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NEWSLETTER

DAM4CO_2

Double-Active Membranes for a sustainable CO_2 cycle

Partners:

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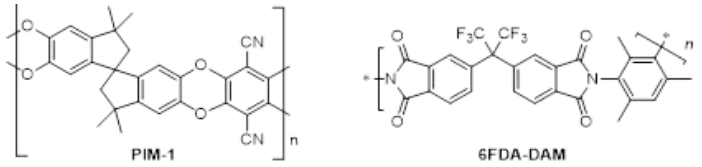
ABOUT DAM4CO2 DELIVERABLES

Deliverable 2.1

This deliverable is the first for WP2 (Synthesis) of the DAM4CO$_2$ project and reports on the preparation of the 1st generation of both soluble and insoluble Polymers of Intrinsic Microporosity (PIMs) and Metal-Organic Frameworks (MOFs) for carbon capture and storage, due in the 10th month of the project.
It focuses on Task 2.1, which aims at the "Synthesis of high-performance membrane-forming polymers (Matrix)", Task 2.2 "Synthesis of dispersible insoluble polymers (Fillers)", and Task 2.3 "Synthesis of Metal-Organic Frameworks (MOFs, fillers)". The lead partner for WP2 is Swansea University (USwan), with the University of Edinburgh (UEdin) responsible for preparing the soluble and insoluble PIMs, and INSTM (Universities of Pisa and Perugia) preparing the MOFs.

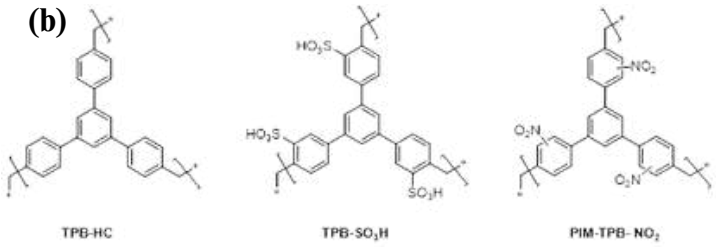
The main objective of the initial synthesis was to produce significant amounts of soluble first-generation polymers, such as polybenzodioxin **PIM-1** and polyimide **6FDA-DAM**, which were synthesised on a multi-gram scale by USwan and UEdin. USwan also prepared six different insoluble PIMs, three based on highly porous triphenylbenzene (**TPB**) and three on ultra-microporous triptycene (**TRIP**). All were functionalised with polar moieties such as nitro and sulfonic groups. These insoluble polymers were used as "fillers" in combination with soluble PIMs (matrix) to prepare the first Mixed Matrix Membranes (**MMMs**) of the project.

(a)



Soluble PIMs "matrix"

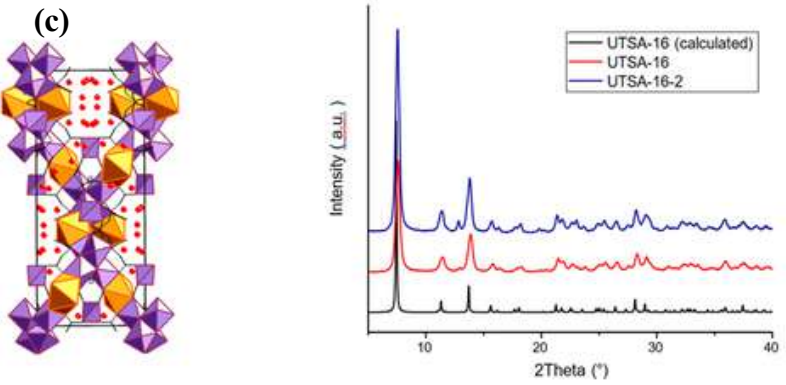
(b)



Insoluble PIMs "fillers"

The INSTM group prepared a series of MOFs with different geometries that, in combination with soluble PIMs, were used to form novel organic-inorganic MMMs. Specifically, universities of **Pisa** and **Perugia** provided MOFs such as Ca−trimesate, Ca−squarate (**UTSA−280**), Zr−squarate nanocrystalline powder, Zn−citrate **(UTSA−16)**, Zirconium fumarate (MIP-203), Zirconium fumarate (**MOF-801**), Zirconium succinate (**MIP-203**), and Zirconium malate. All of them were synthesised using "**green**" methods and non-critical raw materials.
All polymers and MOFs were characterized using basic techniques, which are further detailed in WP3.

