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Graphene, MXene and ionic liquid-based sustainable supercapacitor

GREENCAP - Deliverable report

D4.1. – Upscaling processes and standardization

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Project summary

GREENCAP focuses on developing high-performance, sustainable cylindrical supercapacitors (SCs) that exhibit battery-like energy density, high power densities, and long cycle life, by utilising graphene and MXenes as electrode materials and ionic liquids (ILs) as high-voltage electrolytes. The use of 2D layered materials and ILs will enhance the specific surface area, ion accessibility, and charge storage of the electrodes, while ensuring stability and safety across a wide temperature range.

The consortium consists of academic and industrial partners from seven European partners and Ukraine. GREENCAP addresses the energy storage sector, while also meeting the EU's climate-neutrality goals and the Action Plan on Critical Raw Materials. GREENCAP will validate this SC technology at an industrial scale (TRL 6) and develop a management system to optimize SC integration into high-end applications and the circular economy.

Publishable summary

This deliverable reports the consortium efforts for the upscaling and quality standardization of the supercapacitor (SC) components, from the electrode materials (EMs) and their slurry formulation to the electrolytes based on ionic liquids (ILs).

Specifically, Bedimensional (BeD) few-layer graphene (FLG) flakes production has been upscaled to 3.5 t/year, reaching the target of a production > 10^3 kg/year. Moreover, several other actions have been implemented and/or started. In particular, the quality of the FLG has been assessed by developing a quality control (QC) procedure accordingly to the graphene-dedicated ISO standard (ISO/TS 21356-1) and the procedure for obtaining the REACH registration has been initiated. In parallel, UCAM up-scaled the production of FLG flakes by high pressure homogenization (HPH), reaching a production rate of 10^3 kg/year, while TUD developed an up-scaling systems for the electrochemically exfoliated graphene (EG) reaching an yield of 20 g/h. Regarding the active material selected within the D3.1, Skeleton Materials GmBH (SM) upgraded their production of Curved Graphene (CG), proprietary name of the specific typology of carbide derived carbon developed by Skeleton (SKL), reaching the project target of 10^5 kg/year. In parallel, Carbon Ukraine (CU), with the support of the Trinity College Dublin (TCD), designed the etching system for the upscaled production of MXenes. Solvionic (SOLV) developed the production and quality control protocol of the IL-based electrolytes selected for the prototypes developed in D4.3.

Finally, a QC procedure and upscaling of the electrode slurries has been defined by BeD with the support of SKL and TUD.

Contents

List of Figures

List of Tables

Abbreviations & Definitions

Abbreviation	Explanation
2D	Two dimensional
AFM	Atomic Force Microscopy
BeD	BeDimensional SpA
BET	Brunauer-Emmett-Teller

CG	Curved Graphene
CMC	Carboxyl Methyl Cellulose
CU	Carbon Ukraine
DI	Deionized
DoA	Description of Actions
DoW	Description of Work
ECHA	European Chemicals Agency
FLG	Few layer graphene
FTIR	Fourier-transform infrared
FWHM	Full Width at Half Maximum
HPH	High Pressure Homogenized
IC	Ion Chronomatography
ICP-MS	Inductively Coupled Plasma-Mass Spectroscopy
ICP-OES	Inductively Coupled Plasma-Optical Emission Spectroscopy
IL	Ionic Liquids
KF	Karl Fischer
LoA	Letter of Access
LPE	Liquid Phase Exfoliation
MILD	Minimally Intensive Layer Delamination
MSR	Molten Salt Reactor
QC	Quality Control
SBR	Styrene-Butadiene Rubber
SC	Supercapacitor
SEM	Scanning Electron Microscopy
SIP	Substance Identity Profile
SKL	Skeleton Technologies
SLG	Single Layer Graphene
SOLV	Solvionic
TCD	Trinity college Dublin
TEM	Transmission Electron Microscopy
TGA	Thermogravimetric analysis

TUD	Technische Universitat Dresden
UNIGE	University of Genova
WJM	Wet Jet Milling
XPS	X-ray Photoelectron Spectroscopy
XRD	X-Ray diffraction
XRF	X-Ray fluorescence

1 Introduction

The present document describes deliverable D4.1 on the upscaling and quality standardization of the SC components, including EMs, slurry and ILs-based electrolytes, also focusing on the development of a manufacturing chain line capable to sustain the up-scaled production of advanced SCs. BeD inaugurated its new production plant having a third-party certified production rate of 3.3 t/year, as reported in milestone 8 (Ms8). The certified production rate is higher than the project target, i.e., 10^3 kg/year. BeD has also initiated the procedure for obtaining the REACH certification. In parallel, UCAM implemented a production set-up of HPH FLG reaching a production rate $>10^3$ kg/year, while TUD developed a 20 g/h scale-up system to produce EG. SM upgraded its facility reaching a production capability of CG $>10^5$ kg/year. SOLV adapted its systems to produce electrolytes selected within WP1 and WP2. CU developed a low-fluorine content etching system to produce MXenes reaching a rate of 1kg/day. Moreover, with the support of TCD and TUD, CU designed and is currently developing a molten salt-based etching system for the scale-up production of MXenes, avoiding the use of HF, which raises sustainability and safety concerns.
This work is carried out within WP4, which also aims at the developing of cylindrical cell prototypes together with an established manufacturing chain line capable to sustain their scale-up production.

The WP4 is divided in four main tasks and their respective updates:

- Task 4.1: Upscaling processes and standardization
  The present document
- Task 4.2: Manufacturing chain line
  Preliminary report describing the requirements and necessary actions needed to establish the industrial chain manufacturing of the SCs.
- Task 4.3: Prototyping: cylindrical cell manufacturing, testing and assessment
  Build a demonstrator cylindrical cell, based on industrially available configurations provided by SKL.
- Task 4.4: Preliminary Supercapacitor Management System
- Task 4.5: Updated Upscaling processes and standardization
- Task 4.6: Updated Industrial chain manufacturing
- Task 4.7: Updated Cylindrical prototype cells
- Task 4.8: Supercapacitor Management System

Key achievements:

- BeD scaled-up the production of FLG ($>10^3$ kg/year) and initiation of the REACH certification.
- UCAM scaled-up the system for the production of HPH-FLG ($>10^3$ kg/year)
- SM scaled-up the production of CG ($> 10^5$ kg/year).
- CU scaled-up the production of low-fluorine content MXenes (1 kg/day).
- SOLV developed and scaled-up the production of novel IL-based electrolytes.
- Definition of a QC protocol for the electrode slurry.

- CU with TCD and TUD designed a scale-up system for MXene production using etching methods based on molten salts (HF-free approach).
- SKL scaled-up the slurry optimized within WP3 and based on CG and Na-CMC.

Minor deviations:

- No minor deviation has been encountered

2 Results and Discussion

2.1 Graphene

2.1.1 Wet Jet Milling few-layer graphene

Scale up production of FLG

The production of single- and few-layers graphene (FLG) flakes in BeD is based on the Wet Jet Milling (WJM) technology. The method for the scale up of 2D flake production is already patented by BeD, under patents IT 102015000077259; EP 3380435; US 10407308; JP 6791965; KR 101969118.

Currently, BeD has two established production plants. The pilot production line for the production of FLG flakes with a production rate of 0.2 tonnes per year, in operation since May 2019, and the industrial production plant with third-party certified capacity of 3.3 tons per year. The industrial production plant was completed and certified in July 2024 and inaugurated in October 2024, and it is now in full operation.

Quality Control

The quality and the production reproducibility of the FLG flakes are ensured by quality control (QC) procedures that were established following the ISO TS 21356-1 guidelines. To this end, small quantities (tens of milliliters), as representative of the entire production batch, are received by the QC team of BeD, characterizing their morphological, structural and physico-chemical properties. The QC procedures are described in detail in **D1.1**. In Figure 1 we reproduce the QC flow chart, presented initially in D1.1, for the readers convenience.

