

D6.6: Materials Synthesis and Characterization Access Provision

WP6 - TA2 – Access to material synthesis and characterization



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Responsible person for the deliverable	Pedro Barquinha (UNOVA)
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List of abbreviations

FZJ – Forschungszentrum Jülich GmbH

HMU – Hellenic Mediterranean University

JOR – Joanneum Research Forschungsgesellschaft mbH

OBU - Oxford Brookes University

TA – Transnational Access Activities

TA2 – Access to material synthesis and characterization.

TLO – Technical Liaison Office

TUD – Technische Universität Dresden

RISE – Research Institute of Sweden

RTO - Research and Technology Organizations

SME – Small Medium Enterprise

UoA – Units of Access

UNOVA – Instituto de Desenvolvimento de Novas Tecnologias (UNINOVA)

WP – Work package

WUT – Warsaw University of Technology

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1. Executive Summary

The deliverable 6.6 – Materials Synthesis and Characterization Access Provision belongs to the Work Package 6 (WP6) titled as TA2 – Access to material synthesis and characterization.

The purpose of this report is to provide a comprehensive summary of the proposals related to TA2 which were submitted during the first five calls of EMERGE, i.e., up to December 2023 (M30).

Overall, twenty seven proposals were submitted on the EMERGE website requesting materials synthesis and characterization access provision (TA2). Fifteen projects were successfully completed, two were rejected and eight are currently under evaluation. Two were not suitable for TA2 access provision, therefore, they were carried out at different Transnational Access Activities (TA). Besides that, eight proposals were initially submitted for different TAs but TA2 activities were also included during the project execution.

2. Proposals

2.1 Concluded proposals originally submitted to TA2 access

Fifteen projects, which initially requested materials synthesis and characterization access provision, were successfully completed by the end of 2023. Nine of them were done in-person, three remotely and three on a hybrid mode. Three projects were concluded by the end of 2022 and twelve by the end of 2023. HMU, TUD and WUT hosted four projects each. UNOVA hosted two projects while RISE received one. Table 1 brings the details about each project such as the title, project call and the period of implementation.

Table 1: List of projects submitted for TA2 and concluded by M30.

Call/ Proposal ID	Project Title	EMERGE institutio	User type of access	Period of project implementation
1st Call 3651	Screen printing of TiO ₂ / CeO ₂ powder mixture for oxygen sensors	WUT	In-person	Start date: 25-10-2022 End date: 27-10-2022
1st Call 3652	Graphene-based electrically conductive coatings	HMU	In-person	Start date: 29-11-2022 End date: 01-12-2022
1st Call 3662	Electrochemically exfoliated 2D materials for printed electronics	TUD	In-person	Start date: 29-11-2022 End date: 09-12-2022
2nd Call 3856	Nanocoatings with antiviral properties on cement-based surfaces	UNOVA	In-person	Start date: 22-05-2023 End date: 03-06-2023

3rd Call 4001	Formulating screen printing ink/paste application for resistive heater application	WUT	Remote	Start date: 01-06-2023 End date: 30-06-2023
3rd Call 4010	Thin and flexible electrodes for capacitance measurement	WUT	Remote	Start date: 15-06-2023 End date: 30-06-2023
3rd Call 4014	Bioresorbable Force Sensor using Biodegradable Piezoelectric Material	TUD	In-person	Start date: 26-06-2023 End date: 28-06-2023
3rd Call 4015	Printed Bio-inspired Hydrogel based Substrate with Adhesiveness and Tailored Electrical Conductivity for Sustainable Electronics	TUD	Remote	Start date: 05-11-2023 End date: 14-11-2023
4th Call 4594	Insertion of ultrathin low-conductivity passivation layers in Perovskite solar cell	HMU	Hybrid	Start date: 08-09-2023 End date: 30-11-2023
4th Call 4682	Interface engineering in perovskite solar cells based on 2D materials	HMU	Hybrid	Start date: 31-08-2023 End date: 30-11-2023
4th Call 5549	Fabrication and electrical characterization on two-dimensional (2D) polymers-based flexible organic electronics	UNOVA	In-person	Start date: 03-07-2023 End date: 14-07-2023
4th Call 5814	Screen-printed structures of alternating TiO ₂ and CeO ₂ layers used as an oxygen-sensing materials	RISE	Hybrid	Start date: 07-08-2023 End date: 07-12-2023
4th Call 5816	Integration of ultrasonically exfoliated eco-friendly graphene into perovskite solar cell device fabrication	HMU	In-person	Start date: 23-10-2023 End date: 31-10-2023
4th Call 5824	Thin film carbon nanotubes and membranes-based sensors development and characterization to enhance sensors sensitivity and selectivity	TUD	In-person	Start date: 09-10-2023 End date: 12-10-2023
4th Call 5826	2D/3D CELL-PILs for tissue engineering	WUT	In-person	Start date: 23-10-2023 End date: 02-11-2023

In total, twenty-three users participated in TA2 projects up to the December 2023. Eight projects had two participants, while seven had only one. Most of the proposals were applied from University Institutes (60 %), followed by RTOs (20 %), SME (13 %) and Industry (7 %). Eight out of fifteen projects were carried out by European institutions (53 %) while seven were by non-European ones (47 %). Table 2 summarizes the information about users and their home institution for each proposal.