(c)



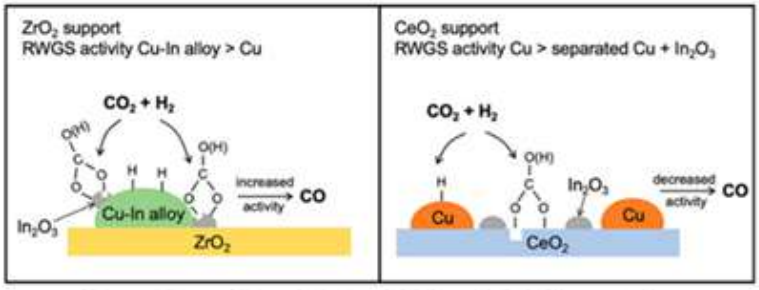
(c) MOFs "fillers"

Deliverable 2.2

This deliverable, the second for WP2 (Synthesis) of the DAM4CO$_2$ project, details the preparation of the first generation of photocatalysts. It focuses on Task 2.4, "Synthesis of 1st and 2nd Generation Photocatalysts", conducted by the Technical University of Valencia (UPV-ITQ).

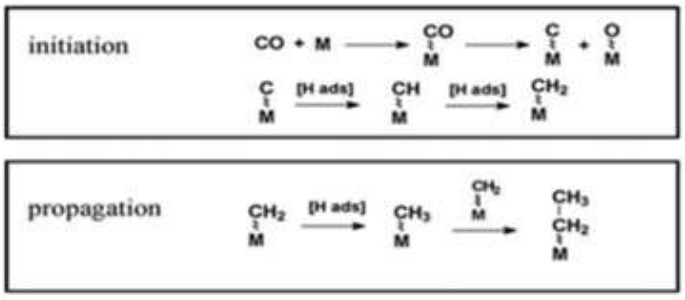
The primary goal is to convert CO_2, captured and separated using mixed matrix membranes (MMMs) prepared by materials produced in Tasks 2.1, 2.2, and 2.3, into high value-added compounds, i.e. hydrocarbons with relatively long chains. Transforming CO_2 into fractions heavier than C_2 involves catalysing two consecutive reactions: the reverse water gas shift (RWGS) and Fischer-Tropsch (FT). Therefore, a crucial aspect of DAM4CO$_2$ is the synthesis of photocatalysts that can facilitate these reactions. Initially, the project planned to synthesise first-generation photothermocatalysts active in these reactions separately, followed by second-generation bifunctional photothermocatalysts.

a)



$$CO_2 + H_2 \rightleftharpoons CO + H_2O \quad \Delta H^\circ = 42.1\ \text{Kj}\cdot\text{mol}^{-1}$$

b)



$$n\text{CO} + (2n+1)\text{H}_2 \rightleftharpoons \text{C}_n\text{H}_{2n+2} + n\text{H}_2\text{O} \quad \Delta H<0$$

a) *Example of photothermal catalyst for RWGS reaction based on copper. Equation 1: Chemical equation of the RWGS reaction.*

b) *Classic mechanism pathway of FT reaction. Equation 2: Chemical equation of FT reaction.*

The first-generation photothermal catalysts synthesised by the UPV-ITQ team are categorised into two groups based on their application in RWGS and FT reactions:

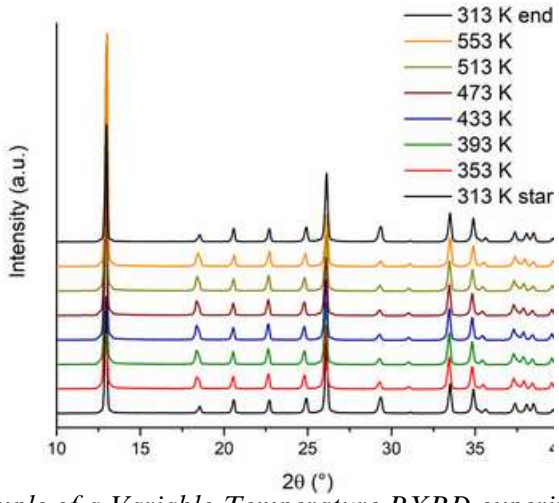
Reverse Water Gas Shift (RWGS) Catalysts:
UPV-Ca-HAP_001: Prepared via co-precipitation using $Ca(NO_3)_2{\cdot}4H_2O$.UPV-Ru-HAP_001: Prepared via co-precipitation with 10% mol substitution of calcium by ruthenium in calcium hydroxyapatite (Ca-HAP1). UPV-Co-HAP_001: Prepared with 10% mol substitution of calcium using $Co(NO_3)_2{\cdot}6H_2O$.