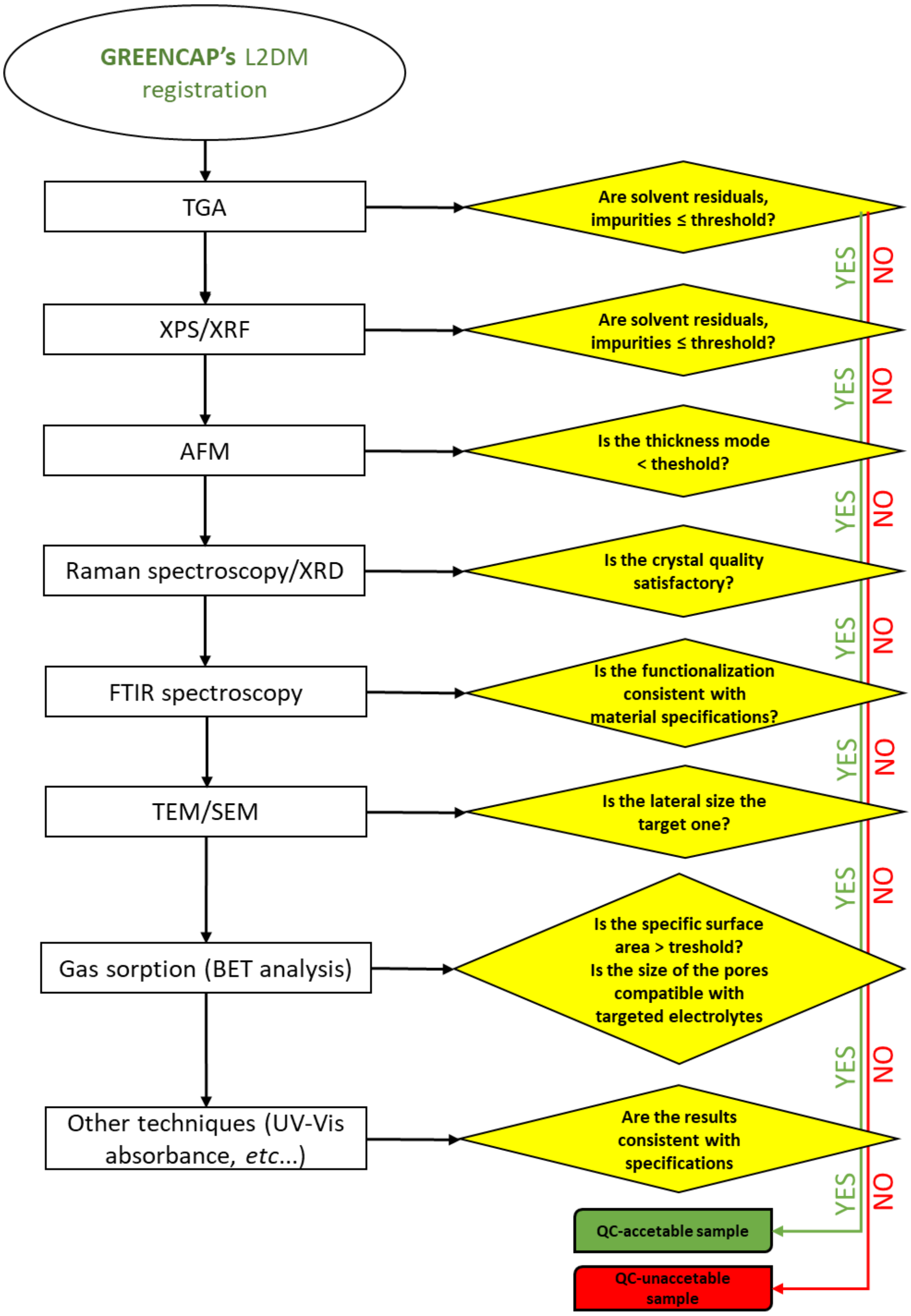


Figure 1: Representative QC flowchart diagram used to ensure the quality and reproducibility of L2DMs amongst different production batches. (Figure 2-4 of the Deliverable 1.1)

Registration ECHA of BeD FLG

The European Chemicals Agency (ECHA) regulation covers the manufacture, import, placing on the market and use of all chemicals on their own, as well as in mixtures and articles, and establishes the principle that it is the responsibility of the company to manage the risks of chemicals and to provide information on the safety of the substances it produces, uses or places on the market.

Producers of chemicals are, therefore, obliged to collect information on the properties of the substances they produce, so that they can be safely managed, and to pass this information on to the ECHA. Otherwise, they are not allowed to manufacture, import or place them on the market.

Registration is mandatory for manufacturers and importers of substances for quantities equal or higher than 1 t/year, regardless of hazard. In such cases they must submit to ECHA the available information on chemical and physical characteristics of the substances. In the case of absence of available data they have the obligation to perform experimental tests in order to characterise the physico-chemical, toxicological and eco-toxicological properties of the material to be registered.

BeD, with the scaling up production of graphene powder foreseen for the year 2025, nominal capacity of 3.5 tonnes of FLG powder, has therefore initiated the REACH registration process (product name: G-LEAF 09900).

The REACH registration of the graphene is possible as a Joint Submission because graphene is already registered in the tonnage band of current interest for BeD *i.e.*, 1-10 t/year (registration number: EC 801-282-5, CAS 1034343-98-0).

Joint submission is possible due to the existence of a consortium of companies that need its REACH registration as producers and they have thus agreed to share technical information concerning the produced substance, but also to share all the administrative costs involved in submitting the documents and inquiries to ECHA. The first company to apply for the REACH registration of a substance, *i.e.* the Lead Registrant, acts as the reference of the consortium and is the one who approves the terms of access of new companies.

In order to enter the consortium, several steps must be followed: (i) to submit a letter of access (LoA), which is the entry fee to the consortium, (ii) to pay the registration fees to ECHA, depending on the company size and tonnage band and (iii) to demonstrate that the substance produced has chemical/physical characteristics consistent with the ones already registered under the same number.The accurate identification of the substance is important for the registration process, so that it ensures that registrants of the same substance belong to the same joint registration, hence facilitating the development of the Substance Identity Profile (SIP) and the reporting of the boundary composition.

Then Inquiry Dossier Submission needs to be confirmed by ECHA in order to obtain the Member Dossier Submission.

BeD is proceeding with a preliminary physico-chemical analysis of its FLG and the compilation of the SIP in a partnership with a third-party, i.e., the University of Genova (UNIGE) (attached quotation – Appendix B).

More specifically, according to the requirements of the Nano Regulation (EU) 2018/1881, each registrant must take into account, among others, the following parameters:

1. Particle size distribution and number fraction of constituent particles.
2. Shape, aspect ratio and other morphological parameters.

3. Functionalisation or surface treatment and identification of each agent, including IUPAC name and CAS or EC number (if applicable).
4. Surface area (specific surface area by volume, specific surface area by mass, or both).

Finally, the values for carbon and oxygen content are required, as they are crucial for the substance definition and (eco)toxicological properties of graphene materials.

Besides its own FLG REACH registration, BED is collecting and sharing with the Greencap partners the information about the registration requirements of FLG and other 2D materials.

2.1.2 Electrochemically exfoliated graphene

TUD focused on the design and development of a scaling-up system for the production of electrochemically exfoliated graphene (EG).

The electrochemical exfoliation of synthetic graphite foils yields high-quality EG with excellent efficiency (see **WP1 results**). More importantly, the EG sheets can be **in situ** functionalized with various chemical groups, including conductive polymers such as polyaniline and polypyrrole, by adding the corresponding monomers to the aqueous electrolyte during the electrochemical exfoliation process (see **WP2**).

At TUD, laboratory tests are carried out using standard setups in a low-volume batch process (**Figure 2a**). However, scaling up the batch process has resulted in reduced graphene quality and yield. These issues are primarily due to challenges in controlling temperature and the presence of free hydroxyl radicals, which can oxidize the EG sheets.

To address these limitations and meet the requirements for pilot-line and large-scale EG production, TUD developed a continuous flow reactor (**Figure 2b**). This reactor allows for the continuous flow of electrolyte and immediate removal of the produced (functionalized) EG, thereby minimizing its exposure to oxidative damage by hydroxyl radicals.

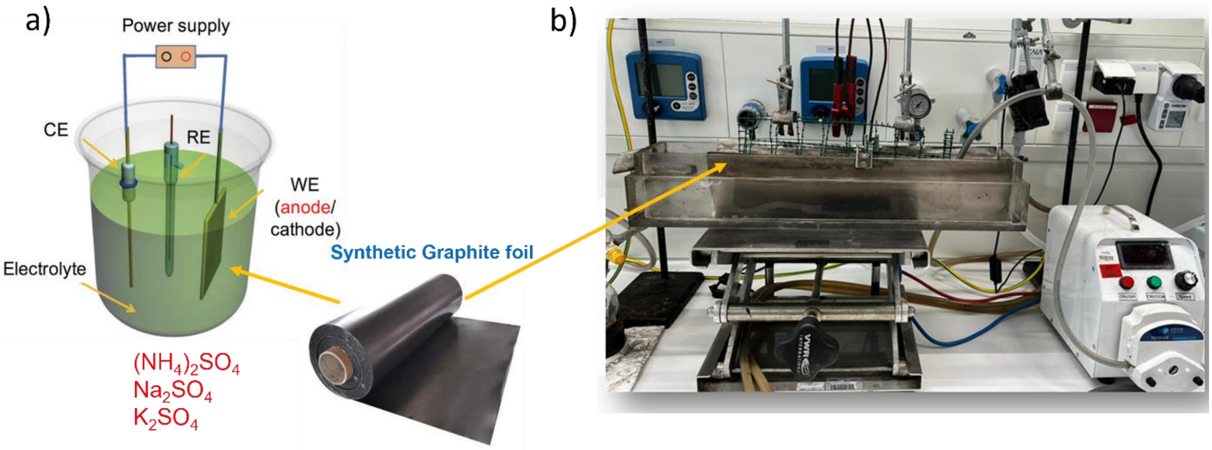


Figure 2: a) Electrochemical exfoliation process at laboratory scale; b) Developed continuous flow reactor for mass production of electrochemically exfoliated graphene.

To optimize reactor geometry for EG production, we designed a research-scale flow reactor with movable electrode slots (**Figure 3**). This configuration allows adjustment of both the distance and height between electrodes, enabling detailed studies on the influence of geometry. In addition, we optimized other key parameters—such as electrolyte concentration, functional group concentration, and temperature—based on results obtained from WP1 and WP2 to improve production in the continuous flow setup.