Table 2: Information about users who carried out concluded projects which were originally submitted for TA2.

Call Proposal ID	User (nationality)	Research role	Home Institution (Country)	Type of Institution
1st Call 3651	Milija Sarajlic (Serbia) Evgenija Milinkovic (Serbia)	Senior Scientist PhD Student	IHTM (Serbia)	RTO
1st Call 3652	Marilena Zappia (Italy)	Post-doc	BeDimensional S.p.A. (Italy)	Industry

1 st Call 3662	Bing Wu (China)	PhD Student	University of chemistry and technology, Prague (Czech Republic)	University
2 nd Call 3856	Sara Dervishi (Albania) Silvana Gjyli (Albania)	PhD Student Senior Scientist	University of Tirana, Faculty of Natural Sciences (Albania) Pespa Alumin SHPK (Albania)	University Industry
3 rd Call 4001	Arun Prakash Aranga Raju (United Kingdom) Ugo Sassi (Italy)	Manager Manager	Levidian Nanosystems Limited (United Kingdom)	SME
3 rd Call 4010	Kyoko Jansson (Sweden)	Manager	Innovation Partners (Europe) AB (Sweden)	SME
3 rd Call 4014	Youssif Merhi (Denmark)	PhD Student	Aarhus Universitet (Denmark)	University
3 rd Call 4015	Monisha Monisha (India)	Post-doc	Aarhus Universitet (Denmark)	University
4 th Call 4594	Sihui Liu (China) Chengning Yao (China)	PhD Student PhD Student	Imperial College London (United Kingdom)	University
4 th Call 4682	Sihui Liu (China) Chengning Yao (China)	PhD Student PhD Student	Imperial College London (United Kingdom)	University
4 th Call 5549	Bowen Zhang	PhD Student	Fraunhofer IKTS (Germany)	RTO
4 th Call 5814	Jelena Stevanović (Serbia) Milija Sarajlic (Serbia)	PhD Student Senior Scientist	Institute of Chemistry, Technology and Metallurgy (Serbia) IHTM (Serbia)	RTO
4 th Call 5816	Iakovos Tzanakis (United Kingdom) Amanpreet Kaur (India)	Professor-scientific coordinator Post-doc	Oxford Brookes University (United Kingdom)	University
4 th Call 5824	Sahira Carolina Vasquez Baez (Dominican Republic) Martina Angeli (Italy)	PhD Student Assistant Professor	Free University of Bozen-Bolzano (Italy)	University
4 th Call 5826	Marta Corvo (Portugal)	Senior Scientist	NOVA FCT (Portugal)	University

In the sections below, one can read the detailed description of projects objectives, requested TA Installations, used TA installations and respective Units of Access (UoA).

Proposal 3651: Screen printing of TiO₂ / CeO₂ powder mixture for oxygen sensors (WUT)

- **Objectives:** A systematic analysis of various TiO₂ and CeO₂ powder combinations obtained by using planetary mill was done by the users during a previous research. Afterwards, the powder was used to make a paste which was screen printed to the substrate with interdigitated electrodes. The uniformity and the repeatability of the

paste screen-printing was an issue, and the users would like to mitigate by using automated screen-printing process.

- Requested TA/installations: TA3.3. Industrial printing / TA2.1. Materials characterization.
- Used TA/installations: Materials characterization (3 UoA).

Proposal 3652: Graphene-based electrically conductive coatings (HMU)

- Objectives: 1) Validation of low-temperature curable graphene-based pastes/inks based on thermoplastic binding agents for metal electrode replacement in solar cells, e.g., perovskite solar cells; 2) validation of low-temperature laminable graphene-based electrically conductive adhesive for solvent-free processes.
- Requested TA/installations: Validation and standardization; TA2 Chemical & physical techniques.
- Used TA/installations: Validation and standardization (2 UoA); Chemical & physical techniques (4 UoA).

Proposal 3662: Electrochemically exfoliated 2D materials for printed electronics (TUD)

- Objectives: A facile and cost-effective method, based on the electrochemical exfoliation of black phosphorus and synchronized electrochemical deposition of aluminium oxide will be proposed to passivate exfoliated black phosphorus. The prepared black phosphorus is expected to exhibit high air stability and then the sample will be applied to a 3D-printed macro-supercapacitor.
- Requested TA/installations: TA2.1. Materials characterization; TA3.2. Functional 2D & 3D printing
- Used TA/installations: Physical techniques (9 UoA); Chemical techniques (2 UoA); Materials characterization (5UoA)

Proposal 3856: Nanocoatings with antiviral properties on cement-based surfaces (UNOVA)

- Objectives: The aim of this proposal is to optimize the synthesis of nano-coated cement-based materials (concrete), which by undergoing the technology of coating with photocatalytic materials will lead to building structures with antiviral, self-cleaning

and antibacterial. The use of TiO₂ nanoparticles supported in zeolite or reduced Graphene Oxide (rGrO) could effectively curb this growing threat through a "contact killing" mechanism.

- Requested TA/installations: TA2.1. Materials characterization; TA2.2. Chemical & physical techniques.
- Used TA/installations: Materials characterization (13 UoA).

