraction study since the development of processing options is based on a thorough characterization of the considered ore. This stage should be conducted as much as possible close to the processing stages for allowing to better adjust the operating conditions of the processing units and then for improving the performances of the processing plants.
- **Development of integrated solutions to process more complex, variable, polymetallic, and unconventional resources, including low grade ores, while reducing the energy, chemicals, water consumption and waste production:**
  - For mineral processing, development of innovative and breakthrough approaches and technologies to reduce energy consumption in comminution steps, to improve the selectivity of the separation and to reduce the amount of waste and the use of water in the downstream concentration processes.
  - For extractive metallurgy, development of sustainable and selective separation processes in extractive metallurgy (hydro-metallurgical (bio) – electro – processes or pyro-metallurgical processes, solvent extraction, and ionic flotation).
  - Development of an eco-design approach coupling simulation softwares used in process development and in Life Cycle Assessment (LCA) studies, allowing to improve the technical, economic, and environmental performances at the early stages of the studies.
- **Digital transformation of the extractive sector with an increased use of Artificial Intelligence (AI)**, particularly machine learning, as a tool to increase the understanding of the fundamental phenomena that are involved in each processing step and, therefore, to improve the process development and the scale transfer.
- **Mathematical modelling and numerical simulations of mineral processing and extractive metallurgy processes:** thermodynamic modelling/thermodynamic databases, DFT and *ab initio* molecular dynamic modelling for adsorption and transport phenomena at interfaces, 'true kinetics' models for the simulation of chemical systems out of equilibrium, CFD modelling for multiphase particulate and catalysed triphasic reactive systems.

Even though there are currently several action plans at European level to address these challenges, the last one being the "Critical Raw Material Act", these plans are mostly dedicated to projects with high TRL to accelerate the relaunch of the mining industry in the European Union. Hence, a gap still exists between the development of breakthrough and disruptive technologies and the available fundings for understanding fundamental phenomena at low TRL. InnovTech bridges this gap at the French level by supporting scientific projects at low TRL (1-4), allowing (1) to increase the level of skills of the French scientific community in the extraction domain, (2) to federate the various scientific groups on common projects focused on mining problematics, and (3) to put them at the forefront at the European and international level regarding the sustainable exploitation of primary raw materials.

France is one of the top 8 publishing countries in geological sciences related to subsurface resources and uses. Regarding process development for exploitation of raw materials, most of the research works are related to extractive metallurgy and there is then a need to support actions in mineral processing. InnovTech aims to fill this need and to go beyond through a synergetic work of all high-level French research groups in order to develop innovative approaches and technologies for addressing the aforementioned scientific challenges.

## 1.2. Main previous achievements

**InnovTech gathers French research groups having high-level expertise in the considered scientific areas, all recognized at an international level**.

Regarding characterization methods and sampling, there are only few applications of the Gy theory of sampling or of the measurement uncertainty to automated mineralogical measurement systems[1]. BRGM has recently developed a methodology allowing to overcome the complexity of objects by combining the Gy theoretical approach to an empirical approach based on repeated sampling plans[2]. This methodology was applied to urban mining and will then be adapted in InnovTech for being relevant to complex ores. Besides, UL has undertaken over the past decade a lot of research works focused on the use of easy-to-handle analytical methods for automated mineralogy and samples characterization[3]. This will pave the way for the development of integrated methods to analyse and characterize samples in terms of chemical and mineralogical compositions.

In mineral processing operations, INP/LGC, BRGM and LGF/MSE have performed a lot of works on the characterization of fragmentation mechanisms and on the development of innovative fragmentation methods. In particular, INP/LGC has showed that even if the historical criteria used to characterize the energy requirements for an ore fragmentation, such as the Bond Index, are effective in the selection and tuning of long-used grinding technologies, these criteria are only indirectly related to the mechanisms that govern ore fragmentation[4]. As a result, they cannot be used to access to the actual grinding efficiency or design new fragmentation technologies that exploit the intrinsic fracture properties of ores. Indeed, several authors have recently shown that the chemical, petrographic and mineralogical properties of an ore have a strong influence on its behaviour during grinding[5]. Theoretical, numerical and experimental approaches will be used here to tackle this bottleneck. They will in particular be based on DEM modelling knowing that the specific approach of physical (mechanistic) and geometric description of the reaction developed by LGF/MSE in heterogeneous kinetics is a national reference for almost 50 years[6]. Regarding the development of innovative fragmentation technologies, BRGM has been working on the electrofragmentation for more than 15 years. This technology aims at generating cracks at the grain boundaries allowing not

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[2] Hubau, A. et al. (2022). Proceedings 10th World Conference on Sampling and Blending.
[3] Fabre, C. et al. (2022). Spectrochimica Acta Part B: Atomic Spectroscopy, 194, 106470.
[4] Bourgeois, F. et al. (2015). International Journal of Mineral Processing, 136, 7-14.
[5] Deniz, V. (2022). Minerals Engineering, 176, 107348.
[6] Soustelle, M. (1970). Journal de Chimie Physique, 67, 240-245.

only to reduce the energy consumption of the fragmentation but also to improve the selective liberation of the minerals and to limit the production of fine particles that hinders the downstream processing steps[7]. This combination of InnovTech partners' expertise will then allow achieving major improvement in ore comminution.

Moreover, INP/LGC, BRGM, and UL have been working intensively on developing models at various scales to understand the fine mechanisms involved during separation processes. In particular, works performed by INP/LGC and BRGM, and more recently by UL with the development of CFD models, allowed gaining understanding in the physical mechanisms that are involved in the Falcon centrifugal separation[8,9]. UL holds also a strong expertise in the field of theoretical chemistry and has been developing, over the past few years, molecular models to understand the physicochemical processes that occurred at the liquid/solid interface[10]. This work will support the experimental approaches that will be developed here about the understanding of the fundamentals of flotation.

Regarding extractive metallurgy processes, an innovative process based on alkaline leaching was developed by BRGM to extract metals (Cr, V, Mo, Mn) from electric arc furnace slags[11]. BRGM has also developed a deep knowledge over the past decades on bioleaching of sulphide minerals, which are most of the time refractory to leaching at atmospheric pressure and moderate temperature. In particular, BRGM demonstrated that the nature of the species and even the physicochemical mechanisms responsible for this passivation remain controversial[12]. BRGM and CNRS/LGC have been working on the development of a leach reactor aiming to perform in situ mechanical abrasion of sulphide minerals' particle passivation layers and this work will be continued in InnovTech. In addition, CEA is widely recognized for the characterization of suspended solids during dissolution, which remains a challenge under real experimental conditions[13]. For such studies, microscopic imaging systems have been developed to observe one or more solid particles during the dissolution phenomenon[14]. These skills will be used in InnovTech for developing innovative experimental equipment that will enable the *in-situ* observation of dissolution reactions.

Furthermore, UL and CEA have been working on the synthesis of new extractants for Li recovery and Mn-Co separation by liquid-liquid extraction (patent in progress) and on the development of physicochemical and engineering models to describe mixers-settlers behavior and to understand leaching and solvent extraction[15]. UL has also a recognized expertise on flowsheet design for the recovery of various substances by leaching, solvent extraction, precipitation, crystallization, and electrometallurgy operations[16]. This expertise is supported by research projects involving thermal treatments of ores and concentrates (chlorination, sulfate roasting), understanding of solid-gas transfers, and kinetics and thermodynamic modelling[17]. Finally, CNRS/ICSM holds a strong expertise in ionic flotation, a method for ion extraction from an aqueous phase that relies on the selectivity of complexation between a metal ligand and a surfactant. In particular, CNRS/ICSM first identified the superchaotrope effect of nano-sized ion species in 2015, on polyoxometalates (POMs).