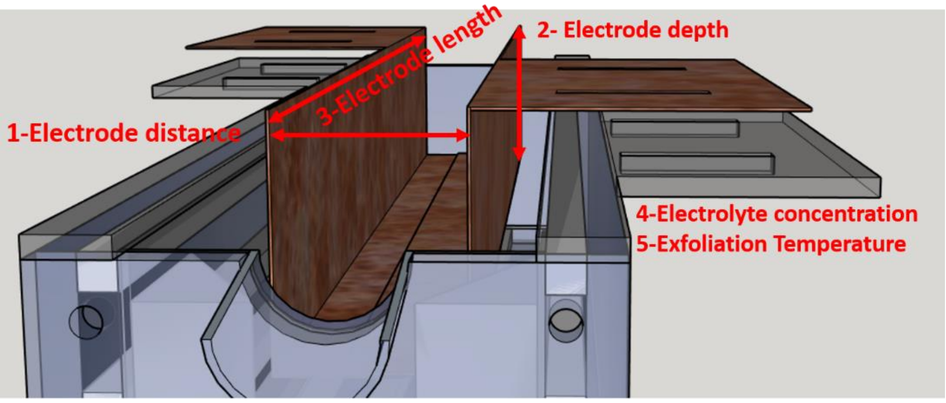


Figure 3: The constructed flow reactor with movable electrode slots.

By fine-tuning all relevant parameters, we achieved a EG production rate of **30 g/h** while maintaining the product quality established in WP1 and WP2. For instance, the functionalized graphene (e.g., polyaniline-functionalized) produced in the continuous flow reactor demonstrates electrochemical properties comparable to those obtained in small-scale experiments (WP2), confirming the reactor suitability for high-quality, large-scale EG production (**Figure 4**).

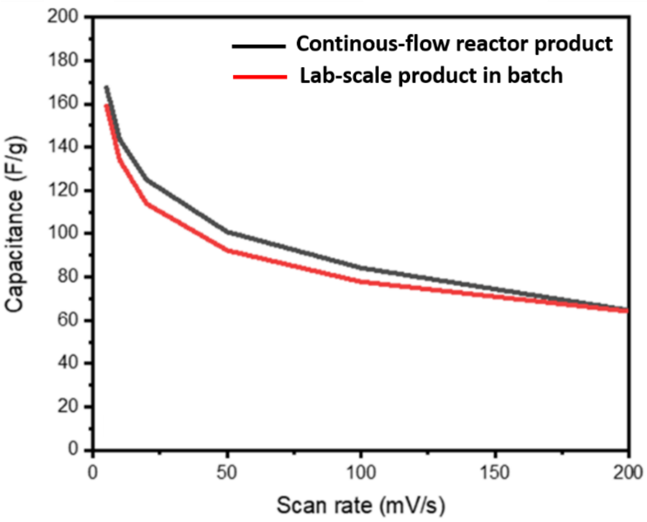


Figure 4: Comparison of the electrochemical performance of functionalized graphene produced in the laboratory and in the flow reactor.

2.1.3 High-pressure-homogenization few-layer graphene

UCAM used liquid phase exfoliation (LPE) of graphite via high-pressure homogenization (HPH) as a scalable method to produce FLG dispersions. UCAM performed a foundation study of a typical setup used by UCAM (PSI-40 HPH with the D202D interaction chamber). The graphite precursor (with a concentration of 1 - 300 g/L), in the form of synthetic graphite or expanded graphite powder, is suspended in a suitable solvent—isopropanol (IPA), or a surfactant-stabilized medium like ethyl cellulose at a concentration optimized to balance viscosity, exfoliation efficiency, and reaggregation risks. PSI-40 HPH setup then forces this suspension through a narrow orifice or interaction chamber (87 - 100 µm diameter) under pressures typically ranging from 1800 to 2000 bar. When running at maximum pressure drop across the micropore, the volumetric flow rate through the micropore is $Q = 26\ \mathrm{L\ h^{-1}} = 7.22 \cdot 10^{-6}\ \mathrm{m^3\ s^{-1}}$. This creates shear rates $> 10^7\ \mathrm{s^{-1}}$, microturbulence, and intense pressure drops that generate cavitation turbulence, which collapse to produce shockwaves. These forces act synergistically to cleave graphite. From a mechanistic standpoint, the advantage of HPH lies in the ability to expose graphite particles to rapid, repeated mechanical stresses without the significant thermal gradients seen in other LPE methods, such as ultrasonication, which suits for long term operation and scale up. The laminar-to-turbulent transition within the interaction chamber ensures that exfoliation occurs across a distributed population of flakes rather than localized zones, contributing to uniform delamination. The closed system design of HPH also allows continuous processing and inline monitoring, offering industrial scalability. To achieve the requested scale up KPI, UCAM installed two HPH equipment and, switched the setup from 1 to 5 L process reservoir and from single to dual slot deagglomeration chamber.

Table 1: UCAM upscaling capabilities of graphene slurry production > 1000 kg/year SLG/FLG.

Demonstrated Upscaling of graphene-ink production			
Two HPH at CGC	Processing capabilities - 50 L/day (~8 h operation time) with graphite content (100 g/L)	Graphene slurry production capabilities - 5.0 Kg/day	>1000 kg/ year Graphene slurry (~ 1250 kg based on 250 working days/year)

UCAM scaled-up the production of graphene-ink and is capable of producing >1000 kg/year of FLG while maintaining the quality of the FLG-based slurries, Figures 5a – f, which meets the description of work (DoW) KPI. A breakdown of the daily processing capabilities leading to > 1000 kg/year FLG is listed in Table 1. An example of reproducibility of FLG-based slurry from 3 batches S1, S2, S3 is also shown. In a Raman characterization study, different batches have negligible changes in D peak position, I(D)/I(G) and Full Width at Half Maximum (FWHM). The viscosity of different batches is also similar, confirming the reproducibility at large scale.

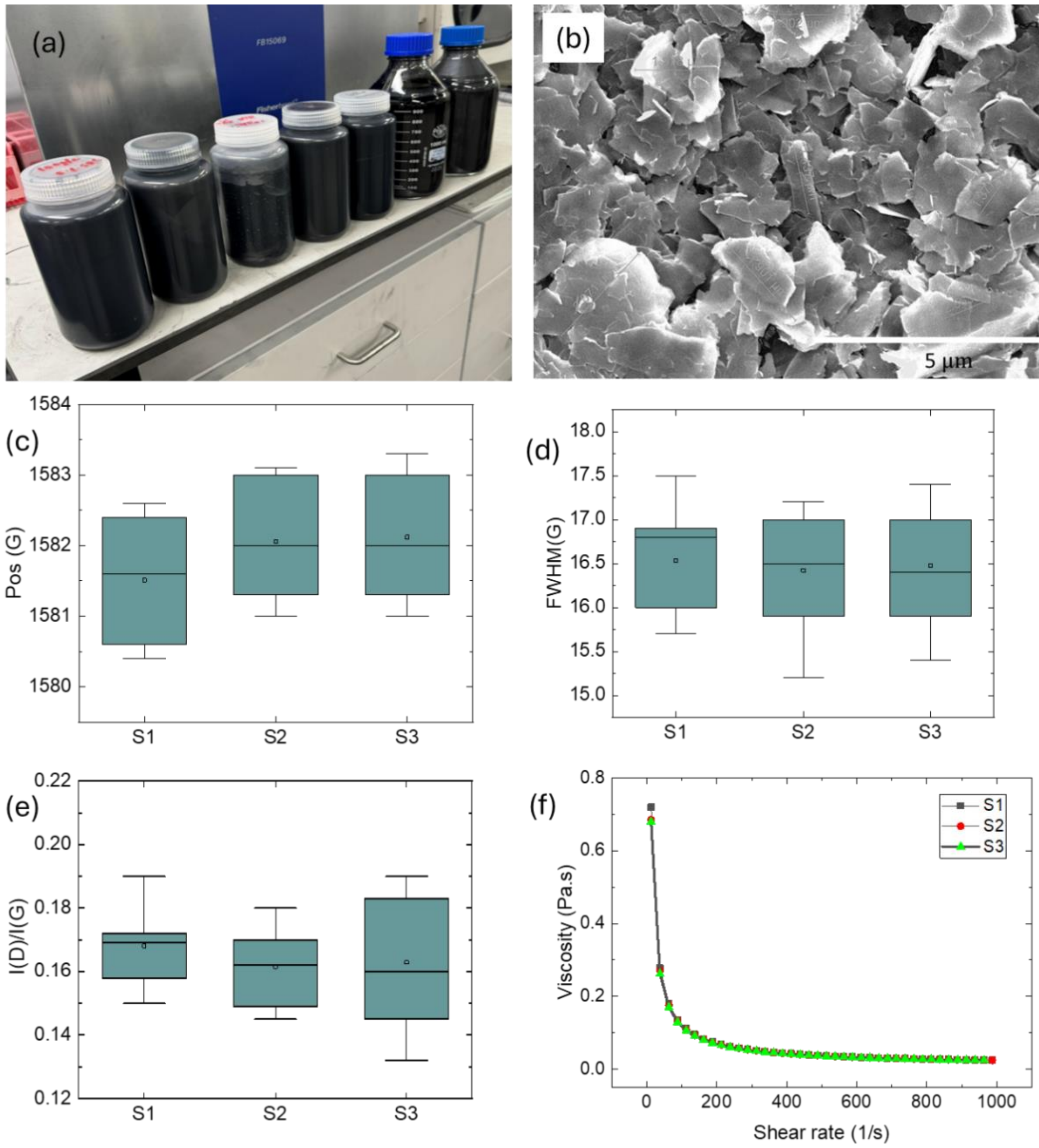


Figure 5: (a) Graphene slurries (in IPA) produced by UCAM via 100 cycles HPH; (b) SEM of graphene flakes (c, d, e) Raman characteristics of graphene-ink, (f) viscosity of graphene-ink for 3 batches.