Proposal 4001: Formulating screen printing ink/paste application for resistive heater application (WUT)

- Objectives: Existing technology for producing flexible resistive heaters are based on metallic meshes such as silver, copper etc. We would like to explore graphene an alternative material for this application. The production of Levidian graphene is from sustainable sources taking methane and waste gas and converting into high-quality graphene and hydrogen using plasma chemistry. Our process will provide a low-cost solution for bulk scale applications. A preliminary exploration of our graphene powders to make inks/pastes for flexible electronics (heater) application. Ideally, we would like to understand if our material is suitable for bulk scale flexible heater application as a low-cost solution.
- Requested TA/installations: TA3.1. Device Preparation; TA2.1. Materials characterization; TA2.2. Chemical & physical techniques
- Used TA/installations: TA1.1 Device design and architecture; TA2 Material synthesis and ink formulation; TA3 Prototype fabrication; TA4 Characterization of prototypes and demonstrators.

Proposal 4010: Thin and flexible electrodes for capacitance measurement (WUT)

- Objectives: The main project goal is to determine the possibility of using printed electrodes as sensors for bladder fluidity monitoring with capacitance monitoring. For that, the set of 50 printed electrodes will be printed. The first essential design consisting of a few layers (conductive layer, isolation layer, etc.) printed on flexible substrates (TPU or PET foil) will be elaborated. The EMERGE partner will support on the technologies requirements and limitations, and several equipments will be used

to assess the electric stability of the electrodes samples in the biomedical application

- Requested TA/installations: TA3.1. Device Preparation / TA2.2. Chemical & physical techniques / TA1.1. Device design and architectures
- Used TA/installations: TA2.1 Materials Characterization (0.4 UoA) / TA 3.3 Industrial printing (1 UoA)

Proposal 4014: Bioresorbable Force Sensor using Biodegradable Piezoelectric Material (TUD)

- Objectives: The overall objective of this experiment was to determine the crystalline and phase changes induced by the post-processing techniques employed on films made of piezoelectric biodegradable Poly-L-Lactide acid. Post-processing methods, such as tensile stretching, were utilized to further optimize and enhance the mechanical and piezoelectric properties to align the material and increase its crystallinity.
- Requested TA/installations: TA2.1. Materials characterization / TA2.2. Chemical & physical techniques
- Used TA/installations: TA2.1 Materials Characterization 2 (3 UoA)

Proposal 4015: Printed Bio-inspired Hydrogel based Substrate with Adhesiveness and Tailored Electrical Conductivity for Sustainable Electronics (TUD)

- Objectives: The goal was to characterize a final material (hydrogel or films) through UV-vis absorption spectrophotometer, XRD, FTIR and DSC-TGA. The material includes commonly used polymer, plasticizer and dopamine. The final aim of this project is to develop novel hydrogel composite that has adhesive properties, electronic properties and is biodegradable
- Requested TA/installations: TA2.1. Materials characterization / TA2.2. Chemical & physical techniques
- Used TA/installations: TA2.1 Materials Characterization 1 (1.25 UoA) / TA2.1 Materials Characterization 2 (3.25 UoA)

Proposal 4594: Insertion of ultrathin low-conductivity passivation layers in Perovskite solar cell (HMU)

- **Objectives:** Perovskite solar cells can reduce their surface recombination by add a low-conductivity interlayer between the absorber and transport layer. To balance the open-circuit voltage and fill factor, the user aims to overcome this challenge by introducing a thick (about 100 nanometers) insulator layer with random nanoscale openings based on the recent publications. Hexagonal boron nitride (h-BN) is a 2D material and consists of hexagonal rings in alternating B and N atoms linked by covalent bonds. Its chemical, electrical, mechanical, and thermal resistance make it a perfect encapsulating material or insulating layer in devices manufacturing. The chemical inert layer of h-BN can also limit the formation of dangling bonds which lead to charge traps. Together with its large band gap (5.5 eV) gives a solution to prevent gate leakage current in small size devices design. Around 100 nm thick h-BN film will be deposited above the hole transporting layer with Perovskite, electronic transporting layer and electrodes on it. The device will then be characterized to see any improvement on the efficiency.
- **Requested TA/installations:** TA3.1. Device Preparation / TA2.2. Chemical & physical techniques
- **Used TA/installations:** TA2.1 Materials Characterization (2 UoA) / TA2.2. Physical techniques (2 UoA) / TA4.1 Device metrology & characterization (10 UoA)

Proposal 4682 Interface engineering in perovskite solar cells based on 2D materials (HMU)

- **Objectives:** This project aims to first characterize solar cells with Ti_3C_2 doping and Ti_2C doping at cell level using Raman, SEM, AFM, PL, and work function. Secondly, the users aim to performance studies of perovskite solar cells doped with another promising MXenes (Nb_2C). By varying the concentration of Nb_2C in the perovskite precursor solution, or by varying the spin-coating times of doping, we plan to discover the best method for the optimal performance of Nb_2C doped perovskite solar cells.
- **Requested TA/installations:** TA2.1. Materials characterization / TA3.1. Device Preparation / TA4.1. Device metrology & characterization

- Used TA/installations: TA2.1 Materials Characterization (4 UoA) / TA4.1. Device metrology & characterization (19 UoA) / TA2.2 Physical Techniques (4 UoA)

Proposal 5549 Fabrication and electrical characterization on two-dimensional (2D) polymers-based flexible organic electronics (UNOVA)