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[14] Marc, P. et al. (2018). Epj Nucl Sci Technol, 4, 2.
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# 2. Detailed project description

## 2.1. Project outline, scientific strategy

The ores considered today in the global mining industry have lower grades and finer textures, requiring then more complex separation steps. Therefore, **their sustainable exploitation requires the development of innovative and efficient methods.**

This major scientific challenge will be addressed in InnovTech by **improving state-of-the-art methods and by developing innovative technologies**. For that, disruptive approaches will be implemented including in particular the use of artificial intelligence/machine learning which has rarely been performed before. These approaches will also include DEM and CFD modeling, quantum-chemistry-based atomistic simulations, and thermo-kinetic modelling. They will be combined with original and high-level experimental works in order to get more insights into the phenomena allowing then to design the most relevant and accurate models. These approaches will allow **improving the understanding of the mechanisms involved in the treatment processes** which is essential for the development of innovative methods. This is particularly the case for the mineral processing steps which are often conducted and improved based on know-how and empirical methods. Regarding extractive metallurgy processes, even if more fundamental studies have already been performed, works is still required in order to improve the understanding of the mechanisms associated in particular to the complexity of the current ores and to the innovative processes. These approaches will also be used in the development of innovative characterization methods in support to the possibility to get more detailed characterization results and to their on-line implementation. **InnovTech will then be pioneer in the field of ore characterization, mineral processing, and extractive metallurgy.** Today, many projects focus on lithium extraction considering the large global demand in this commodity. However, a lot of critical raw materials (CRM) identified in the European Union present significant bottlenecks in their processing and, therefore, the choice was made in this PC to focus on CRM other than lithium. In particular, it was chosen to focus on tungsten which has been identified as one of the most critical substance at EU and French level for several years[18,19], and on the ones related to sulfide ores such as Co and Sb and also Cu. It has been chosen to work on Cu even if it is not classified yet as CRM since it is strategic for the emerging technologies.

**Beyond the scientific objectives, InnovTech also aims at federating the scientific community working on ore processing.** It will put together scientific communities working in various fields of geosciences, of process engineering and of computer sciences with a strong link with PC6 and then with the scientific community working in the environmental assessment of processes. The strategy implemented will be strongly based on the development and consolidation of scientific collaborations between the various partners, in particular through the implementation of cotutelle in the supervision of about 20 non-permanent researchers (PhD students, post-doctoral researchers).

[18] BRGM (2022). Fiche de criticité Tungstène
[19] European Commission (2023). Study on the Critical Raw Materials for the EU

# 2.2. Scientific and technical description of the project

## 2.2.1 Objectives

To achieve its main objectives described in section 1.1, InnovTech has defined five High-Level Objectives (HLOs) related to each Work Package (WP), which are described below. These HLOs are broken down into Specific Objectives (SpOs) supported by corresponding Tasks (Table 2.1).

**Table 2.1 : Objectives of InnovTech**

| |
|---|
| **HLO1: To implement an effective management to ensure the project's successful outcomes and the dissemination of the results (WP1. Project management, communication & dissemination)** |
| **HLO2: To develop automatic characterization and data compilation tools for a better knowledge of ores (WP2. Advanced characterization of materials)** |
| SpO2.1. To create large datasets containing SEM, µXRF and optical images of up to 300 samples collected in France and to make them available for the scientific community (T2.1) |
| SpO2.2. To calculate the fundamental sampling error attached to the data acquired by mapping (automatic mineralogy) on samples of various masses and grain sizes using the Gy theory (T2.2) |
| SpO2.3. To bring the monitoring equipment out of the laboratories, closer to the industrial facilities (e.g. crushed material on conveyor belt, pulps in pipes) (T2.3 & T2.4) |
| **HLO3: To develop technological innovations for a more environmentally friendly and a more efficient mineral processing (WP3. Technologies for mineral processing)** |
| SpO3.1. To link particle scale fragmentation properties with the performance of comminution technologies, and to improve the understanding of the mechanisms of an innovative fragmentation technology (T3.1) |
| SpO3.2. To improve the understanding of the phenomena that govern the separation of fine particles in slurry and to develop predictive models for enhanced gravity separation and flotation processes (T3.2) |
| SpO3.3. To develop a comprehensive understanding and modelling of the molecular mechanisms involved in adsorption at mineral/water interfaces for improved flotation performance (T3.3) |
| SpO3.4. To develop a dry electrostatic separation route for processing ores with high separation yields and low energy consumption (T3.4) |
| SpO3.5. To explore the mechanisms involved in thermochemical processing of mine wastes for their valorization as lightweight building materials (T3.4) |
| **HLO4: Design of new processes for the recovery of metals from complex ores by improving the knowledge of the physical-chemistry involved by means of new tools and modelling – Application to tungsten and sulfide ores (WP4. Technologies for extractive metallurgy)** |
| SpO4.1. To develop an integrative flowsheet relying on new chemistry and new technologies to improve the sustainability of tungsten production (T4.1) |
| SpO4.2. To improve the knowledge on the passivation mechanisms occurring during leaching of sulphur minerals and to improve their dissolution in moderate temperature and pressure conditions. (T4.2) |
| SpO4.3. To develop experimental tools for the characterization of suspended solid in dissolution systems as a support to thermo-kinetic models (T4.2) |
| **HLO5: To develop complementary experimental and modelling approaches, including environmental and techno-economic considerations, for the integration of mineral processing and hydrometallurgical processes into sustainable process flowsheets (WP5. Process integration)** |
| SpO5.1. To implement a comprehensive approach to the S and Fe cycle in hydrometallurgical processes for promoting their valorization (T5.1) |
| SpO5.2. To create a predictive tool that allows a multi-scale development of a specific process and to couple process simulation software with LCA software for improving the development of sustainable flowsheets (T5.2 & T5.3) |

### 2.2.2 Methodology

InnovTech is structured into 5 Work-packages (WP), as presented in Figure 1. WP2 is dedicated to the development of advanced characterization methods of materials, which will allow feeding the models developed in the other WPs. The development of innovative processes, including breakthrough technologies, will be performed in WP3 and WP4. These WPs are strongly interconnected to WP2 since characterization is crucial for the development of innovative processes and to WP5 for developing simulation and modelling tools of each unit step integrated in a global flowsheet. Finally, WP1 deals with the project management, communication, and dissemination of the results. An overview of the staff effort is given in Table 2.2.