Fischer-Tropsch (FT) Photocatalysts:
Standard (Ni/NiO) - Al_2O_3 - SiO_2 (Commercial): Purchased to compare the photocatalytic activity with ITQ-UPV synthesised materials.NiAl-LDH-Ox: Prepared by mixing $Ni(NO_3)_2{\cdot}\ 6H_2O$, $Al(NO_3)_3 \cdot 9H_2O$, and urea in deionised water, with modifications in amounts and temperatures by UPV-ITQ team. NiTiAl-LDH-MOxide (MMM463): A layered mixed metal oxide with a metal ratio of 4:0.5:0.5 for nickel, titanium, and aluminium.

Deliverable 3.1

Deliverable D3.1 defines the protocols of characterization, derived from the experimental activities of Tasks from 3.1a to 3.1f carried out in the first 9 months of the project by consortium members from INSTM, USwan, UPV-ITQ and CNR.

The characterization step is essential to rationally drive the choice of the best materials to be employed in the preparation of the MMMs used for CO_2 capture and conversion. For these reasons, it is mandatory to plan a protocol for basic and advanced characterization that could become a milestone in the pristine materials characterization that could be routinely applied to the following generations of materials, to help and speed up the selection of the best fillers and matrices for MMMs.

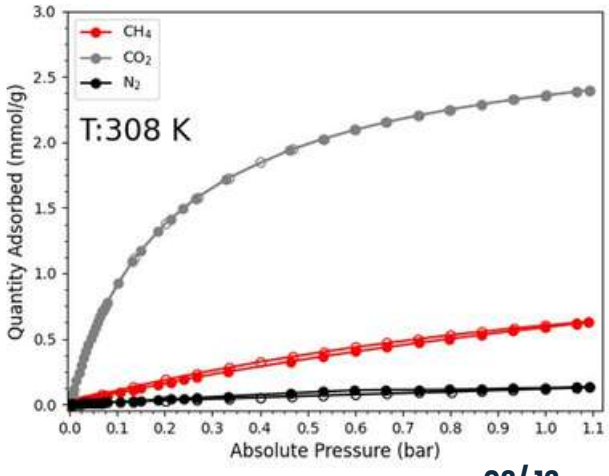


Example of a Variable-Temperature PXRD experiment

This deliverable starts from a basic characterization step to assess the success of the syntheses, and the investigation of materials properties evolves towards a multi-technique characterization approach to shed light on the physico-chemical features and on the adsorption/catalytic properties of the materials. For instance, the thermal stability of MOFs is evaluated monitoring the diffraction profile as a function of temperature to assess the possible occurrence of phase transitions induced by loss of solvents or the loss of crystallinity upon framework collapse. CO_2 adsorption/desorption experiments at different temperatures are employed to quantitatively investigate the capacity of MOFs and polymers to adsorb CO_2 and to explore the affinity of fillers towards N_2 and CH_4 , to assess their selectivity towards the CO_2 molecule.

Information on the structural and dynamic properties at atomic level, as well as on MOF-CO_2 and polymer-CO_2 interactions and CO_2 status within material are acquired by SSNMR measurement using activated or CO_2 -loaded samples.

Photocatalysts synthesized in WP2 are characterized to disclose their surface properties, and to correlate them with their catalytic activity. For instance, in-situ IR spectroscopy with probe molecules provides an overview of the different sites present on the catalyst surface, i.e. it could be adopted to investigate the presence of rather small differences in their acid strength. On the other side, the adsorption of CO_2 at room temperature (or at the temperature of the reaction) followed with IR gives information about the way the molecule interacts with the catalysts' active sites, undergoing activation, possibly through the formation of carbonate-like species. Another essential step to drive the choice of the best photocatalysts to produce MMMs for CO_2 conversion is the evaluation of their redox and electronic properties. This is done by a combination of different techniques such as: X-Ray Photoelectron spectroscopy (XPS) and in situ XPS, Cyclovoltammetry (CV), Temperature programmed reduction (TPR), Transient Absorption Spectroscopy (TAS). SSNMR can be applied also in the case of advanced characterization of the catalysts, and the characterization protocol will be modified according to the NMR-active nuclei and paramagnetic metal present in the catalyst sample.