- Objectives: The main goal is to fabricate several sets of flexible transistors based on novel 2D polymers. Furthermore, it focusses on the evaluation of mechanical stability and performance evolution on all transistors under various strains after different cyclic deformation cycles.
- Requested TA/installations: TA2.2. Chemical & physical techniques / TA4.1. Device metrology & characterization
- Used TA/installations: TA2.1. Materials characterization (4 UoA) / TA4.1. Device metrology & characterization (1 UoA)

Proposal 5814 Fabrication and electrical characterization on two-dimensional (2D) polymers-based flexible organic electronics (RISE)

- Objectives: 1) Screen printing of CeO₂ and TiO₂ strips and stacked layers onto PET and Ceramic substrates for oxygen sensing applications. 2) Obtaining the various designs of printed materials, with different geometry within each. 3) Investigation of the photonic sintering of printed structures on PET and Ceramic substrates; influence of different sintering parameters will be examined in home institution. 4) Laser cutting of alignment frame for ceramic sensor substrates.
- Requested TA/installations: TA3.3. Industrial printing / TA2.1. Materials characterization
- Used TA/installations: TA1.1 Device Design and architecture (1.5 UoA) / TA2.1. Materials characterization (0.8 UoA) / TA3.3. Industrial printing (2.5 UoA)

Proposal 5816 Integration of ultrasonically exfoliated eco-friendly graphene into perovskite solar cell device fabrication (HMU)

- Objectives: At higher level, the research going on at Oxford Brookes University (United Kingdom) aims to employ our ultrasonically exfoliated eco-friendly graphene

to develop low-temperature processed electrically conductive graphene-based pastes and inks replacing metal-based electrodes to be used in perovskite solar cells and temperature-sensitive semiconductors. The objectives at the EMERGE project were: (1) electrical and morphological characterization analyses of graphene ink-based spray-coated thin films; (2) validation of thermoplastic binding agents-based low-temperature curable graphene-based pastes for replacing metal electrodes in perovskite solar cells and (3) validation of a graphene-based, low-temperature laminable electrically conductive pastes for operations without solvents.

- Requested TA/installations: TA3.3. Industrial printing / TA2.1. Materials characterization
- Used TA/installations: TA2.1 Materials characterization (5 UoA) / TA2.2. Chemical & Physical techniques (2 UoA)

Proposal 5824 Thin film carbon nanotubes and membranes-based sensors development and characterization to enhance sensors sensitivity and selectivity (TUD)

- Objectives: To investigate the properties of spray-coated carbon nanotube (CNT)-based films on polyamide foils for the development of gas sensors. To study the morphology and permeability characteristics of spin-coated polydimethylsiloxane (PDMS) membranes designed for NH₃ gas sensors.
- Requested TA/installations: TA4.1. Device metrology & characterization / TA2.1. Materials characterization
- Used TA/installations: TA2.1 Materials Characterization (7 UoA)

Proposal 5826 2D/3D CELL-PILs for tissue engineering (WUT)

- Objectives: The aim of this proposal was to develop 2D/3D cellulose-poly(ionic liquid) composites as hydrogels for tissue engineering. The presence of PILs is expected to improve ion transport and provide the stabilization for additional components such as therapeutical agents. Specifically incorporating conductive additives into cellulose composites enhances the regenerative potential of scaffolds. Conductive properties support cell adhesion, proliferation, and differentiation. Therefore, users aim 1) to obtain composites from methacrylated gelatine and poly(ionic liquids); 2) to tune the

ratios and viscosity of the composites to obtain stable inks for 3D printing; 3) to print hydrogels from the optimized inks using the Bio X-3D bioprinter; 4) to use different ink formulations, introducing cellulose nanofibers; 5) to study the influence of methacrylated gelatine analogues in the printability of the inks; 6) to obtain freeze-dried scaffolds from the printed substrates.

- Requested TA/installations: TA3.3. Industrial printing / TA2.1. Materials characterization
- Used TA/installations: TA1.1 Device Design and architecture (0.9 UoA) / TA2.2 Chemical & physical techniques (2.4 UoA) / TA 3.3 Industrial printing (3.4 UoA)

2.2 Proposals submitted and currently under evaluation

During the EMERGE 5th Call, eight proposals (6387, 6388, 6407, 6422, 6425, 6430, 6431 and 6435) were submitted requesting materials synthesis and characterization access provision, and they are all currently under evaluation. Six of them were applied by universities while two from the industry. Moreover, four institutions are from the European Union (Denmark, Portugal, Italy and Poland) while two are from the United Kingdom, one from Serbia and one is from Turkey. Table 3 brings information about the proposals which are under evaluation.

Table 3: List of proposals submitted for TA2 access to the EMERGE infrastructure in M30 which are under evaluation.