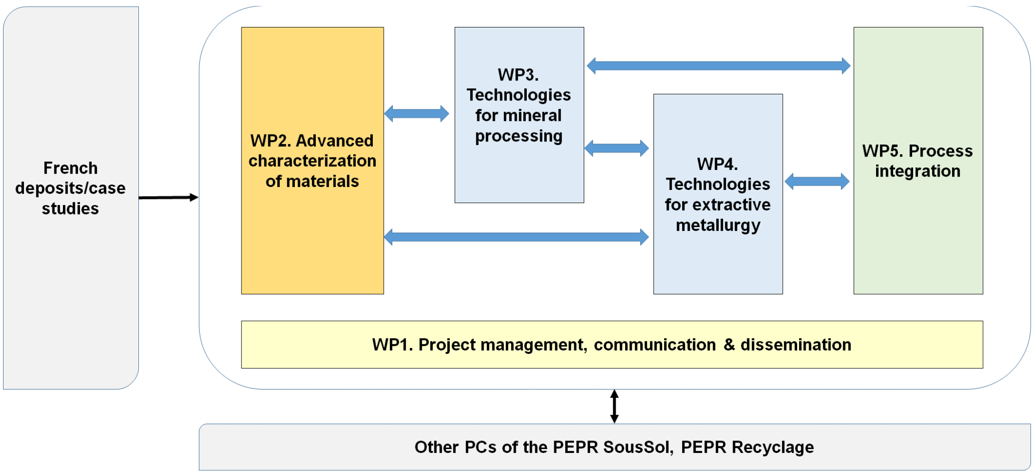


**Figure 1 : WP structure of InnovTech**

**Table 2.2 : Summary of staff effort (Total T/ *Non-Permanent NP*)**

| | WP1 | | WP2 | | WP3 | | WP4 | | WP5 | | Total | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | T | *NP* | T | *NP* | T | *NP* | T | *NP* | T | *NP* | **T** | ***NP*** |
| BRGM | 14.2 | *0.0* | 22.5 | *0.0* | 69.8 | *36.0* | 33.7 | *18.0* | 36.8 | *24.0* | **177.1** | ***78.0*** |
| UL | 8.0 | *0.0* | 66.0 | *60.0* | 162.0 | *144.0* | 58.0 | *48.0* | 0.0 | *0.0* | **294.0** | ***252.0*** |
| CNRS/LRGP | 0.0 | *0.0* | 0.0 | *0.0* | 0.0 | *0.0* | 0.0 | *0.0* | 0.0 | *0.0* | **0.0** | ***0.0*** |
| CNRS/ICSM | 0.0 | *0.0* | 0.0 | *0.0* | 0.0 | *0.0* | 22.2 | *18.0* | 0.0 | *0.0* | **22.2** | ***18.0*** |
| CNRS/LGC | 0.0 | *0.0* | 0.0 | *0.0* | 0.0 | *0.0* | 4.0 | *0.0* | 0.0 | *0.0* | **4.0** | ***0.0*** |
| CNRS/LGF/MSE | 0.0 | *0.0* | 0.0 | *0.0* | 19.0 | *15.0* | 15.0 | *12.0* | 0.0 | *0.0* | **34.0** | ***27.0*** |
| INP/LGC | 0.0 | *0.0* | 39.0 | *36.0* | 0.0 | *0.0* | 12.0 | *12.0* | 12.0 | *12.0* | **63.0** | ***60.0*** |
| MINES Paris | 0.0 | *0.0* | 0.0 | *0.0* | 15.0 | *12.0* | 0.0 | *0.0* | 0.0 | *0.0* | **15.0** | ***12.0*** |
| UPPA | 0.0 | *0.0* | 0.0 | *0.0* | 0.0 | *0.0* | 12.9 | *12.0* | 0.0 | *0.0* | **12.9** | ***12.0*** |
| CEA | 0.0 | *0.0* | 43.2 | *36.0* | 0.0 | *0.0* | 66.2 | *48.0* | 8.4 | *6.0* | **117.8** | ***90.0*** |
| **Total PMs** | **22.2** | **0.0** | **170.7** | ***132.0*** | **265.8** | ***207.0*** | **224.0** | ***168.0*** | **57.2** | ***42.0*** | **740.0** | ***549.0*** |

Close links with other PCs of the PEPR SousSol will be performed all along the project lifetime, in particular with PC6 for the environmental assessment of the processes under development, PC8 for the legal aspects related to the mining activities and with PCs related to the study sites such as PC11 (French Guiana) and PC13 (Massif Central). Close links will also be performed with other PEPR, such as the PEPR Recyclage. Moreover, the works performed in the first years of InnovTech will allow identifying relevant ideas and proposals that could be submitted to the calls for Expression of Interest and to the calls for collaborative projects. A special attention will be given to involve actors from several PCs, in particular the ones related to the study sites, to favour transdisciplinary works and to enhance the implementation of the results to French case studies.

## WP1: Project management, communication & dissemination (Leader: BRGM)

### Task T1.1. Project management and reporting (Leader: BRGM - Participants: UL/GeoR, WP Leaders)

**The Management Committee (MC) will be the operational management body of the project responsible for monitoring the project at a scientific level.** MC will be chaired by the coordinator (BRGM) and will involve the associated project manager (UL/GeoR) and the leaders of the WPs. They will work in close collaboration with the co-directors of the PEPR SousSol, with the coordinators of the other PCs of the PEPR SousSol, and with the Partners of the other PEPR (such as the PEPR Recyclage) for promoting links between InnovTech and other PCs or other PEPR. The coordination will be done by a detailed monitoring of the progress of each WP and corrective measures will be taken if needed. MC meetings will be organised twice a year (one online and one face-to-face). The project coordinator will prepare all the documents required by the ANR with the contribution of WP leaders. The reports will be prepared in collaboration with project Partners.

### Task T1.2. Legal and financial aspects (Leader: BRGM - Participants: UL/GeoR)

**The coordinators will provide support to the Partners on legal and financial aspects of the project issues**. In particular, BRGM and UL/GeoR are responsible for establishing the Consortium Agreement (CA) in which Intellectual Property (IP) management between the Partners will be defined, and for preparing other legal documents for the project. Financial management of the project will follow the guidelines given by the ANR.

### Task T1.3. Communication, dissemination, and networking (Leader: UL/GeoR - Participants: all)

**Communication and Dissemination plays a major role to maximize the impact of InnovTech on the scientific and industrial communities at the French and at the international levels.** The MC will define the communication/dissemination/networking actions according to the project achievements. More details are given in Section 4.

## WP2: Advanced characterization of materials (Leaders: UL/GeoR & BRGM)

### Task T2.1. Multipurpose database and sample collection (Leader: UL/GeoR - Participants: BRGM)

**The objective is to create a collection of samples and an associated database**. Up to 300 samples collected mainly from the PEPR SousSol will be prepared with a preserved part (hand piece or core), a flat sawed slice, at least one thick section and one thin polished section, and a powder. Samples are expected from InnovTech partners and from other PCs of the PEPR Sous-Sol. Samples outside the PEPR will be selected if required to cover French rock types (mainland and overseas) not available internally. Optical photography will be done on all sub-samples. MLA (Mineral Liberation Analysis) will be done on the flat slice and thin section using µXRF and SEM-EDS, respectively. That should produce up to one million reference spectra associated with a mineral name. A classification approach (link with T2.3) will then allow the automatic recognition of a mineral via the acquisition of a new spectrum, hence rendering automated point counting available. A transfer function will be studied to make automatic recognition of a mineral and automated point counting feasible in all facilities equipped with µXRF or SEM-EDS. The collection of samples will be made available to the community as well as their complementary measurements or observations.

### Task T2.2. Sampling methods (Leader: BRGM - Participants: UL/GeoR, UL/CRAN)

Automated mineralogical systems are increasingly used to characterize solids and particles (rock thin sections, waste rocks, tailings, soils, sediments, etc.). Although some of these devices enable quantitative determination (of a targeted phase content, of the liberation size, *etc.*), a detailed understanding of the representativeness of these results compared to the large volume of corresponding material is rarely discussed. Jones and Cheung (1988)[20] illustrated this problem by giving the example of a gold ore of 1 g/t Au. If the grain size of the free gold is 1 µm, it is necessary to analyse two polished sections to be theoretically representative. If the grain size is 100 µm, it takes about 20 000 polished sections (one polished section = approximately 1 g of solid).

**This task proposes to evaluate the sampling error and/or the confidence interval with respect to the data provided by the analytical tool**. This calculation will be correlated to the number of particles analysed, the particle size and the intrinsic properties of the material considered. A methodology will be designed to determine the sample preparation method to implement and the appropriate number of "sample" (as polished section) to consider reaching a given representativeness level. This work will be performed on some of the samples analysed in T2.1.

### Task T2.3. Signal and image processing methods (Leader: UL/CRAN - Participants: BRGM, CEA, UL/GeoR)

In Task T2.3, **machine learning methods will be investigated to develop innovative processing of datasets produced in T2.1**. Three aspects will be covered:

1. To provide a methodological support to T2.1 on the classification of spectral data.
2. To estimate the (possibly non-linear) mapping between two datasets of the same sample acquired on two different instruments so that the automated point counting method becomes transferable to the entire community.
3. To combine and to merge images acquired by multimodal imaging systems, including RGB, VNIR and SWIR measurements.

The complexity of data processing will increase from simple approaches (correlation and search for trends) to more demanding (un)supervised algorithms based on low-rank tensor decompositions. In this task, the objective is to bring monitoring equipment out of the laboratories, closer to the transit areas and to analyse the composition of crushed material directly on conveyor belts. This implementation will pave the road to the development of integrated methods that combine automated in-situ characterization stages and numerical-based process adaptations (digital twin). In particular, this task aims at building an automated equipment able to analyse materials directly on conveyors, in the processing plants.

### Task T2.4. In-line monitoring for liquid and pulps (Leader: CEA - Participants: UL/CRAN)

**The aim of T2.4 is to develop online monitoring tools for real-time data acquisition on solutions containing particles (i.e. slurries) in pipes**. The tool will be developed from an existing device dedicated to solutions and composed of an acquisition chain (Probes, fibers, UV-Visible-IR and Raman spectrometers). The data will then be processed by a digital spectra deconvolution tool. The use of IR spectroscopy in Attenuated Total Reflectance (ATR) mode is one option that could be considered. Hardware and software adaptations and optimizations will be made to switch from the monitoring of solutions to that of particles suspended in water. Data fusion approaches will be mutually beneficial between T2.3 (conveyor belts) and T2.4 (pipes).

[20] Jones, M.P. and Cheung, T.S. (1988). Automatic method for finding gold grains in ores and mill products. In Proceedings of the Asian Mining Conference; Institution of Mining and Metallurgy: London, UK, pp. 73–81.