2.2 MXenes

2.2.1 Molten Salt approach

CU, with the TCD and TUD inputs, is working on the development of a molten salt reactor concept and design for MAX-phase etching to synthesize MXenes via this approach using a scalable designated reactor. The reaction mechanism in molten salt reactor is defined as following: once melted, molten salts act as solvents, dissolving solid reactants and solvating ions through strong polarization. They also efficiently transport reactant species via convection and diffusion. Molten salt-assisted routes significantly expand the range of the MAX phase precursors available for MXene synthesis, offering extensive opportunities to tailor electronic conductivity, hydrophilicity, and surface chemistry of MXene materials. This etching method operates as a continuous process, eliminating the need for manual intervention or exposure to hazardous chemicals. Additionally, the mild reaction conditions in molten salts enable environmentally friendly and sustainable chemistry, paving the way for scalable and commercially viable MXene fabrication.

Working Principle

A molten salt reactor for MXene synthesis operates by utilizing molten salts as both the reaction medium and the etching agent (see Figure 6). Transition metal halide salts (e.g. NaCl, KCl, $CuCl_2$) serve as Lewis acids to etch MAX phase precursors under high-temperature conditions (700 °C). The molten salts act as solvents, dissolving reactants and facilitating the transport of ions through convection and diffusion.

Homogeneously mixed powders of MAX-phase, $CuCl_2$, KCl, and NaCl salts in appropriate atomic ratios are placed into a crucible serving as reaction chamber. Then, under continuously flowing argon and heating to 700 °C at 5 °C/min, hold for 5 h for the chemical reaction to take place, following by cooling to room temperature. The etching is carried out at 700 °C, with the molten salt providing a shield to avoid oxygen attack to the MXene sheets that are being formed at this high temperature. After the etching, the Cu metal formed on the MXene can be removed by treatment with $CuCl_2$ in HCl or with acetonitrile. According to our calculations, in order to achieve a production rate of approximately 1 kg/day, we need to start from a reactor having a volume of 3-5 L.

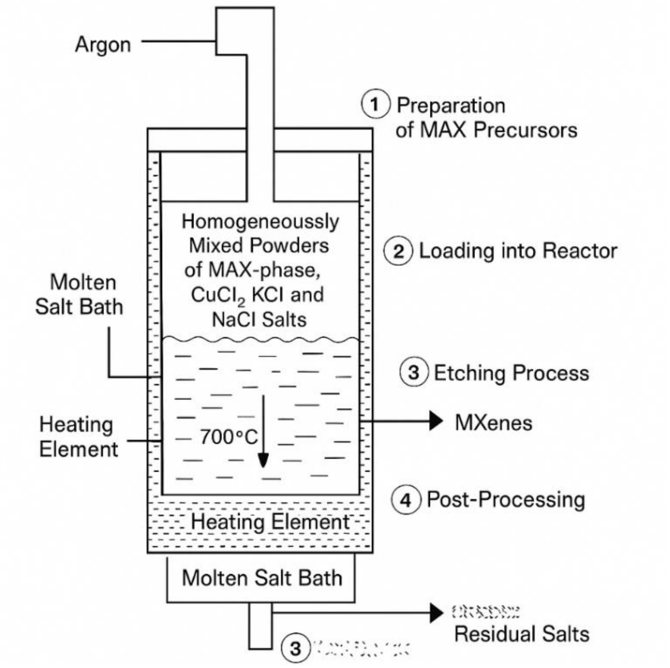


Figure 6:Molten-Salt Etching Process.

Process Flow in the proposed reactor concept

1. **Preparation of MAX Precursors:**
   MAX phases are prepared and ground into fine powders for uniform etching. Starting MAX phase weight: 1.2-1.5 kg, to account for material loss during the etching process.

2. **Loading into Reactor:**

The precursors are mixed with the molten salts (e.g. NaCl/KCl) and optional additives, like $CuCl_2$, to facilitate the etching. Typically, the molten salt mixture ratios are 70:30 or 80:20 of NaCl:KCl, but also other mixture ratios are possible, depending on the desired etching efficiency. Salt Mixture: Weigh and mix approximately 3-4 kg of a molten salt mixture, typically NaCl/KCl in a 70:30 or 80:20 ratio, with an optional $CuCl_2$ additive (5–10 wt%) to act as a catalyst for the etching reaction.

3. **Etching Process:**
   The reactor is heated to the required temperature (i.e. 700°C). Molten salts dissolve and transport reactant species to enable the etching reaction. Surface termination groups are introduced or modified based on the molten salt composition. The reactor is heated gradually at a rate of 5°C per minute until it reaches the target temperature of 700°C. This heating process should take approximately 2-3 h. Maintaining Reaction Conditions: Once the target temperature of 700°C is achieved, the temperature is maintained for approximately 5 h to allow the molten salts to react with the MAX-phase material, selectively etching out the A-element from the MAX-phase to form MXene sheets.

4. **Post-Processing**:
   After etching, the reactor is cooled, and the MXenes are separated from the molten salt. Residual salts are removed using appropriate techniques (e.g., washing with deionized - DI- water or solvents).

Reactor design considerations:

Material Compatibility: The reactor materials must resist corrosion caused by molten salts.

Energy Requirements: While the process is energy-efficient, maintaining high temperatures requires careful energy management.

Reactor Volume:

The required reactor volume for molten salt etching to produce 1 kg of exfoliated MXenes is approximately 1.8 to 2.4 L. However, for practical purposes, the reactor size should be scaled up slightly to accommodate material handling, reaction efficiency, and any additional space for gas flow or mixing, so a reactor with a 3 to 5 L capacity would be recommended for this activity.

Material & Design Criteria:

- Construction materials: Inconel, alumina, or coated stainless steel.
- Thermal insulation.
- Gas-tight design.
- Crucible-based loading.
- Internal mixing for uniformity.

Key Features of Molten Salt Reactors for MXene Synthesis:

- Uniform Heat Distribution, which is critical for homogeneous reactions and consistent product quality;
- Controlled surface termination enabling the introduction of halide groups (e.g., Cl, Br, I) in specific ratios, tailoring MXene properties, like conductivity and hydrophilicity;
- The reactor design minimizes oxygen exposure, preventing unwanted oxidation and ensuring the purity of MXenes.

Advantages of Molten Salt Reactors:

- Eco-Friendly Process: The method eliminates the need for hazardous chemicals, like HF, reducing environmental and safety concerns;
- Scalability: molten salt reactors are scalable, supporting the industrial production of MXenes;
- Versatility: Applicable to a wide range of MAX phase precursors and capable of synthesizing MXenes with tailored properties.

Benefits of Scaling Up.

Molten salt reactors represent a cutting-edge solution for the sustainable and scalable synthesis of MXenes. By leveraging the unique properties of molten salts, these reactors address the limitations of traditional methods, paving the way for the widespread adoption of MXenes in various technological domains. The proposed reactor concept can be further used as a model for development of large-scale reactors, as it can meet the industrial-scale demand for MXenes in energy storage, catalysis, and electronics due to cost reduction (energy use and material handling reduce per-unit production costs) and sustainability. Improved salt recycling and reduced chemical waste contribute to a more sustainable process. Scaling up MXene synthesis in molten salt reactors is a feasible and impactful step toward industrial production. By addressing challenges through innovative reactor design, process optimization, and sustainability measures, molten salt reactors (MSRs) can support the growing demand for MXenes in advanced materials applications.

2.2.2 Low-Fluoride (<5%) Etching Alternative:

Several etching routes has been developed and successfully applied to achieve low-fluoride (< 5 % total F^-) MXene synthesis conditions (see Figure 7).

Etchant: Mixture of dilute HCl with minimal LiF/NH_4F (<5% total F^-).

Conditions: Room temperature to 60°C for 12–24 h.

Post-Treatment: Neutralization, exfoliation, delamination.

The proposed etchant route can be applied to different MXenes (some synthesis conditions need to be adjusted depending on the MXene type). Here, we demonstrate etching routes for the model Ti3C2 MXene (both forms - delaminated and multilayer).

Delaminated MXene

Etching approach: Minimally Intensive Layer Delamination (MILD) method, HCl with minimal LiF (<5% total F^-).

$$2Ti_3AlC_2 + 12LiF + 6HCl = 2Ti_3C_2 + 6LiCl + 3H_2 \ (4.8\% \ total \ F^-)$$

Conditions: Synthesis temperature 35°C for 24 h

Post-Treatment: Neutralization, exfoliation, delamination.