Call/ Proposal ID	Proposal title	Home Institution (Country)	Type of Home Institution	Pretended EMERGE institution(s)
5 th Call 6387	Detailed electrical characterization and comparison of different types of graphene	Institute of Chemistry, Technology and Metallurgy (Serbia)	University	HMU
5 th Call 6388	Addressing the issue of hydrogen gas evolution in printed alkaline Zn-Mn flexible batteries, via surface morphology and impurities analysis	Zinergy UK Ltd (United Kingdom)	Industry	UNOVA
5 th Call 6407	Bioresorbable Force Sensor using Biodegradable Piezoelectric Material	Aarhus Universitet (Denmark)	University	TUD
5 th Call 6422	Scalable Fabrication of Printed Stretchable Electronics	University of Coimbra (Portugal)	University	RISE
5 th Call 6425	High-energy and long-cycling lithium-sulfur pouch cell using Few-Layer-Graphene coated Aluminium current collector	BeDimensional Spa (Italy)	Industry	TUD
5 th Call 6430	Prototype fully recyclable screen printed electronics	Swansea University (United Kingdom)	University	RISE

5 th Call 6431	Patient-Specific Numerical Modeling in Palliative Solutions for Infants with Dependent Pulmonary Blood flow.	Istanbul Medipol University (Turkey)	University	FZJ RISE
5 th Call 6435	Study of electrodes' surface modification effect with new perylenediimide derivatives	Lodz University of Technology (Poland)	University	FZJ TUD

*Although FZJ is not formally a WP6 member, two projects were submitted requesting FZJ as hosting partner for materials synthesis and characterization access provision. The TLO will decide if the project will be reassigned to other TA or if another partner from WP6 will host it.

2.3 Proposals submitted to TA2 but reassigned to other TAs

Two proposals were submitted requesting materials synthesis and characterization transnational access but, after the evaluation from the TLOs, were reassigned to other more suitable TAs. Proposal 4009 (3rd Call) was carried out at industrial printing and validation and standardization utilities from WUT; and proposal 4077 (3rd Call) was done using device metrology and characterization equipments at TUD. Table 4 summarizes the information about the reassigned projects.

Table 4: List of proposals submitted for TA2 access to the EMERGE infrastructure from 2021 to 2023 but were approved for other TAs access.

Call/ Proposal ID	Proposal title	Home Institution (Country)	Type of Home Institution	EMERGE Institu- tion	Transnational Activities Accessed
3 rd Call 4009	Screen printing and paste preparation based on Al 1%Si powder for flexible, wearable, self-powered sensor used as human respiration detector	IHTM (Serbia)	RTO	WUT	Industrial printing (TA3.3) Validation and standardization (TA4.2.)
3 rd Call 4077	FIB lamella preparation on photovoltaic cells for TEM observations	Fraunhofer IKTS (Germany)	RTO	UNOVA	Device metrology & characterization (TA4.1)

2.4 Proposals submitted to other TAs but requiring access to TA2 during implementation

From the beginning of EMERGE project to December 2023 (M30), eight proposals were initially submitted to different TAs, but also included materials synthesis and characterization activities (TA2) during the project execution. Three projects were carried out at RISE (5320, 5827 and 6421), two at HMU (5829 and 6198), two at TUD (6427 and 6473), and one at JOR (3986).

Table 5: List of projects originally submitted to other TAs but reassigned to TA2 during project execution.

Call/ Proposal ID	Proposal title	Home Institution (Country)	Type of Home Institution	EMERGE Institution	Original Transnational Ac- tivity Requested
3rd Call 3986	Design concept of a piezo-electric self-adhesive skin patch for wound healing applications	Beiersdorf AG (Germany)	Industry	JOR	Device Preparation (TA3.1.)
4th Call 5320	Photonic sintering of reverse offset printed structures	VTT (Finland)	RTO	RISE	Functional 2D & 3D printing (TA3.2) Device metrology & characterization (TA4.1)
4th Call 5827	Manufacturing and Characterization of Flexible Thermoelectric Modules and associated electronics through printing techniques	ParsNord Thermal Comfort ApS (Iran)	SME	RISE	Device Preparation (TA3.1.) Device metrology & characterization (TA4.1)
4th Call 5829	Lagrange-scale laser-printed bio-inspired metasurfaces for boosting the mechanics of flexible photonic and electronic devices	Empa, Swiss Federal Laboratories for Materials Science and Technology (Switzerland)	RTO	HMU	Functional 2D & 3D printing (TA3.2) Device Preparation (TA3.1.)
5th Call 6198	Functionalized MXene-based Halide Perovskite Solar Cells for Self-Power Electronics	Institut Català de Nanociència i Nanotecnologia (Spain)	RTO	HMU	Functional 2D & 3D printing (TA3.2) Device Preparation (TA3.1.)
5th Call 6421	Study of optical reduction of graphene oxide for photonic sensing applications	Indian Institute of Technology Bombay (India)	University	RISE	Prototype fabrication (TA3)
5th Call 6427	Dopant variation for controlled doping of the active layer in vacuum evaporated solar cells	Åbo Akademi University (Finland)	University	TUD	Theory (TA1) Characterization of prototypes and demonstrators (TA4)

6 th Call 6473	Printable Graphene-Conducting Polymer Inks for Wearable Sensor Innovations	Linköping University (Sweden)	University	TUD	Prototype fabrication (TA3) Characterization of prototypes and demonstrators (TA4) 44
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3. Summary of equipments used

A summary of all equipments used during the experiments carried out through material synthesis and characterization access provision up to M30 can be found on Table 6. The data was retrieved from each project timesheet provided by respective EMERGE Institutes TLOs. Further details about the equipments and methodologies related to TA2 offered by EMERGE host institutions can be found on deliverables D6.1 - First report on the proper methodologies for preparation and characterization of ink/paste out of different materials and D6.2 - Second report on the proper methodologies for preparation and characterization of inks/paste out of different organic/inorganic materials.