## WP3: Technologies for mineral processing (Leaders: INP/LGC & BRGM)

For this WP, it was chosen to work on scheelite skarns from the "French Massif Central study site" (PC13). This choice is motivated by the high specific energy required for grinding this type of ore and by the natural friability of scheelite. In such ores, the comminution challenge is to avoid producing excessive amounts of fine valuable particles that are difficult to recover or lost in the fine products. Since InnovTech should start before PC13[21], and since organizing and collecting sample may require several months, the works of WP3 will start using a Portuguese scheelite skarn ore that presents many similarities with French scheelite skarns and for which several hundred kg are already available at UL/GeoR and BRGM facilities. Among similarities of this Portuguese skarn ore with the French ones, it can be mentioned its fine liberation size (below 200 µm), which makes the use of a froth flotation processing stage almost mandatory, and a similar mineralogical composition (considering for example ores from Fumade, Coat-an-Noz, and other French sites). Another advantage of considering this Portuguese ore in a first approach is that the characterization of its chemical, mineralogical, and physical properties has already been carried out in a previous project. Using this Portuguese sample will then allow starting the exploratory works as soon as the samples are received by the partners. The results obtained with this sample will be capitalized, fine-tuned, and validated using the French samples once they will be available.

### Task T3.1. Fragmentation properties of ores and study of an innovative fragmentation technology (Leader: INP/LGC - Participants: BRGM, LGF/MSE, MINES Paris, UL/GeoR)

Generally, comminution uses 50% of the world mineral processing plants' energy consumption; hence, energy optimization of industrial grinding technologies remains an industrial priority. **T3.1 tackles the central issue of the *mass specific comminution energy for a given degree of fragmentation and/or mineral liberation* from both fundamental and technological ends**.

- Sub-Task ST3.1.1. Closing the gap between elementary ore fragmentation properties and the performances of comminution technologies (Leader: INP/LGC - Participants: BRGM, LGF/MSE)

Historically, comminution process research has been relying upon an equipment-to-particle top-down approach, which has reached limits for process optimization. **ST3.1 proposes to push the boundaries of comminution process design and optimization by building a bridge between the elementary fragmentation properties of an ore and the performance of comminution machines through a bottom-up methodology.** The works will start from the generation of particle-scale fragmentation data using both dedicated auscultation tools and DEM simulation of the elementary fragmentation events measured on these tools. Coupled machine learning and phenomenological models will then be used to capitalize the large quantity of fragmentation data produced to fill the gap between particles' elementary fragmentation properties and the performance of a selection of comminution machines, including the innovative electrofragmentation technology investigated in ST3.1.2. In doing so, the intended work will derive ore fragmentation indices that capture the "mechanical texture" of an ore as opposed to its "mineralogical texture".

- Sub-Task ST3.1.2. Investigation of the electrofragmentation technology for ore treatment (Leader: BRGM - Participants: MINES Paris, UL/GeoR)

**The objective of ST3.1.2 is to investigate the potential of an innovative fragmentation technology that uses high-voltage pulses, known as electrofragmentation, which has the potential to both reduce comminution energy and improve the liberation of mineral phases.**

[21] The submission date of InnovTech is planned for May 2023 while the submission date of PC13 is planned for September 2023.

ST3.1.2 will include works in order to understand and model the fragmentation mechanisms of ores in presence of an electric field by combining theoretical and experimental methods. Works will also be done with a lab-scale equipment for evaluating the performance of electrofragmentation in terms of energy consumption and mineral liberation and to investigate its potential benefit on subsequent separation stages, particularly flotation.

### Task T3.2. Development of hydrodynamic approaches for the beneficiation of fine suspensions (Leader: UL/IJL - Participants: UL/GeoR)

**This task aims at improving the understanding and control of flows and hydrodynamic interactions (in addition to mechanical and physical-chemical interactions) in separation processes in order to improve their selectivity.**

- Sub-Task ST3.2.1. Intensification of fine and coarse particle flotation by coupling physicochemical and hydrodynamic approaches (Leader: UL/GeoR)

**ST3.2.1 aims at improving flotation selectivity by coupling physicochemical and hydrodynamic approaches.** Works will first investigate the use of acoustic wave energy (ultrasonic) and hydrodynamic innovations (new concept of flotation cell) for the intensification of the flotation process. They will then be dedicated to the intensification of the coarse particles' flotation using the new concept of Combined Microbubbles Flotation, in particular through a fundamental investigation of the role of the bubble size (from nano to micrometric) on heterocoagulation processes.

- Sub-Task ST3.2.2. Modeling of gravimetric separation processes by physical analysis and numerical simulation of transport and interactions between particles (Leader: UL/IJL - Participants: UL/GeoR)

**The objective of ST3.2.2 is to improve separator models by improving the way the hydrodynamic effects are included**. This subtask relies on numerical simulations to quantify parameters and effects that are not accessible experimentally (such as the hydrodynamic interaction forces between fine particles). The Falcon centrifugal separator will be considered as a first case study since it represents a disruptive technology to tackle the challenge of fine particles processing.

### Task T3.3. Fundamentals of flotation (Leader: UL/GeoR - Participants: UL/LPCT)

- Sub-Task ST3.3.1. Understanding the hydration mechanisms of mineral surfaces involved in scheelite flotation (Leader: UL/GeoR - Participants: UL/LPCT)

**ST3.3.1 will investigate the influence the surface speciation of minerals as a function of the pH from tungsten skarns by combining experimental and theoretical techniques**. For that, *ab initio* molecular dynamics simulations combined with artificial intelligence will be used to calculate surface acidity constants. Adsorption (isotherms) and spectroscopic (XPS, non-linear optics) techniques will be applied to study the interaction of water at various pH with the surfaces of the considered minerals. All the models will be shared with the scientific community since they could be useful for many other fields (water splitting, dye-sensitized solar cells, etc.).

- Sub-Task ST3.3.2. Understanding the interactions between flotation reagents and minerals at the nanoscale (Leader: UL/GeoR - Participants: UL/LPCT)

**The objective of ST3.3.2 is to investigate the adsorption mechanisms of various flotation reagents on the mineral surfaces related to tungsten skarns ores.** Works will include advanced simulation techniques combined with various experimental analysis methods (isotherms, XPS, DRIFTS). Special attention will be taken to consider the ions influence since it is known that their adsorption on surfaces can have a significant impact on flotation performances. The synergistic effects of collectors and co-collectors will also be investigated, in particular through molecular simulations, as it could allow to identify solutions for improving the selectivity of the collectors.

- Sub-Task ST3.3.3. Development of an efficient scheelite flotation process (Leader: UL/GeoR)

**ST3.3.3 aims to develop new formulations of scheelite-selective, efficient, sustainable, and economically viable reagents formulations for extracting scheelite from skarns.** Various experimental techniques will be implemented in order to select the best molecules, with an emphasis on the use of bio-sourced and biodegradable molecules. Works will also include the development of a mesoscopic model describing and predicting flotation performance based on the physicochemical properties of the systems. In order to feed the developed numerical model, an on-line control of the operating conditions and of various parameters of the flotation process will be implemented.

### Task T3.4. Water saving technologies for metal recovery and valorization of mining waste (Leader: BRGM - Participants: UL/GeoR)

Significant amounts of mining waste have been generated over the past centuries by the mining industries, which contain significant amounts of valuable metals and minerals that could be recovered using innovative technologies. **The objective of T3.4 is to investigate innovative methods to recover the metals and to valorize mining waste without the use of water.**

- Sub-Task ST3.4.1. Development of an innovative sorting method based on triboelectrostatic separation for metal recovery from mining waste (Leader: BRGM - Participants: UL/GeoR)

**This ST aims at investigating and optimizing the triboelectric separation to recover the metalliferous phases contained in the mining waste**. Different parameters will be considered, using a design of experiments methodology, to investigate the effect of each parameter on the separation efficiency. The products generated from each test, *i.e.*, the conductor fraction (metal-rich fraction) and the non-conductor fraction (mineral fraction containing silicates for example) will be analyzed to perform mass balances. The non-conductor fraction of the test giving the best results will be used as a basis of the works planned in ST3.4.2.