Multilayered MXene

Etchant 1: Mixture of diluted H_2SO_4 (20-30%) with LiF (4.8-4.5% total F^-).

$$2Ti_3AlC_2 + 12LiF + 3H_2SO_4 = 2Ti_3C_2 + 2Li_3AlF_6 + 3Li_2SO_4 + 3H_2$$

Conditions: Synthesis temperature 35°C for 24 h

Post-Treatment: Neutralization

Etchant 2: Mixture of diluted $H2SO_4$ (20-30%) with NaF (3-2.8 % total F^-).

$$2Ti_3AlC_2 + 12NaF + 3H_2SO_4 = 2Ti_3C_2 + 2Na_3AlF_6 + 3Na_2SO_4 + 3H_2$$

Conditions: Synthesis temperature 35°C for 24 h

Post-Treatment: Neutralization

Etchant 3: Mixture of diluted H_2SO_4 (20-30%) with NH_4HF_2 (4.7-4.5 % total F^-).

$$2Ti_3AlC_2 + 6NH_4HF_2 = 2Ti_3C_2 + 2(NH_4)_3AlF_6 + 3H_2$$

Conditions: Synthesis temperature 35°C for 24 h

Post-Treatment: Neutralization

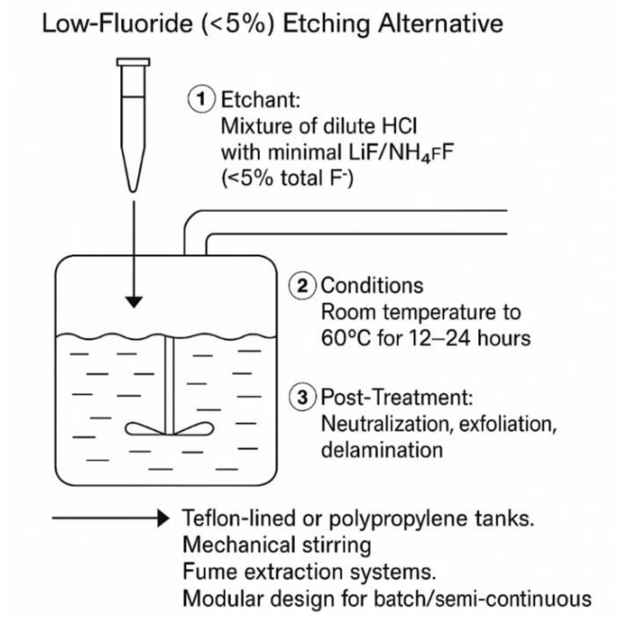


Figure 7: Low-Fluoride Etching Process.

Reactor Design Modifications

- Teflon-lined surfaces of the reactor and polypropylene tanks.
- Mechanical stirring.
- Fume extraction systems.

- Modular design for batch/semi-continuous operation.

These etching reactors were fully designed, developed, and manufactured by the CU team. The latest reactor models have been upgraded and tailored specifically for the processes used in the Greencap project, serving as a prototype for a scalable MXene production system with a capacity of up to 1 kg per day.

Figure 8: a) Modified Laboratory etching reactor with control system and proprietary software developed by CU; b) Previous generation of Etching reactor system; c) Complete view of the system developed by CU; d) Control unit of the etching reaction system; e,f) Interface of the control system at the connected PC; g,h) Touch screen display of the control unit.

Currently, these reactors are in operation at Carbon-Ukraine's dedicated laboratory, installed within chemical fume hoods and equipped with an argon (or other inert gas) supply to ensure safe and stable functioning.

The upgraded reactor concept (wet chemical etching process), developed and established by CU incorporates an advanced engineering design featuring a computer control system with CU proprietary software for precise and user-friendly regulation of key synthesis parameters, due to pre-set processing regimes. This includes programmable heating and cooling, automated feeding of starting powders, and continuous mixing of the etching solution, allowing for consistent and reproducible synthesis and materials end properties on a semi-industrial prototype scale (see Figure 8).

The MXene synthesis methods and processing routes were initially developed and optimized within WP1 by TCD and CU. Material and electrode samples made of various MXene-based slurry formulations were prepared and delivered to project partners for further electrochemical testing, characterization and experimental electrode slurry formulation. Following the analysis and evaluation of these test samples, all etching methods for MXenes were successfully reproduced within WP4. The proposed scale-up concept was then validated and tested using the advanced reactor system developed by CU.

The proposed reactor concept by wet chemical etching with low-fluoride **(<5% total F$^-$**) can serve as a foundational model and prototype for the development of large-scale systems capable of meeting the industrial-level demand for MXenes across various application areas, including energy storage, catalysis, and electronics. Its design enables significant cost reductions by optimizing energy consumption and streamlining material handling processes, ultimately lowering per-unit production costs. Moreover, the system promotes a more sustainable synthesis approach by incorporating resource-efficient operation and safer chemical management. This scalable and modular reactor design proposed by CU holds strong potential for commercial deployment, bridging the gap between laboratory-scale synthesis and full-scale industrial production. Aiming at the up-scaled production of low-fluorine content MXenes, CU is currently collecting the information needed for obtaining the REACH certification once the production would reach the 1 t/year target.

2.2.3 Results

Molten salt etching:

The molten salt approach demonstrated effective etching of MAX-phase materials to produce MXenes in an eco-friendly and scalable manner. Trials using homogeneously mixed powders of Ti_3AlC_2 (MAX-phase), $CuCl_2$, KCl, and NaCl confirmed successful removal of the A-layer and formation of MXene sheets. The process was carried out at 700°C in a continuous-flow argon environment, held for 5 h to ensure full etching.

Morphology: SEM and TEM analyses revealed **well-separated, layered MXene flakes** with minimal oxidation or agglomeration.
Surface Chemistry: The molten salt environment allowed **controlled surface terminations** (mainly halides), leading to tunable hydrophilicity and electronic properties.

The MSRs represent a viable strategy for large-scale MXene synthesis, offering advantages in scalability, sustainability, and safety compared to traditional HF-based methods.

< 5 % fluoride-containing etching:

The method involved mild etching of MAX-phase materials using a dilute HCl solution supplemented with LiF or NH_4F.

- **Reaction Conditions**: Operating at **room temperature to 60°C for 12–24 h** provided effective etching, though slower compared to molten salt methods.
- **Flake Quality**: Resulting MXenes have shown good structural integrity, although **flake delamination was more variable** and required post-sonication.
- **Post-Processing**: Residual salts and by-products were easily removed via DI water washing, followed by dilution of acidic products until neutralization followed by mechanical delamination, usually via sonication or shaking to produce single layer flakes.
- **Reactor Design**: Modified plastic-lined reactors with **mechanical stirring** and **fume extraction systems** ensured safe and reproducible operation.

This process presents a lower-cost and lower-risk option for facilities looking to avoid the use of HF while still achieving functional MXene materials suitable for downstream applications like electrode formulation.

2.3 Skeleton carbide-derived carbon

2.3.1 Preliminary report on upscaling/standardization processes for CG

The upscaling process to allow for the annual production of 10 000 kg of CG required upgrades of the facilities and infrastructure at SM. Silicon carbide is purchased as a starting material, and due to the convenient proximity of an industrial chemical compound in the region it was possible to draw a chlorine gas pipeline directly into the facility. As a result, a significantly larger throughput can be achieved in the chemical reactor (Equation 1).

$$SiC_{(s)} + Cl_{2(g)} \rightarrow C_{(s)} + SiCl_{4(g)} \qquad (1)$$

Here, 33 500 kg of SiCl is reacted with 142 120 kg chlorine gas, resulting in the required 10 000 kg CG. The byproduct consists of 141 901 kg gaseous silicon tetrachloride, for which distillation and storage facilities have been commissioned. Additionally, argon (ca. 40 000 kg) and sodium hydroxide (ca. 40000 kg) are required for the production as process gas and reagent, respectively.

Milling

The CG is processed using planetary ball mills. This is an efficient method that leverages the high-energy impact of balls within the mill to exfoliate and powdering the materials. This technique is particularly advantageous because it does not consume any media, making it a cost-effective and environmentally friendly option. The planetary ball mills operate by rotating the jars at high speeds, causing the balls to collide with the material particles, thereby breaking them down into thinner carbon flakes. This process ensures uniformity and high quality of the produced CG, verified by particle size distribution analytics.

Hydrogenation

A rotary kiln reactor, which is an industrial rotating and inclined furnace, designed for chloride removal from CG operates at approximately 1000°C under a controlled reducing atmosphere, with hydrogen gas at a concentration of 95%. The primary purpose of this high-temperature treatment is to convert residual chloride ions, typically present in the ppm range, into gaseous HCl through hydrogenation. The kiln rotates at a speed of about 0.5 to 1 revolution per minute, ensuring good mixing and uniform heat exposure. This process consumes 1 000 kg hydrogen as well as 1 000 kg argon as a process gas per 10 000 kg CG. SM is increasing its CG production by over 3-fold with the use of rotary kiln reactors which will be commissioned during 2025.

Summarize the metrics and method of standardization for the quality control procedure for CG:

Summarizing the metrics and methods of standardization for the QC procedure for CG is crucial to ensure consistency, reliability, and safety in production. By defining and documenting these metrics, such as particle size distribution, purity levels, and residual chloride content, manufacturers can maintain high standards and meet regulatory requirements. Standardization methods, including precise calibration of equipment and adherence to validated protocols, help in minimizing variability and errors. This systematic approach not only enhances the overall quality of CG but also builds trust with customers and stakeholders, ensuring that the final product consistently meets the desired specifications and performance criteria. The QC steps taken are outlined here:

BET

BET measurements for pore size distribution involve evaluating the specific surface area of CG by gas adsorption, which is measured as a function of relative pressure. This technique helps determine the pore size and volume, providing crucial insights into the material porosity and surface characteristics. BET is performed on the chlorinated CG as it comes out of the ball mill, as well as the final product after hydrogenation.

Chlorine content

The total chlorine content of the chlorinated and final product after hydrogenation is determined. Chlorinated sites in the CG significantly deteriorate the performance of the material, so monitoring the content is crucial.

Ash content

The ash content of the chlorinated and hydrogenated CG is determined. From the ashes of the hydrogenated material, a screening for trace metals is performed (ICP-OES or ICP-MS). Trace metals can indicate the purity of the starting material and can also affect the performance of the final material. The surface groups of the hydrogenated CG are also analysed by a third-party lab.

Particle Size Distribution

The particle size of the milled CG and the final hydrogenated product are monitored using scanning electron microscopy (SEM) by a project partner. The particle sizes determine the active surface area of the material and must be monitored closely to ensure a consistent product.