Table 6: List of equipments used for material synthesis and characterization access provision (TA2) up to M30, and respective UoA.

Call/ Proposal ID	Proposal title	EMERGE Institution	Originally submitted to TA2	Equipment used	TA2 UoA
1 st Call 3651	Screen printing of TiO ₂ / CeO ₂ powder mixture for oxygen sensors	WUT	Yes	Bruker Dektak XT Profilometer Rheometer Brookfiel	3
1 st Call 3652	Graphene-based electrically conductive coatings	HMU	Yes	Fisherbrand, Elmasonic S40H, S30H and S15H; Beckman Coulter Allegra X-22 Centrifuge; FungiLab, Viscolead adv; Ossilla Four-Point Probe	4
1 st Call 3662	Electrochemically exfoliated 2D materials for printed electronics	TUD	Yes	Dissolver, Ultrasonication, Centrifuge, Plasma oven, Spin coater, Spin coater, AFM, SEM, Viscometer, Tensiometer	2
2 nd Call 3856	Nanocoatings with antiviral properties on cement-based surfaces	UNOVA	Yes	SEM/EDS XPS Microwave synthesizer - CEM corporation Benchtop Nabertherm furnace UV-VIS spectrophotometer lambda 365+	13

				X-ray diffractometer (PANalytical's X'Pert PRO MRD)	
3rd Call 4001	Formulating screen printing ink/paste application for resistive heater application	WUT	Yes	Electrical measurement setup, thermal camera Rheometer Brookfiel Pastes/inks production line	3
3rd Call 4010	Thin and flexible electrodes for capacitance measurement	WUT	Yes	Digital Microscope Keyence Keithley 218A nanovoltmeter	0.38
3rd Call 4014	Bioresorbable Force Sensor using Biodegradable Piezoelectric Material	TUD	Yes	Buker D8 Discovery X-ray diffractometer	3
3rd Call 4015	Printed Bio-inspired Hydrogel based Substrate with Adhesiveness and Tailored Electrical Conductivity for Sustainable Electronics	TUD	Yes	Buker D8 Discovery X-ray diffractometer UV VIS Spectrometer	5
4th Call 4594	Insertion of ultrathin low-conductivity passivation layers in Perovskite solar cell	HMU	Yes	Atomic force microscope: Park XE7 Ambient pressure photoemission spectroscopy system: KP Technology APS04 Sheet-to-sheet pilot line: MBraun GB Centrifuge setups	4
4th Call 4682	Interface engineering in perovskite solar cells based on 2D materials	HMU	Yes	Atomic force microscope: Park XE7 Ambient pressure photoemission spectroscopy system: KP Technology APS04 Sheet-to-sheet pilot line: MBraun GB Centrifuge setups	8
4th Call 5549	Fabrication and electrical characterization on two-dimensional (2D) polymers-based flexible organic electronics	UNOVA	Yes	Benchtop Atomic Layer Deposition (ALD) - Beneq TFS 200 Optical Microscope	4
4th Call 5814	Screen-printed structures of alternating TiO ₂ and CeO ₂ layers used as an oxygen-sensing materials	RISE	Yes	Optical microscope Photonic sintering: Ceradrop F-Serie	0.9
4th Call 5816	Integration of ultrasonically exfoliated eco-friendly graphene into perovskite solar cell device fabrication	HMU	Yes	SHIMADZU UV Probe Spectrophotometer Van der Pauw: Ecopia HMS-3000 hall measurement system Atomic force microscope: Park XE7 Bruker Dektak XT Profilometer	7

				Ambient pressure photoemission spectroscopy system: KP Technology APS04 Hielscher UP200Ht ultrasonic tip	
4 th Call 5824	Thin film carbon nanotubes and membranes-based sensors development and characterization to enhance sensors sensitivity and selectivity	TUD	Yes	Scanning Electron Microscope Atomic Force Microscope & Profilometer Raman Spectroscopy	7
4 th Call 5826	2D/3D CELL-PILs for tissue engineering	WUT	Yes	Viscometer: Brookfields DV2T Pastes/inks production line Electrical measurement setup	2.4

4. Expected Scientific Impact of Concluded Projects

The completion of the user-projects led to interesting results that are conveniently being used by users to proceed with their research activities, while keeping collaboration with the EMERGE institutions, where these activities were carried out. Relevant information was collected from the “user report” survey that users must complete after conclusion of their TA projects. A brief description of the main achievements obtained during the research activities is provided below:

Proposal 3651: Screen printing of TiO₂/ CeO₂ powder mixture for oxygen sensors (WUT)

The main achievements obtained with the research activities developed at WUT are listed below:

- Screen printing TiO₂ and TiO₂/CeO₂ pastes on different substrates.
- Profile measurements for estimate of the layers thickness.
- Viscosity measurements of the pastes that were used for screen printing.

The proposers are working on a publication, where the data obtained during the implementation of the project in WUT will be included and EMERGE project will be acknowledged.

Proposal 3652: Graphene-based electrically conductive coatings (HMU)

Various graphene-based pastes were processed in form of films to be tested as electrodes in carbon-based solar cells. The carbon pastes were formulated in various solvents, with a major focus on alcoholic mixtures, to be compatible with the underlying perovskite and charge-transporting layers. Thickness and conductivity of the resulting graphene-based electrodes were evaluated as a function of the deposition techniques, systematically varying deposition parameters. In addition, laminable graphene-based electrodes were fabricated and applied to solar cells by means of a bench-top laminator at temperature lower than 120 °C to ensure the compatibility with the perovskite solar cell structure.