- Sub-Task ST3.4.2. Thermochemical valorization of mining waste into lightweight building materials (Leader: UL/GeoR - Participants: BRGM)

**ST3.4.2 aims at valorizing mining wastes by transforming them into lightweight construction materials (LWMs) using a thermochemical process**. The specific objectives are to immobilize several elements (especially heavy metals) and/or separate them by selective extraction during LWMs synthesis. The works will cover the understanding of the expansion process, the thermal synthesis of lightweight materials and the evaluation of the functional properties of LWMs. Several properties such as density, porosity, water absorption, mechanical strength will be measured. A particular attention will be devoted to the status and immobility of heavy metals in the LWMs as well as energy efficiency and environmental performances. This will allow defining an innovative sustainable approach for the transformation of mining residues into valuable products.

## WP4: Technologies for extractive metallurgy (Leaders: UL/GeoR & CEA)

### Task T4.1. New chemistry for the development of a sustainable process to extract tungsten from ores (Leader: UL/GeoR - Participants: BRGM, CEA, CNRS/ICSM, CNRS/LRGP, LGF/MSE)

Conventional processes for tungsten concentrates usually implement the following operations: alkali digestion to produce a sodium tungstate, purification steps to remove impurities, and crystallization to produce ammonium paratungstate, which can be calcined into tungsten oxide. The alkali digestion usually operates at 175-200 °C and under a pressure up to 3 MPa in the presence of 10-20% sodium hydroxide for 3-4 hours. The purification stage of the leach solution usually involves solvent extraction at acidic pH with amine diluted in kerosene modified with isodecanol as extraction solvent. Therefore, the leaching conditions are hard, and the reagent consumption may be important. **T4.1 aims at developing an innovative flowsheet, including new technological bricks, to improve the sustainability of tungsten production.** T4.1 will have strong interaction with WP3 since works planned in T4.1 will be performed on the tungsten concentrate that will be produced in T3.3.

- Sub-Task ST4.1.1. Upstream processing (Leader: BRGM - Participants: UL/GeoR, CEA, CNRS/ICSM, CNRS/LRGP, LGF/MSE)

**ST4.1.1 aims at developing an appropriate pre-treatment (thermal treatment, microwaves, high-energy grinding, or ionic flotation) to facilitate leaching under soft conditions.** Thermodynamic calculations will be performed to figure out new approaches to convert tungsten-bearing mineralogical phases into tungsten salts highly soluble in slightly acidic or alkaline media under mild conditions. The mechanisms involved at the solid/liquid interface will be investigated to find ways to improve the kinetic of the thermal treatment. In parallel, direct leaching of the concentrate with a combination of appropriate reagents and microwave, use of DES as new leaching reagent as well as leaching combined with high-energy grinding will be investigated to leach tungsten concentrate under mild conditions.

- Sub-Task ST4.1.2. Tungsten extraction, concentration and/or purification (Leader: UL/GeoR - Participants: CEA, CNRS/ICSM, UL/LRPG)

**In ST4.1.2, new extracting agents will be synthesized and their extraction properties towards tungsten and the main impurities will be investigated in order to identify the most interesting extractant for its implementation in a flowsheet.** Works will include formulation of the extraction solvent which will then be tested on the leaching solutions. The results will be used to propose a physicochemical model to describe tungsten and impurities extraction and to design the solvent extraction flowsheet. This model will be implemented in the Parex+ code in order to simulate the process and optimize it. Finally, the flowsheet will be tested in laboratory-scale mixer-settlers in order to produce a purified tungsten solution.

A special attention will be paid to combine all these treatment steps in order to reduce reagent and energy consumption as well as effluent generation while keeping the process cost as low as possible.

### Task T4.2. Thermo-kinetic modelling and experimental data acquisition at different scales for the study of dissolution/precipitation mechanisms involved in leaching processes (Leader: CEA - Participants: BRGM, CNRS/LGC,CNRS/ICSM, UPPA)

**The objective of T4.2 is to acquire experimental data coupled with thermo-kinetic modelling at different scales (from the microscopic to the reactor scale) to study dissolution and precipitation mechanisms occurring in leaching processes**. Sulphur minerals, which are most of the time refractory to the dissolution at atmospheric pressure and moderate temperature, will be considered here since they constitute an interesting case study. Indeed, the nature of the species and even the physicochemical mechanisms responsible for this passivation remain controversial (elementary sulfur, polysulfur, neo-formed phases, etc.)[22] and, currently, high-temperature and high-pressure lixiviation processes have to be implemented to avoid this passivation phenomena and

[22] Ajeddig, Y. (2022). PhD on « Modélisation mathématique de l’oxydation des minerais sulfurés en réacteur chimique triphasique discontinu : application à la biolixiviation des minerais sulfurés », Latep, BRGM.

keep fast kinetics. Different innovative approaches will be implemented to produce the experimental data needed for the modelling:

- Sub-Task ST4.2.1. Mechanically assisted dissolution of refractory mineral phases for improving the understanding of the passivation mechanisms (Leader: CNRS/LGC - Participants: BRGM, CEA)

**Elimination of sulphide minerals' particle passivation layers will be studied in ST4.2.1** by *in situ* mechanical abrasion (using millimeter-sized grinding glass beads stirred in the pulp) or *in situ* high-energy grinding (at nanometric scale). Experimental campaigns will aim at exploring the impact of the size of the grinding beads as well as of the redox potential, which is a key parameter in sulphide leaching chemistry. The introduction of bacteria in the reactor, in order to promote bioleaching, will also be investigated.

- Sub-Task ST4.2.2. Development of an experimental device allowing the determination of microscopic dissolution kinetics and in-situ observation of neoformed phases (Leader: CEA - Participants: BRGM)

**The objective of ST4.2.2 is to develop a simple and low-cost microscopic instrument**, based on a lens-less imaging system coupled to a microfluidic observation cell, capable of characterizing solid suspensions in a corrosive environment in a liquid volume of millimeter thickness. The final tool aims to allow a drastic acceleration of the acquisition of experimental data and to propose a deeper characterization of solid particles including not only the size of the particles but also relevant information such as the crystalline facies or the roughness.

- Sub-Task ST4.2.3. In-operando observation of mineral phases dissolution using Environmental Scanning Electron Microscopy (ESEM) and Energy Dispersive X-ray Spectroscopy (X-EDS) (Leader: CNRS/ICSM).

**The methodology that will be implemented in ST4.2.3 is based on the acquisition of micrographs and elemental maps of a region of interest at the surface of the dissolving sample**[23]. This method will be tested on ore concentrates and the compilation of the images obtained with time will allow evaluating the dissolution kinetics of the mineral phases according to the dissolution conditions defined in ST4.2.1 and 4.2.2. This work will allow a better understanding of the mechanisms of dissolution and precipitation of secondary phases with the aim to optimize the recovery of the elements of interest in the carrier phases.

- Sub-Task ST4.2.4. Thermo-kinetic modelling of a hydrometallurgical reactor and acquisition of thermodynamic properties of neo-formed solid phases (Leader: UPPA - Participants: CNRS/LGC)

**ST4.2.4 will work on the improvement of a macro model which simulates the bioleaching of pyrite in a batch reactor**. This sub-task will focus on enhancing the expression of the precipitation kinetics and the description of solid solutions. Some key thermodynamic data will be acquired at CNRS/LGC, with a specific focus on the behavior of jarosite compounds (calorimetric properties and mixing properties).

## WP5: Process integration (Leaders: BRGM & CEA)

Process integration is crucial in the development of sustainable technologies, considering not only the techno-economic criteria but also environmental aspects. In particular, it is essential to consider these criteria as early as possible since they can be used as a support tool to improve the performances and the sustainability of the whole flowsheet. **The objectives of WP5 are three-fold:**

[23] Podor, R. et al. (2019). 3D-SEM height maps series to monitor materials corrosion and dissolution. Mater Charact, 150, 220.

- **To address a comprehensive approach to the S and Fe cycle**, which could be beneficial for many hydrometallurgical processes. This could facilitate the definition of new flowsheets that integrate waste streams as new resources.
- **To create a predictive tool that allows a multi-scale development of a specific process**, including a comprehensive evaluation in terms of environmental impacts, energy consumption and water resource management.
- **To couple process simulation software with LCA software** to improve the efficiency of (bio)chemical process LCAs, which in turn serves as a decision-making tool at early stages of technology development.

Works performed will mainly be focused on case studies coming from the “French Massif Central study site” (PC13), and particularly on case studies with critical raw materials such as skarn or sulfidic ores. However, other study sites (such as French Guyana and Rhine graben) can be considered, depending upon the type of resources targeted (sulphide-containing or oxidized metallic ores).