Table 2: Quality control employed techniques

Quality Control	Method	Instrument	Chlorination	Milling	H_2 treatment
Surface area & pore size distribution	Inhouse	BET	Yes	No	Yes
Total chlorine content	Inhouse	multi-EA 4000	Yes	No	Yes
Ash content	Inhouse	Muffel Furnace	Yes	No	Yes
Particle size distribution	Inhouse	Particle size analyzer	No	Yes	Yes
Surface functional groups	Inhouse (External partner)	Elemental analyzer	No	No	Yes
Metal content	Inhouse (External partner)	ICP – OES	No	No	Yes
Graphitization	Inhouse (projects partners)	SEM	No	No	Yes

2.4 IL-based electrolytes

The most promising electrolytes within the Greencap project are currently $Pyr_{11}BF4$ and $Pyr_{13}BF4$ in acetonitrile. $N_{1113}FSI$ was investigated in the previous stages of the project. The production and formulation of these compounds takes place at SOLV in Toulouse (France). The chemical process of ionic liquid (IL) synthesis has been developed by SOLV over 20 years and the corresponding know-how is protected by trade secrets.

Table 3: Materials employed for the IL-based electrolytes production.

IUPAC name	Abbreviation	EC	CAS
Dimethylpyrrolidinium tetrafluoroborate	$Pyr_{11}BF_4$	834-341-9	69444-51-5

N-propyl-N-methylpyrrolidinium tetrafluoroborate	$Pyr_{13}BF_4$	801-426-7	327022-59-3
Acetonitrile	ACN	200-835-2	75-05-8

2.4.1 Batch

Initial production of $N_{1113}FSI$, $Pyr_{13}BF_4$ and $Pyr_{11}BF_4$ were performed at the bench in batch type reactors (see Figure 9 below). The synthesis of each IL presented its own specific problems in terms of raw materials, processes and purification. All of these ILs required significant development focused on the purification as to achieve the high purity needed for energy storage applications. The batch-based process is compatible with the production of ILs on the kg scale. SOLV works with reactors up to 30L and can be used for a capacity of **150 kg/month** of pure ionic liquid.

Figure 9: Batch type reactor used for IL synthesis.

2.4.2 Pilot line

The batch synthesis process involves moving products and reactants from one reactor to the next. SOLV pilot line, (established before the Greencap project), is based on a continuous flow process wherein reagents are circulated, mixed and processed with a pump system allowing the whole method to be automated. For this, different parameters must be controlled (temperature, flow rates, heat distribution, mix speed/geometry, residence time…). Fine tuning of these parameters is crucial to maximising yields and product purity. Currently, for certain ILs, such as FSI-based ILs, an output of **1.5 t/month** of pure ionic liquid has been achieved. This corresponds to approximately 4-5 t/month of formulated IL electrolytes in ACN. Transfer of IL synthesis from batch to flow reactor requires significant development given the large number of parameters to fix. A simplified illustration of such processes is shown in the figure below:

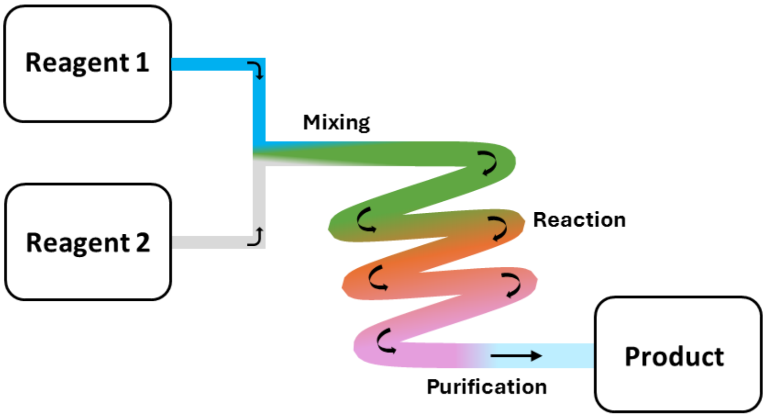


Figure 10: General continuous flow synthesis process.

This technology is the heart of SOLV technology and allows the production of a range of different ILs on the same equipment, including the ones used in GREENCAP. SOLV flow reactor presents several advantages to batch scale processes:

- more efficient heat transfer
- improved mass transfer/mixing
- easy ramp up/down
- in-line downstream processing, improved safety features
- less waste/water/energy
- improved yield

All these factors lead to an average **decrease of 10% in the cost** of ionic liquids when compared to batch-based production, see Figure below.

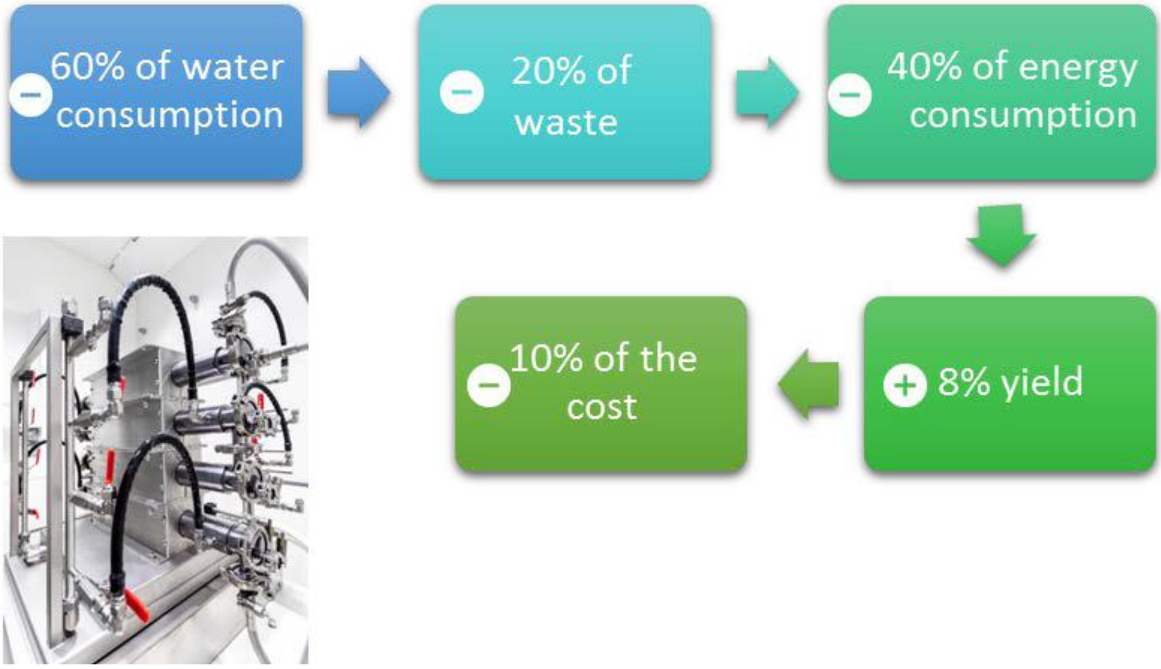


Figure 11: Typical improvements of continuous flow reactions compared to batch systems.

2.4.3 Production lines

As part of SOLV development, the company objective is to start the installation a full industrial line at our site in Toulouse, capable of producing **260 t/year of pure ILs** by 2026. The production line would essentially be an upscaled version of the continuous flow reactor used in the pilot line allowing for higher throughout but also **greater efficiency** in terms of energy and materials wastage. Given that this technology involves multiple reactors of small capacity, the production lines can be easily duplicated, allowing **greater flexibility** in terms of reactivity to customers and market needs, while **reducing CAPEX** and its associated risks.