Proposal 3662: Electrochemically exfoliated 2D materials for printed electronics (TUD)

Regarding the main results obtained with the research activities developed during the project execution at TUD, the user reported the following:

- Learned how to use the printer to make electrode for electronic device;
- Grasped the utilization of 3D printing machine;
- Successful obtained the few-layered black phosphorus via the electrochemical exfoliation technique: compare with other exfoliated technique, such as liquid exfoliation and tape exfoliation, the electrochemically exfoliated sample with high-quality of few layers and high yield;
- Prepared the MnO₂ and MnO₂/Ag nano dispersions, the introduction of Ag nanoparticles can improve the electron conductivity of MnO₂;
- After the plasma treatment of ITO/PDMS substrate, the first try with nano graphene suspension get ideally printed electrodes as we want. However, the other samples don't work well like graphene suspension;
- The networking brought some new idea to improve the experiments.

Proposal 3856: Nanocoatings with antiviral properties on cement-based surfaces (UNOVA)

The main achievements obtained with the research activities developed at UNOVA are listed below:

- The users have Achieved a pure anatase form of TiO₂;

- Enhanced photocatalytic properties of the samples (80% reduction of Rhodamine B in 30 min under UV light);
- Gained an understanding on equipments such as XRD, XPS, and SEM.

Proposal 4001: Formulating screen printing ink/paste application for resistive heater application (WUT)

Regarding the main results obtained with the research activities developed in the WUT, the user reported the following:

- Achieved two different formulations based on various binder system - type L and M.
- Obtained 18%-20% graphene loading on both binder systems – type L paste which had good conductivity was chosen for the heater application.
- Simple square profile was printed on paper and foil – 2 layers ~10 µm thick.
 - Foil – 450 Ohm/sq
 - Paper – 350 Ohm/sq
- Demonstration of heating element reached more than 120°C without any degradation. Beyond 5 W, the substrates were damaged.

Proposal 4010: Thin and flexible electrodes for capacitance measurement (WUT)

The electrodes were successfully designed and produced through the screen-printing process. The following limitations on using the electrodes for biomedical applications were found:

- Due to the lack of firm adhesion of commercially available simple electrodes to the abdomen, their angle can change with even small movements of the user, and they are significantly affected by accumulated sweat. Custom electrodes designed for stimulation purposes can also heat up when worn for extended periods. This can lead to interference from different layers of tissues and disturbances in the capacitance value and correlation of impedance and voltage signals. The project has identified limitations in the current performance of these electrodes.
- Printed electronics methods offer a low-cost and effective means of producing user-friendly electrodes. With a thickness of approximately one hundred micrometers, these electrodes will be almost imperceptible during all-day use.

- The biodegradable materials, thin layer construction, exact size and optimal placement will reduce unexpected affects by skin, moisture and heating. We will conduct performing test in the laboratory setting and a small number of field test.

Proposal 4014: Bioresorbable Force Sensor using Biodegradable Piezoelectric Material (TUD)

By employing Grazing Incidence Wide-Angle X-ray Scattering (GIWAXS), a precise quantification of the alteration in crystalline content at various processing ratios was accomplished. Additionally, a comprehensive investigation of the phase transitions was conducted. The utilization of GIWAXS enabled a meticulous analysis of the crystalline structures present in the samples, providing valuable insights into the effects of different processing conditions on the resulting material properties. The obtained data not only allowed for a quantitative assessment of the changes in crystalline content, but also facilitated a thorough examination of the phase changes occurring throughout the post-processing stages. This advanced characterization technique played a crucial role in the comprehensive understanding of the materials' structural transformations, and contributes valuable information to the existing scientific literature, aiding future research in the field.

Proposal 4015: Printed Bio-inspired Hydrogel based Substrate with Adhesiveness and Tailored Electrical Conductivity for Sustainable Electronics (TUD)

Regarding the main results obtained with the research activities developed in the TUD, the user reported the following:

- The user got an understanding about the interactions between different molecules and behaviour of prepared polymer through XRD.
- Based on the results, the user will fabricate 3D printed sensors such as temperature sensor or pH sensor.

Proposal 4594: Insertion of ultrathin low-conductivity passivation layers in Perovskite solar cell (HMU)

The main achievements obtained with the research activities developed at HMU are listed below:

- Sample preparation for the experiment
 - Prepared the h-BN ink for backup;
 - Prepared spray coating h-BN samples over ITO/PTAA substrates and ITO/PCBM substrates;
 - Prepared Liquid-air assembling coating h-BN samples over ITO/PTAA substrates.
- Characterization of the samples
 - AFM analysis over the samples to see the thickness, area, and distribution of the h-BN.
- Data analysis
 - Figure out the future direction to modification the coating and ink preparation.
- Demonstration of solar cell manufactory process
- Demonstration of automatic spray coating process
- Substrates preparation for future experiments
 - Prepared ITO/PTAA and ITO/SAM substrate for future experiments.
- Ensure the characterization needed for publication.