### Task T5.1. Minimization of waste streams and increase of circularity (Leader: BRGM - Participants: CNRS/LGC)

**The study of the key reactions of bio-oxidation and bio-reduction of Fe and S, from a chemical, kinetic and thermodynamic point of view will be performed** to find new alternatives of valorisation of these elements. The works will include a theoretical part with modelling and bibliographic studies. Experimental works will also be performed, aiming in particular to measure the kinetics under non-restrictive conditions and to compare chemical and biological processes.

### Task T5.2. Development of predictive process simulation tools by integrating kinetic and thermodynamic models in simulation software (Leader: BRGM)

Selected kinetic and thermodynamic models developed in T5.1, as well as in other WPs of InnovTech, **will be integrated using the most adapted simulation software** (USIM PAC, SysCAD, HSC Chemistry) with the objective to couple them and to produce reliable data. Different scenarios will be tested by changing either the input (e.g type of ore, reagents) or the process operating conditions.

### Task T5.3. Inclusion of LCAs as a decision-making tool in process development and integration (Leader: BRGM - Participant: CEA)

A communicating pathway between process simulation software, including not only mass balance but also economic costs, and environmental LCA software will be created. In particular, BRGM will combine existing simulators developed in previous projects (for instance using USIM PAC for mineral processing and/or SysCAD for biohydrometallurgical processes) with LCA software such as GaBi or SimaPro. For dissolution and downstream processes, CEA will couple the numerical tool PEPS, currently under development at CEA, with GaBi or SimaPro. The final objective is to combine both BRGM and CEA approaches from mineral processing to downstream processes simulations with LCA tools for being able to identify the origin of the main environmental impacts. T5.3 will be performed in close collaboration with works performed in PC6, through a post-doc position (BRGM) and a short-term contract (CEA) shared between these two PCs. **This eco-design approach will allow fine-tuning the flowsheet under development to reduce not only the environmental impacts of the flowsheet but also to improve its technical-economic feasibility.**

## 2.3. Planning and KPI

InnovTech will run 6 years, from 2024 to 2029. Timing of the project is given in the Gantt chart (Figure 2). Most of the works will be performed during the four first years of the project as it is planned that the last years of the project are dedicated to projects obtained under the calls for Expression of Interest and under the calls for collaborative projects that will be opened all along the PEPR SousSol lifetime. These projects could be related either to topics that will not be investigated in details during the first years of InnovTech due to the limited budget available or not investigated at all but still identified as addressing interesting challenges such as valorisation of mining waste and co-processing of various kind of waste, or they can deal with ideas deriving from the first results obtained in InnovTech.