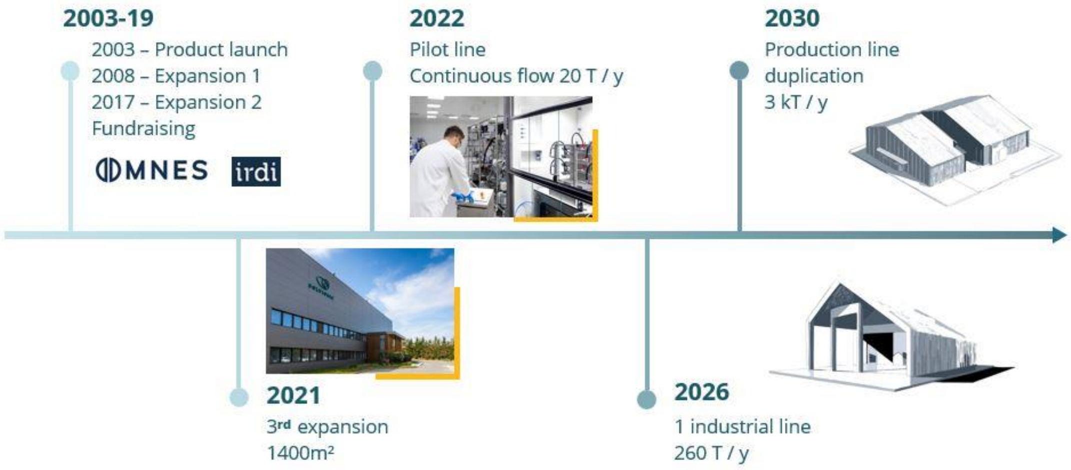
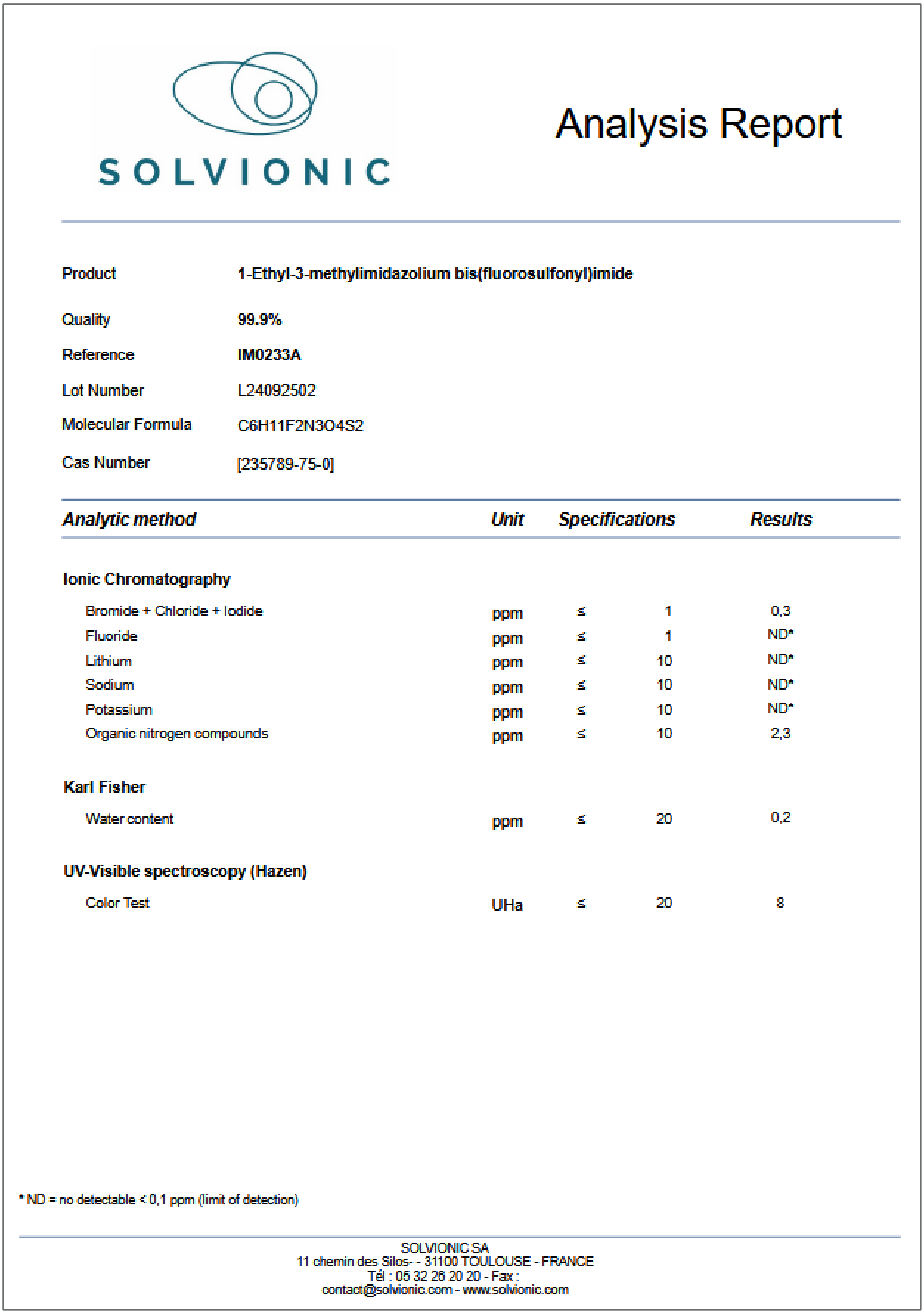


Figure 12: SOLV development roadmap.

2.4.4 Quality control

SOLV has **a dedicated QC laboratory** that measures the quality of products at every stage of the production: from raw materials to final products. For this, SOLV has an internal standardised system for categorising the quality of ILs or electrolytes and validating their conformity versus certain specifications. Given the nature of energy storage systems, electrolyte grade materials are the highest purity (grade A), which are at **minimum 99.9% pure** (≤ 1 ppm halide; ≤ 10 ppm organic nitrogen compounds) and contain **less than 20 ppm** of water. Different methods are used to quantify trace compounds including ion chromatography (IC) and Karl Fischer (KF) methods. In addition to the quantification of trace elements, SOLV is also equipped to measure physicochemical properties of electrolytes (density, viscosity, conductivity, electrochemical stability window…) and can use these physical properties as criteria for QC. Unique batch numbers are attributed to products as to **ensure traceability** and are conditioned in a protective atmosphere to **guarantee shelf life.** Importantly, the methods and equipment that SOLV is currently using are **compatible with upscaled production volumes**. SOLV generates **certificate of analysis** for each batch of ionic liquid which are available on SOLV’s website.



SOLVIONIC

Analysis Report

Product	**1-Ethyl-3-methylimidazolium bis(fluorosulfonyl)imide**
Quality	**99.9%**
Reference	**IM0233A**
Lot Number	L24092502
Molecular Formula	$C_6H_{11}F_2N_3O_4S_2$
Cas Number	[235789-75-0]

Analytic method	*Unit*	*Specifications*		*Results*
Ionic Chromatography				
Bromide + Chloride + Iodide	ppm	≤	1	0,3
Fluoride	ppm	≤	1	ND*
Lithium	ppm	≤	10	ND*
Sodium	ppm	≤	10	ND*
Potassium	ppm	≤	10	ND*
Organic nitrogen compounds	ppm	≤	10	2,3
Karl Fisher				
Water content	ppm	≤	20	0,2
UV-Visible spectroscopy (Hazen)				
Color Test	UHa	≤	20	8

* ND = no detectable < 0,1 ppm (limit of detection)

SOLVIONIC SA
11 chemin des Silos- - 31100 TOULOUSE - FRANCE
Tél : 05 32 26 20 20 - Fax :
contact@solvionic.com - www.solvionic.com

Figure 13: Example of COA for EMImFSI. Available at https://solvionic.com/en/content/support#certificats.

2.5 Electrode Material inks/slurries

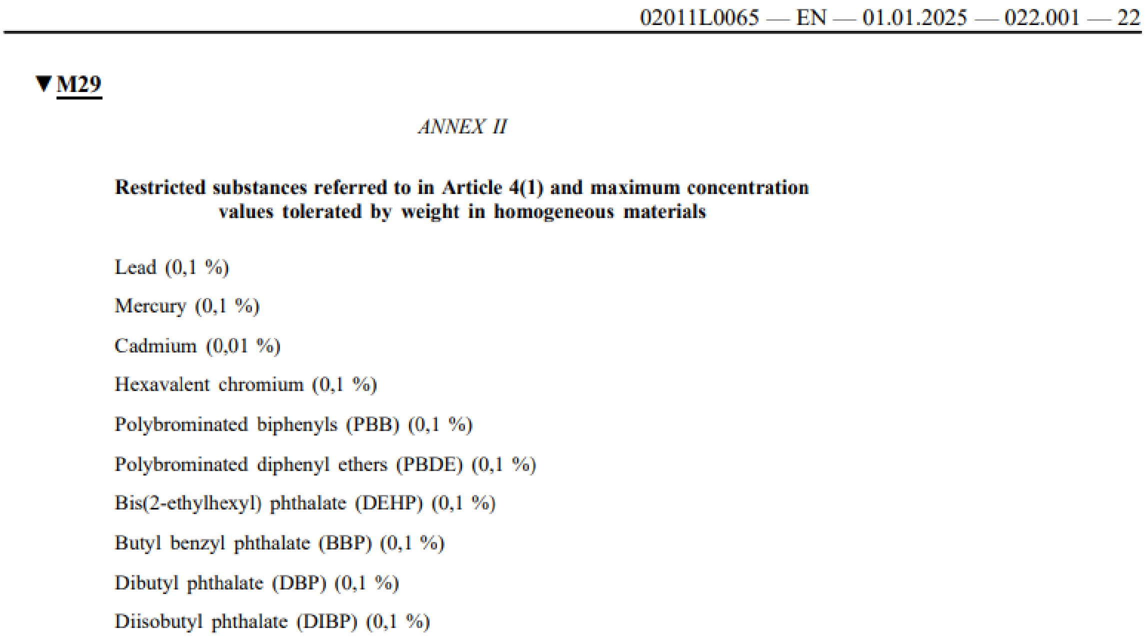
The slurry selected in T3.1 and described in D3.1 will be upscaled by SKL. Specifically, BeD produced the materials (FLG) and organized the shipment to SKL having the slurry scale-up production infrastructure. Both the aqueous binder and the active material are provided by SM. Specifically, the slurry scale-up production refers to the increased capacity of the 20 g laboratory-scale amount up to the 80 kg required to produce the cylindrical cell electrodes. For this purpose, BeD organized the shipment of ca. 3.5 kg of FLG, equal to the 2 w% necessary for the slurry formulation selected in D3.1 ($CG_{94}FLG_2CMC\text{-}SBR_4$). Moreover, to produce the cylindrical cell industrial prototype, SOLV provided 1 kg of both 1 M $Pyr_{13}BF_4$/ACN and 1.2 M $Pyr_{11}BF_4$/ACN electrolytes to SKL.

2.5.1 Slurry quality control

The quality of the slurries is evaluated using a grindometer, a viscometer and monitoring the solid to liquid mass ratio. In detail, the grindometer evaluated the dispersion particle size, which should not exceed 10 µm for obtaining homogeneous slurries and coatings. Specifically, the smaller particle sizes increase the surface accessible to the electrolyte and the contact between the particles, determining higher conductivities and densities. However, particles under 10 µm tend to aggregate during storage and limit the slurry stability over time, which requires additional mixing processes after long storage periods. The viscometer is used for evaluating the viscosity of the dispersion, which should be ca. 1000 cP for doctor blading deposition and < 1000 cP for spray coating techniques. The solid percentage of the total mass of the slurry, which satisfies these requirements, is expected to be 33 %.