Proposal 4682 Interface engineering in perovskite solar cells based on 2D materials (HMU)

The main achievements obtained with the research activities developed at HMU are listed below:

- Discussed the manuscript plan (finished the characterization of SEM, PL, AFM, Raman at the cell level), identify the most important thing – bandgap values of Ti_2C MXenes; work function of MXenes to be done.
- Obtained the sample of a perovskite layer on glass for Raman Spectroscopy.
- Obtained a better understanding of the fabrication process of the perovskite solar cells, by spin coating and key parameters.

Proposal 5549 Fabrication and electrical characterization on two-dimensional (2D) polymers-based flexible organic electronics (UNOVA)

The main impact of this work is the production of the 2D polymer-based transistor and the characterization of their electrical performance under strain as flexible electronics. This will allow the complete investigation and analysis of mechanical response on 2D polymers with deformation. More importantly, the failure mechanism of 2D polymers can be unveiled to improve and rationalize the synthesis strategies of reliable 2D polymers. These outcomes would vastly improve the knowledge on flexible devices based on 2D polymers.

Proposal 5814 Fabrication and electrical characterization on two-dimensional (2D) polymers-based flexible organic electronics (RISE)

Regarding the main results obtained with the research activities developed in the RISE, the user reported the following:

- Screen printing of CeO₂ and TiO₂ on commercial Dropsense ceramic substrates with pre-patterned gold IDE electrodes and platinum heater.
- Both inks were printed, either in the form of the stripes next to each other OR one over the other using Horizon DEK II screen printer.
- Four layers of each ink were printed to obtain thicker films.
- Thickness of two-layer TiO₂ was found to be approximately 1.5 μm.
- The sensors were heated at 120 °C before printing each subsequent layer for oxygen sensing application.
- As an exploratory activity, some sensors were investigated for photonic sintering using Ceradrop – Pulseforge photonic sintering system. Several pulse patterns and pulse energies were investigated.
- One of the sensors was tested after photonic sintering for desired conductivity, mimicking the actual measurement protocol used by users in home labs. The resistance was found to be around 30 to 40 MOhm while platinum heater was sourced with 8 V, approx. 0.7A current.
- LASER cutting of hard cardboard frame was carried out to align the DropSense sensors on printing board, allowing well aligned printing patterns.

- Further investigation of oxygen sensing capability, chemical properties and morphological information, of printed sensors and printed + photonic sintered sensors will be carried out by users in their home lab.

Proposal 5816 Integration of ultrasonically exfoliated eco-friendly graphene into perovskite solar cell device fabrication (HMU)

The users have produced graphene inks (0.5-1 mg/ml) in water and alcohol mixes in Oxford Brookes University (OBU) followed by their fabrication of spray-coated and slot-die coated films on both glass and flexible PET substrates in HMU, Greece. From the obtained preliminary results, it has been found that spray-coated transparent graphene films (20 mm×15 mm, thickness ~ 70 nm) prepared from graphene inks in water-IPA solvent demonstrated promising electric conductivity of 16 S/m and electron mobility of 3×10^2 m²/V.s without annealing. In addition, films (20 mm×15 mm, thicknesses 200-350 nm) of electric conductivity 0.60-0.80 S/m and electron mobility ~ 1.5×10^2 m²/V.s were also prepared using slot-die coating method (refer to Objective-1; Tasks 1, 2 in EMERGE-2023). Based on these properties, the obtained films would theoretically be a very good candidate as an interlayer on top of the electron transport layer (ETL) in both Perovskite Solar Cell (PSCs) & Organic Photovoltaics (OPVs). However, the users still have unfinished work plans 3-6 from previous EMERGE-2023 which are mainly focussed on producing highly concentrated graphene inks (at least 2 mg/ml) and pastes for their film fabrication to be tested for the construction of perovskite solar cell devices. Additionally, graphene based developed pastes can be benchmarked against in-house commercial carbon pastes and graphene hybrids in solar cell device geometry.

The users plan to submit a new proposal during 2024 to complete the unfinished experiments and finalize the data, after which an article will be submitted to a high-impact journal.

Proposal 5824 Thin film carbon nanotubes and membranes-based sensors development and characterization to enhance sensors sensitivity and selectivity (TUD)

The users have effectively analysed the surface characteristics of the spray-coated carbon nanotube (CNT) film. They also enhanced the understanding on the quality of the spray

coated carbon nanotube process performed at the sensing technologies laboratories. The CNTs film surface morphology, topography, and structural features are critical factors for quality control in applications like sensor development, where performance and reliability are paramount. These analyses not only help the users to enhance our carbon nanotube film fabrication processes at UNIBZ but also pave the way for more efficient and effective sensors, electronic devices, and other applications reliant on the unique properties of carbon nanotubes.

The users are currently including the results of the experimental worked performed during this EMERGE experience on an article entitled “Highly sensitive and selective ammonia detection through PDMS-coated printed carbon nanotube chemiresistive sensors”.

Proposal 5826 2D/3D CELL-PILs for tissue engineering (WUT)

The studies allowed the users to obtain stable inks with adequate viscosity for 3D printing. The tuning of the printing parameters enabled the 3D printing of hydrogels from alginate, methacrylated gelatine, and cellulose nanofibers inks. These inks were studied in the absence and presence of PILs, and a methacrylated mucin. Several cross-linking agents and methods were used. Finally, the hydrogels were freeze-dried to obtain the respective scaffolds.