**Figure 2: Gantt chart of InnovTech**

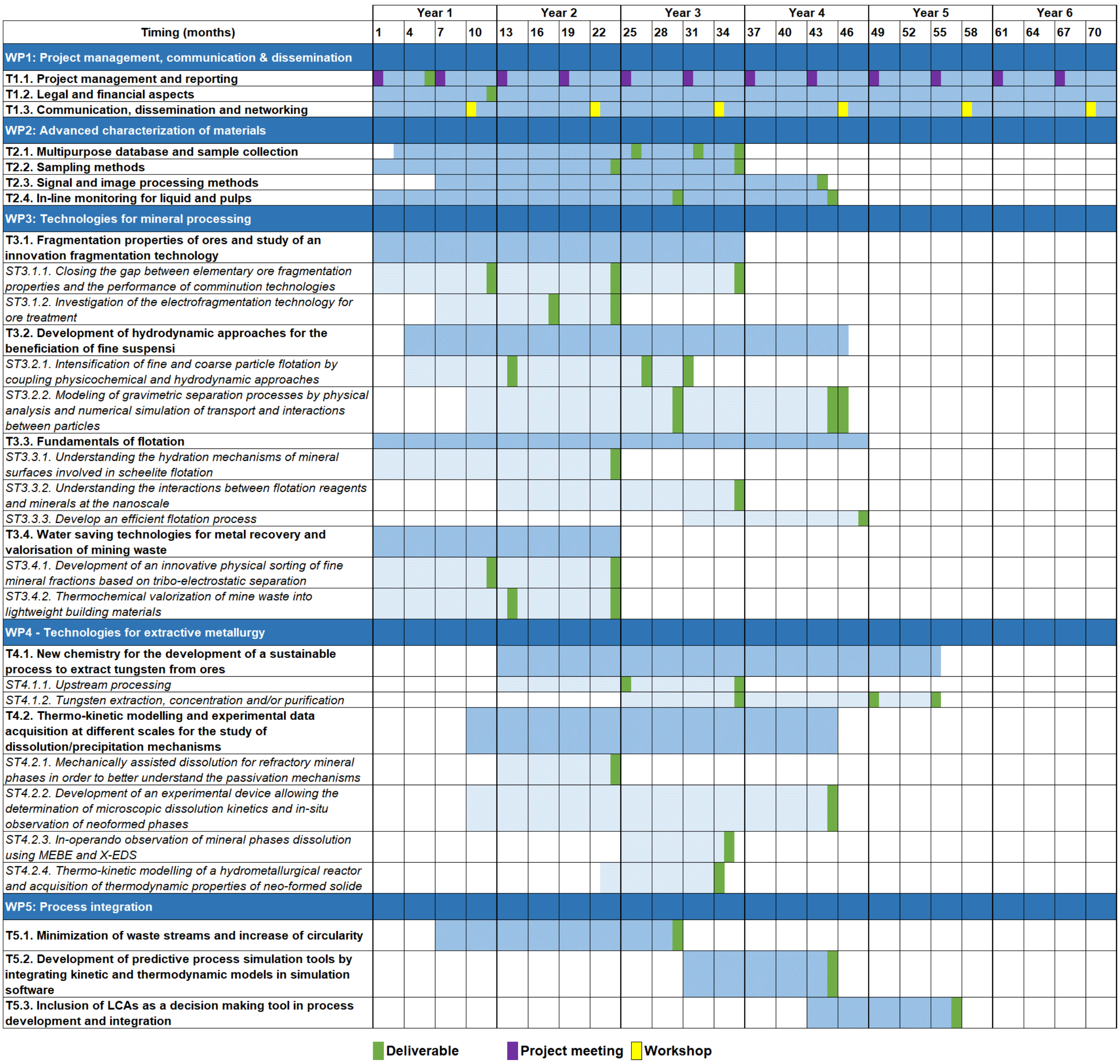

**Table 2.3 : List of deliverables**[24]

| # | Title (Led by, Type, Due (Month)) |
|---|---|
| **D1.1.1** | Data Management Plan (BRGM / DMP / M6) |
| **D1.2.1** | Consortium Agreement (BRGM, CA, M12) |
| **D2.1.1** | Collection of samples and subsamples physically installed in Nancy (UL/GeoR, S, M26) |
| **D2.1.2** | Database with sample name, preparation methods, optical images and µXRF + SEM-EDS data (UL/GeoR, D, M32) |
| **D2.1.3** | Transfer function for use of automated spectrum identification in other facilities (UL/GeoR, R, M36) |
| **D2.2.1** | Sampling methodology (BRGM, R, M24) |
| **D2.2.2** | Estimation of uncertainty for measuring metals content by automated mineralogy system (BRGM, R, M36). |
| **D2.3.1** | Data Fusion and Clustering: Algorithms and Results (UL/CRAN, M, M44) |
| **D2.4.1** | Publication on ATR analysis technique and related models (CEA, P, M30) |
| **D2.4.2** | Manuscript of the PhD performed in Task T2.4 (CEA, M, M45) |
| **D3.1.1** | State of the art on modeling ore fragmentation (INP/LGC, R, M12) |
| **D3.1.2** | State of the art on DEM models used in ore fragmentation (LGF, R, M12) |
| **D3.1.3** | Modelling of electrofragmentation mechanisms (MINES Paris, R, M18) |
| **D3.1.4** | Comparison of performances of a comminution flowsheet with electrofragmentation to a conventional one (BRGM, R, M18) |
| **D3.1.5** | Particle-scale fragmentation results from experimental and DEM simulation works and first links with machine learning approaches (INP/LGC, R, M24) |
| **D3.1.6** | Influence of electrofragmentation on performances of concentration steps (UL/GeoR, R, M24) |
| **D3.1.7** | Manuscript of the PhD performed in Sub-Task ST3.1.1 (INP/LGC, M, M36) |
| **D3.1.8** | Links between the elementary fragmentation properties of an ore and the performances of comminution machines (INP/LGC, R, M36) |
| **D3.2.1** | Influence of use of acoustic wave energy in flotation (UL/GeoR, R, M14) |
| **D3.2.2** | Jigging separation physical model and principles of the separation (UL/IJL, R, M14) |
| **D3.2.3** | Effect of micro and nanobubbles size on flotation separation performances (UL/GeoR, R, M27) |
| **D3.2.4** | Empirical and phenomenologic models for Falcon centrifugal separation (UL/IJL, R, M30) |
| **D3.2.5** | Control of the local hydrodynamic regime and bubble size distribution (UL/GeoR, R, M31) |
| **D3.2.6** | Model for Falcon separation based on experiments and numerical simulations (UL/IJL, R, M45) |
| **D3.2.7** | Models for gravity separation methods applied to fine particles processing (UL/IJL, M, M46) |
| **D3.3.1** | Hydrated molecular models of scheelite, fluorite, calcite and dolomite for different pH (UL/GeoR, R, M24) |
| **D3.3.2** | Database of computed adsorption enthalpies of flotation reagents on the four minerals (UL/LPCT, D, M36) |
| **D3.3.3** | Optimized process flowsheet for ultimate scheelite concentration (UL/GeoR, R, M48) |
| **D3.4.1** | Mechanism and preliminary results of physical sorting of mining waste by electrostatic separation technique (BRGM, R, M12) |
| **D3.4.2** | Characterization of LWM obtained with thermal synthesis (UL/GeoR, R, M14) |
| **D3.4.3** | Final report on electrostatic separation of mining waste (BRGM, R, M24) |
| **D3.4.5** | Definition of the adequate process and equipment for thermal expansion of the mine waste (UL/GeoR, R, M24) |
| **D4.1.1** | Influence of pre-treatment on W leaching from scheelite samples (CNRS/ICSM, R, M25) |
| **D4.1.2** | Kinetic study during thermal pre-treatment (LGF/MSE, R, M25) |
| **D4.1.3** | Thermal treatment of ores and concentrates with appropriate reagents to facilitate upstream operations (UL/GeoR, R, M25) |
| **D4.1.4** | Synthesis of amine extractants (CNRS/LRGP, R, M34) |
| **D4.1.5** | Tungsten alkaline leaching assisted by microwave irradiation (BRGM, R, M36) |
| **D4.1.6** | Solvent extraction flowsheet based on the use of a new extractant and physicochemical model of extraction (UL/GeoR, R, M49) |
| **D4.1.7** | Calculation of an optimized flowsheet for W recovery and purification in mixer-settlers (CEA, R, M55) |
| **D4.2.1** | Process capabilities using mechanically assisted leaching on sulphide minerals (CNRS/LGC, R, M24) |
| **D4.2.2** | Development of the thermo-chemical modelling of a hydrometallurgical reactor (UPPA, R, M34) |
| **D4.2.3** | In operando observation of the dissolution of mineral concentrates by ESEM and EDS (CNRS/ICSM, R, M35) |
| **D4.2.4** | Development of an experimental device allowing the determination of microscopic dissolution kinetics and in-situ observation of new phases (CEA, R, M45) |
| **D5.1.1** | Biochemical cycle of Fe and S in (bio)hydrometallurgical processes (BRGM, R, M30) |
| **D5.2.1** | Integration of kinetic and thermodynamic models on bioleaching process simulation (BRGM, R, M36) |
| **D5.3.1** | Coupling process simulation and LCAs - Report on methodology + case study (CEA, R, M42) |

Key Performance Indicators (KPI) will be used to follow progress of InnovTech and corrective actions will be taken if some KPI are not representative of a high-level scientific project. They will be chosen based on the ones considered in the PC Governance of the PEPR SousSol. This will allow optimizing the management of InnovTech and of the PEPR SousSol as a whole. In particular, these KPI include: the number of scientific articles published in international peer-reviewed journal, the number of

[24] DMP: Data Management Plan, CA: Consortium Agreement, D: Database, P: Publication, M: PhD manuscript, R: Report, S: Sample collection

presentations to international conferences, the number of patents submitted, the percentage of PhD students and postdoctoral researchers that are hired on permanent positions after one year following the end of their contract, the number of scientific articles that are co-authored by at least one partner from InnovTech and one partner from another PC and the ones that are co-authored by at least one partner from InnovTech and one partner from another PEPR, the number of scientific events organized in InnovTech and the number of the participants, the number of proposals submitted to French and European calls for projects including at least two partners of InnovTech, the number of commercial projects directly or indirectly related to InnovTech, the number of collaborations between partners of InnovTech and companies.

# 3. Project organisation and management

## 3.1. Project manager

**Kathy BRU** is a senior expert in process engineering, graduated in 2005 from IMT Mines Albi (France). She has more than 17 years of experience in mineral processing at BRGM. Her research interests are focused on the comminution processes, and more especially on the selective fragmentation processes, and on the gravity and magnetic separation technologies. She works on the mineral processing of primary and secondary resources. She is involved as coordinator or WP leader in several European and French funded projects dealing with the mineral processing of ores or with the recycling of waste for a sustainable supply of raw materials in Europe. She has published around 30 papers in international journals and international conference proceedings and has given keynotes in conferences. She is also acting as a reviewer for international journals.

## 3.2. Organization of the partnership

InnovTech has 8 partners covering the whole value chain of raw materials production, from characterization of materials to metal recovery, including skills related to technical, economic, and environmental assessment of processes. The consortium is complementary and well balanced, as described below.

**BRGM**, the InnovTech coordinator, is France's leading public institution in the earth science field for the sustainable management of natural resources (including mineral resources) and surface and subsurface risks. BRGM is actively involved as a coordinator or a partner in several European and French projects dedicated to the production of raw materials including works on the development of innovative sustainable processes, on Life Cycle Assessment and on sustainability indicators.

**CNRS/UL** is Europe's leading University in Mining and Minerals Engineering according to the Shanghai Ranking. It covers the entire mineral resources value chain, from academic understanding of the Earth system to exploration, exploitation, processing, manufacturing, recycling, mine closure and underground storage. **Yann Foucaud**, who is the associated project manager of InnovTech, works for UL/GeoR as associate professor in mineral processing and applied mineralogy. He develops research about fundamental physical and physicochemical mechanisms involved in the separations, mainly in froth flotation, by theoretical and experimental methods.

**CEA** is a French R&D organism specialized in defense and security, low carbon energies, technological research for industry, and fundamental research in the physical sciences and life sciences. CEA is actively involved in several European and French projects dedicated to the development of innovative hydrometallurgical processes for the extraction or recycling of critical materials, including process development (experimental and simulation) and Life Cycle Assessment.

**ICSM** is a Joint Research Unit between CEA, CNRS, University of Montpellier and the Ecole Nationale Supérieure de Chimie de Montpellier. ICSM develops an integrated approach combining fundamental research with exploratory R&D in chemistry. ICSM provides scientific expertises and technological platforms for analytical chemistry, radiochemistry, sonochemistry, physical chemistry of multiphasic fluids, nanomaterials for recycling in energy and environment sectors.

**LGC** is a leading research laboratory in the field of chemical and process engineering. It develops theoretical research and advanced technologies at the core of matter and energy transformation processes. LGC is supported by 3 supervisory bodies, the CNRS, the Institut National Polytechnique de Toulouse and the Université Paul Sabatier de Toulouse. It addresses major societal challenges, including water and industrial effluents, energy production, solids processing and health engineering.

**MINES Paris - PSL**, part of the University of Paris, Sciences and Letters, is the first engineering school in France by its volume of partnership research. The research activities of the Geosciences Center aim to i) improve knowledge of the primary resources of the subsoil and advance the techniques for their exploitation, ii) promote the use of the subsoil and ii) predict the effect of climate change and anthropogenic modifications on water resources and natural risks.

**Laboratory LGF** brings together all the potential of the **Mines of Saint-Étienne** in the fields of materials, mechanics, and processes. Its expertise in the field of chemical engineering is applied to dispersed systems: grains, particles, drops, bubbles, porous media. Its research activities focus on the characterization of granular materials and unit operations in the granular phase.