2.5.2 RoHs Certification

Regardind RoHs (Restriction of Hazardous Substances) Directive, it was adopted by the European Union, and it places restrictions on the content of certain substances (see Figure 14) in electrical and electronic devices.



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▼**M29**

ANNEX II

Restricted substances referred to in Article 4(1) and maximum concentration values tolerated by weight in homogeneous materials

Lead (0,1 %)

Mercury (0,1 %)

Cadmium (0,01 %)

Hexavalent chromium (0,1 %)

Polybrominated biphenyls (PBB) (0,1 %)

Polybrominated diphenyl ethers (PBDE) (0,1 %)

Bis(2-ethylhexyl) phthalate (DEHP) (0,1 %)

Butyl benzyl phthalate (BBP) (0,1 %)

Dibutyl phthalate (DBP) (0,1 %)

Diisobutyl phthalate (DIBP) (0,1 %)

Figure 14: Annex II of the Article 4(1) of the RoHs directive. Restriction list of the hazardous materials with the maximum admitted content.

RoHS and other efforts to reduce hazardous materials in electronics are motivated in part to address the global issue of consumer electronics waste. In addition to the high-tech waste problem, RoHS reflects contemporary research over the past 50 years in biological toxicology that acknowledges the long-term effects of low-level chemical exposure on populations. New testing is capable of detecting much smaller concentrations of environmental toxicants. Researchers are associating these exposures with neurological, developmental, and reproductive changes. Device manufacturers are therefore required to declare compliance with the RoHS directive by making a declaration of conformity if the analysis of the constituents can exclude the percentage presence of the substances listed above.
In the case of Greencap supercapacitors, from the analysis of the main constituents, we can consider ourselves compliant and therefore make the declaration of conformity for placing on the market.

2.6 Contribution to project (linked) Objectives

The present deliverable is an important contribution to the key objectives of the GREENCAP project, as defined in the Description of Action (DoA) Part B on pages 5 and 6. More specifically, it reports the upscaling and standardization of CRM-free 2D materials for the fabrication of the SC electrodes:

- The production of SLG/FLG flakes has been upscaled for different production methods, *i.e.* WJM (> 10^3 kg/year), HPH (> 10^3 kg/year) and electrochemical exfoliations.
- The production of CG has been scaled up to the project objective (> 10^5 kg/year).
- The production of MXenes with low-fluorine content (> 1kg/day)
- Quality control procedures have been developed, based on ISO TS 21356-1 standard, assessing the production SLG/FLG.
- The REACH registration procedure has been started for BeD FLG.
- SOLV has carried out the development and production of IL-based electrolytes at the batch-scale (> 150 kg/month). The ILs selected within WP1, WP2 and WP3 are compatible with the scaled-up production within SOLV pilot plant (1.5 t/month).
- SKL implemented the scale-up production of the slurry-based CG and on the formulations optimized within the WP3 and described in D3.1.

2.7 Contribution to major project exploitable result

- Sustainable and scaled-up production of FLG (>10^3 kg/year) by BeD, UCAM (Type: T)
- Scaled-up electrochemical exfoliation of graphite by TUD (Type: T)
- Development of a system for the scaled-up production of CRM-free MXenes with a low-fluorine content (Type: T)
- Sustainable synthesis processes for IL-based electrolytes (Type: M)
- Novel IL-based electrolytes for high energy SC (Type: T)
- Know-how on the deposition and processing of CRM-free 2D EMs (Type: T)
- Novel high-capacitance EMs based on CRM-free L2DMs, including functionalized/hybrid materials (Type: T)

3 Conclusions and Recommendations

Deliverable D4.1 describes the consortium efforts for scaling-up the production of the electrode and electrolyte materials fulfilling the project objectives. Moreover, D4.1 describes the parameters and the procedure for scaling-up the production of the slurries. The described efforts and key exploitable results pave the way to the direction of the industrialization of the Greencap innovative supercapacitor technology.

4 Risks and interconnections

4.1 Risks/problems encountered

Risk No.	What is the risk	Probability of risk occurrence[1]	Effect of risk[1]	Solutions to overcome the risk
WP4.1	Upscaling of CRM-free MXenes production: MXene production capability lower than kg/day scale for a single reactor and using alcohol-based solvents.	2	2	Increase the process temperature for the iodine etching method to increase the MXene formation kinetics. Adoption of common chemical industry processes to carry out high temperature molten salt etching, being supported by industrial players interested in this activity (e.g. Carbon-Ukraine, with which TCD strongly collaborates).
WP4.2	Upscaling and prototyping: developed materials cannot be provided in sufficient quantity for the massive slurry production.	2	3	Screening of multiple electrode slurry formulations, minimizing the amounts of materials produced with insufficient quantities and focusing on the most scalable ones.
WP4.3	Upscaling and prototyping: upscaled materials show low performance.	2	1	Decreasing batch sizes and increasing batch number can mitigate this risk as the materials properties can be maintained on a smaller scale, while providing sufficient amount of materials needed for the SC prototype.

[1] Probability risk will occur: 1 = high, 2 = medium, 3 = Low

4.2 Interconnections with other deliverables

The efforts carried out and described within this document are at the basis of the whole WP4. Specifically, it is dedicated to the exploitation of the knowledge and research performed for the production of an industrial prototype such as the cylindrical cell. It poses the foundations of a successful industrial manufacturing chain for the large-scale production of SCs based on novel 2D materials and ILs. This document is finely connected with all the other deliverable of this WP and it will be followed by an update in month 36.

5 Deviations from Annex 1

No deviations

6 References

7 Acknowledgements

The author(s) would like to thank the partners in the project for their valuable comments on previous drafts and for performing the review.

Project partners:

#	Partner short name	Partner Full Name
1	BeD	BEDIMENSIONAL SPA
2	SOLV	SOLVIONIC
3	FSU	FRIEDRICH-SCHILLER-UNIVERSITAT JENA
4	SKL	SKELETON TECHNOLOGIES OU
5	TCD	THE PROVOST, FELLOWS, FOUNDATION SCHOLARS & THE OTHER MEMBERS OF BOARD, OF THE COLLEGE OF THE HOLY & UNDIVIDED TRINITY OF QUEEN ELIZABETH NEAR DUBLIN
6	TUD	TECHNISCHE UNIVERSITAET DRESDEN
7	UNISTRA	UNIVERSITE DE STRASBOURG
8	SM	SKELETON MATERIALS GMBH
9	UNR	UNIRESEARCH BV
10	CNR	CONSIGLIO NAZIONALE DELLE RICERCHE
11	UCAM	THE CHANCELLOR MASTERS AND SCHOLARS OF THE UNIVERSITY OF CAMBRIDGE
12	CU	Y CARBON LLC

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8 Appendix A - Quality Assurance Review Form

Question	**WP Leader**	**Reviewer**	**Project Coordinator**
	Paul Ionescu (SKL)	Ali Shaygan Nia (TUD)	Francesco Bonaccorso (BED)
1. *Do you accept this Deliverable as it is?*	Yes	Yes	Yes
2. *Is the Deliverable complete?* - *All required chapters?* - *Use of relevant templates?*	Yes	Yes	Yes
3. *Does the Deliverable correspond to the DoA?* - *All relevant actions preformed and reported?*	Yes	Yes	Yes
4. *Is the Deliverable in line with the GREENCAP objectives?* - *WP objectives* - *Task Objectives*	Yes	Yes	Yes
5. *Is the technical quality sufficient?* - *Inputs and assumptions correct/clear?* - *Data, calculations, and motivations correct/clear?* - *Outputs and conclusions correct/clear?*	Yes	Yes	Yes
6. *Is created and potential IP identified and are protection measures in place?*	Yes	Yes	Yes
7. *Is the Risk Procedure followed and reported?*	Yes	Yes	Yes
8. *Is the reporting quality sufficient?* - *Clear language* - *Clear argumentation* - *Consistency* - *Structure*	Yes	Yes	Yes

9 Appendix B – Quotation

Università di Genova | DCCI DIPARTIMENTO DI CHIMICA E CHIMICA INDUSTRIALE

Genova, 29/05/2025

All'att.ne Dott. Francesco Bonaccorso
Dott.ssa Elisa Mantero
Dott.ssa Lavinia Crosia
BeDimensional S.p.A.
Lungotorrente Secca 30 R
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Tel: 010 236 4170
www.bedimensional.com
P.iva 02389840998

Oggetto: Offerta per analisi di due campioni in polvere di nitruro di boro e uno di grafene

Spett.le BeDimensional,

a seguito di Vostra gent.le richiesta, siamo a proporvi la nostra migliore offerta su base forfettaria per lo svolgimento di analisi di caratterizzazione di due campioni di nitruro di boro e uno di grafene mediante le seguenti tecniche di caratterizzazione:

- Specific surface area (SSA)
- Volume specific surface area (VSSA)
- FT-IR
- Determination of Loss on Ignition

L'importo per Voi riservato in esclusiva è di **1500 €** + Iva di legge.

Il pagamento delle prestazioni avverrà a seguito della presentazione di fattura elettronica da parte dello scrivente

Firma per Accettazione

____

Il Responsabile del Laboratorio

Antonio Comite

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