**University of Pau** is training 14,000 students per year in law, economics, management, human sciences, sports, and sciences of technology. The UPPA has received the IDEX or I-SITE excellence label (‘Initiatives for Science, Innovation, Territories and Economy’) and is a member of the European alliance of universities UNITA-Universitas Montium.

Besides their expertise, the partners of InnovTech own experimental facilities and characterisation laboratories that are crucial for the execution of works. They are presented in Table 3.1.

**Table 3.1 : Main experimental facilities of the InnovTech partners**

| | |
|---|---|
| **BRGM** | The platform for innovation PLAT’INN (1 000 $m^2$) is dedicated to conduct test work from lab to pilot scale, with samples from “g” to “tonne”. It includes more than 200 devices in mineral processing and in (bio)-hydrometallurgy. Various kind of equipment are also available for chemical and mineralogical characterization. BRGM holds the MIMAROC platform which is based on the innovative coupling between experimental cells and a µ-X-ray tomograph allowing to carry out thermal, hydraulic, chemical and mechanical tests and to visualize the processes in-situ or in-operando. BRGM has also access to the SOLSA corescanner that combines non-destructive and fast profilometric, RGB, XRF and hyperspectral measurements to provide detailed morphological, geochemical and mineralogical information on samples. |
| **UL** | The STEVAL platform (2 000 $m^2$) for mineral processing is an academic pilot plant unique in Europe. It gathers all equipment for mineral processing, including crushing, milling, classification, and all types of separations. STEVAL is dedicated to the flowsheet development and the scale transfer in mineral processing, for teaching and research, and can operate from lab to pilot scale at up to 200 kg/h of continuous operations. UL also has the HydroLab laboratory and the HydroVal platform, which are dedicated to hydrometallurgy experiments at lab scale and pilot scale. |
| **LGC** | The LGC operates a large array of instrumented laboratory and pilot scale processing equipment that supports all the laboratory's research programs and scientists. The LGC also hosts a shared analytical platform with several high-performance analytical measurement techniques. |
| **MINES Paris** | The Geosciences Centre at MINES Paris is equipped with a platform dedicated to physical characterisation of geomaterials and analysis of their mechanical, hydraulic, and thermal |

| | |
|---|---|
| | behaviours. It provides both experimental and numerical resources to assess and monitor the behaviour of geomaterials under real conditions. |
| **UPPA** | The experimental platform of the thematic “Thermodynamic of the multi-phasic, electrolytic and reactive systems and associated processes” gathers a dozen of triphasic/ biphasic batch reactors which are instrumented to follow-up the pressure, temperature and composition phases over time. |
| **ICSM** | Numerous equipment is available for the analyses of solids, surfaces, and liquids (SEM, XRD, ICP-OES, ICP-MS, SWAXS, DLS, XRF, Raman spectroscopy, NMR, etc..). |
| **LGF/ MSE** | Several instrument platforms are available in support of research activities. In particular, they include equipment for thermal analysis (such as TGA, TGA/DSC, TGA/SM and TGA/AE) and for textural characterization of powder (e.g. X-ray tomography, BET specific surface area, etc.). |
| **CEA** | The Chimene Platform is dedicated to the study of hydrometallurgical processes for metal extraction and refining, including dissolution and separation micropilots and equipment for solution or solid characterization. |

## 3.3. Management framework

### 3.3.1 Management, organization between partners and strategy for the management of Intellectual Property (IP)

Since the management of InnovTech and the organization between partners are crucial for the success of InnovTech, they have been included in a dedicated WP: WP1. All issues related to IP will be managed in Task 1.2. In particular, the use of knowledge generated in the project and IP is governed by the Consortium Agreement (CA) which will include key background for the project. Foreground results will be owned by the project partner carrying out the work generating the results. If any results are created jointly by at least two partners and it is not possible to distinguish between the contribution of each, such work will be jointly owned by the contributing project partners. Details concerning jointly owned results, joint inventions and joint patent applications will be addressed in the CA.

### 3.3.2 Critical risks for implementation

The table below summarises the critical risks identified, their degree of likelihood (L) (low=1, medium=2, high=3), their impact (I) (low=1, medium=2, high=3) and the countermeasures planned to address them. This list will be continuously updated during the lifetime of the project.

| Risk | L/I | WP | Mitigation (M) or Contingency (C) Plan |
|---|---|---|---|
| Key partner leaves the consortium | 1/2 | All | (C): Tasks assigned to the leaving participant are re-allocated among the consortium or new participant could be invited. |
| Difficulty to recruit PhD students and postdoctoral researchers | 1/3 | All | (M): The project team will use its significant experience in this type of recruitment and its large network of academic partners to hire high-level young researchers. |
| Delayed arrival of samples | 1/2 | WP2, 3 & 4 | (M): Samples with geochemical similarities are already available at the partners facilities and will be used to begin the works. |
| Difficult to integrate models and/or to couple the various softwares. | 2/2 | WP5 | (C): Indirect methods using intermediate types of files (such as Excel files) will be used. |
| Conflicts in ownerships and user rights of results | 1/3 | All WPs | (M): IPR defined in the Consortium Agreement. Timely identification of new results and agreement of ownership and user rights between contributing partners. |

## 3.4. Institutional strategy

The institutional strategy of BRGM, which leads InnovTech, is described in detail in the description of the project Governance. In particular, InnovTech is fully in line with the scientific strategy of BRGM aiming to contribute to the major challenges of the society such as the climate change and the energy and digital transition requiring a more sustainable and responsible production of raw materials including in particular the development of flexible processes for the valorisation of complex resources. This strategy is shared by the other partners of InnovTech, as indicated in the description of their key activities and annual and activity reports[25]. Indeed, all the partners defined a strategy that is based on a multiscale approach aiming at understanding the fundamental phenomena to develop disruptive and breakthrough technologies, in addition to increase the use of the new digital tools (such as artificial intelligence) in the extraction sector. Moreover, most partners involved in InnovTech defined a pluriannual strategy that includes the participation in the subsurface uses, to increase the independency of France and European Union in terms of primary raw materials. In particular, InnovTech fits totally with the objectives and performance contract (OPC) of the CNRS in which the cycle of resources for sustainable societies is addressed in the axis 2.4. Another example is the CEA strategy since this one dedicated to make CEA "Proactive in its commitment to upholding the scientific, technological and industrial sovereignty of France and Europe to safely secure the present and the future."

Besides, all the partners of InnovTech provide important infrastructure, material (equipment, platforms, labs…), and human (salary of permanent positions) resources to ensure the success of this PC. This involvement of the structures will cover the whole duration of InnovTech and PEPR SousSol, which will support the scientific success of the project.

# 4. Expected outcomes of the project

## 4.1. Main outcomes of the project

The main outcomes of the project will consist of (1) high-level scientific results that will, most often, combine theoretical/numerical and experimental approaches, (2) breakthrough innovations including not only technologies but also experimental tools and devices, and (3) database of numerical models that can be disseminated in open-access and used by the international community. These outcomes will contribute to the current and planned action plans related to subsurface uses and in particular to its exploitation. Moreover, indirect outcomes such as a significant increase in the synergy between the InnovTech partners working in various fields is expected.

## 4.2. Valorisation of the outcomes

Communication and Dissemination plays a major role to maximize the valorisation of the outcomes of InnovTech at the French and at the international levels. In particular, scientific events such as workshops and scientific days will be regularly organised during the project lifetime, one year at the French level and the other year at the international level. Dedicated communication actions around the various scientific events of this PC will be undertaken in order to promote the scientific outcomes

[25] Some example are given here : https://www.cnrs.fr/sites/default/files/news/2020-01/COP.pdf for CNRS, https://www.cea.fr/multimedia/Lists/StaticFiles/rapports/annuel/fr/index.html for CEA; https://www.minesparis.psl.eu/Ecole/Rapport-d-activite/ for Mines Paris.

of InnovTech. Moreover, a common strategy for publications will be set up and will favour the publications in open access peer-reviewed international journals. This InnovTech strategy for communication, dissemination, and promotion of the results will be integrated in the dedicated strategy defined in the Action 6 ‘Communication and outreach’ of the PEPR SousSol Governance. In particular, it will rely on the PEPR SousSol website and social networks, on its open science guidelines and the organisation of dedicated events and workshops.

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