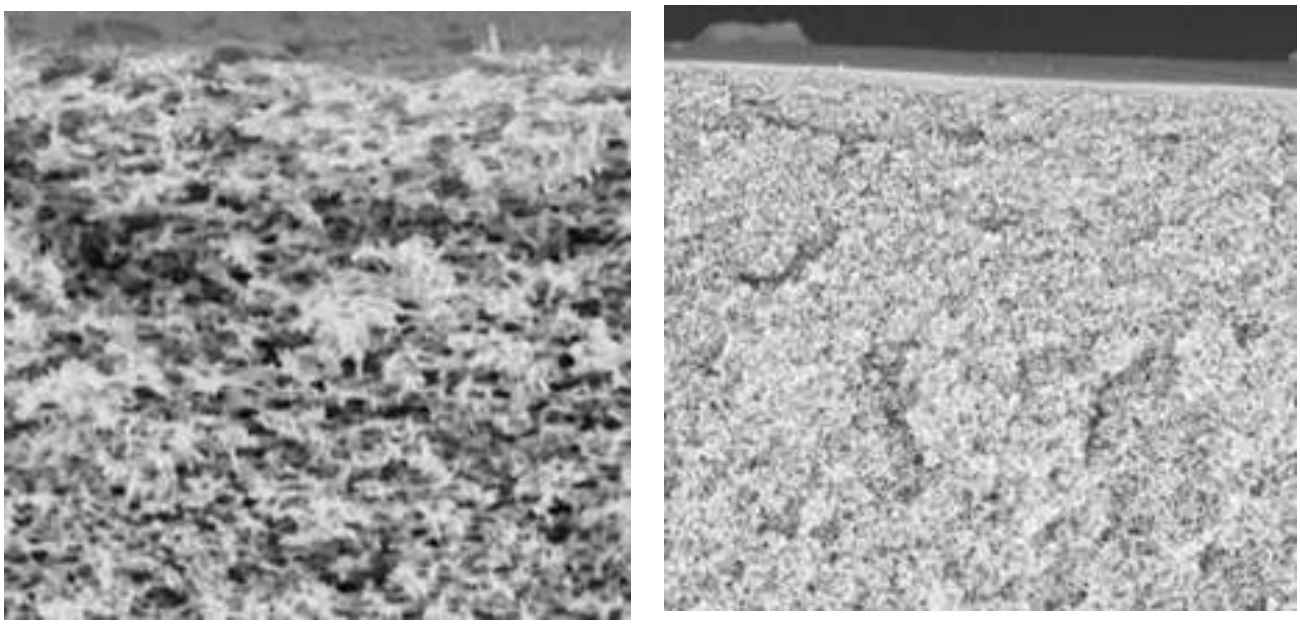


Deliverable 4.1

Major progress was made at CNR in the screening of the optimum conditions for the preparation of polymeric membranes for use as the support of our Double Active Membranes. After a first selection of a broad series of potentially interesting green polymers and green solvents, via a series of solubility tests, the most promising polymer/solvent combinations with the potential to produce suitable membranes via the phase inversion process with water as the nonsolvent were identified.

The solvents included a selection of green chemicals such as Acetyl-tributyl citrate (ATBC), Cyrene (Dihydrolevoglucosenone), Cyrene 2-MeTHF blend, Dimethyl carbonate (DMC), Dimethyl Isosorbide (DMI), Methyl sulfoxide (DMSO), γ-Valerolactone (GVL), Triethyl phosphate (TEP), Trimethyl phosphate (TMP) and these were compared with more traditional solvents N,N dimethylacetammide (DMA), N,N dimethylformammide (DMF), 1-methyl-2 pyrrolidone (NMP).

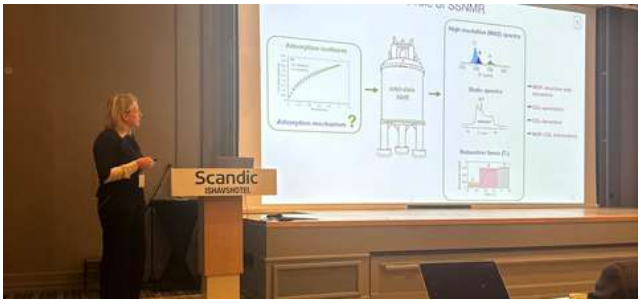
The PHA (polyhydroxyalkanoate) and PHB (polyhydroxybutyrate) green polymers-based solutions were most difficult to handle due to the strong tendency to gelation at lower temperatures in these solvents, and phase inversion also resulted in mostly in irregular structures.

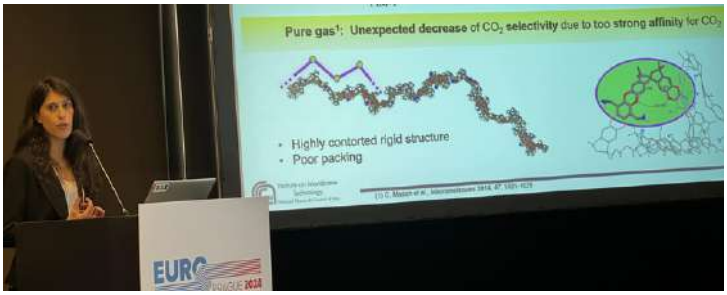


Sponge-like morphology produced from Matrimid in γ-Valerolactone (left) and P84 in DMSO (right) upon coagulation in water

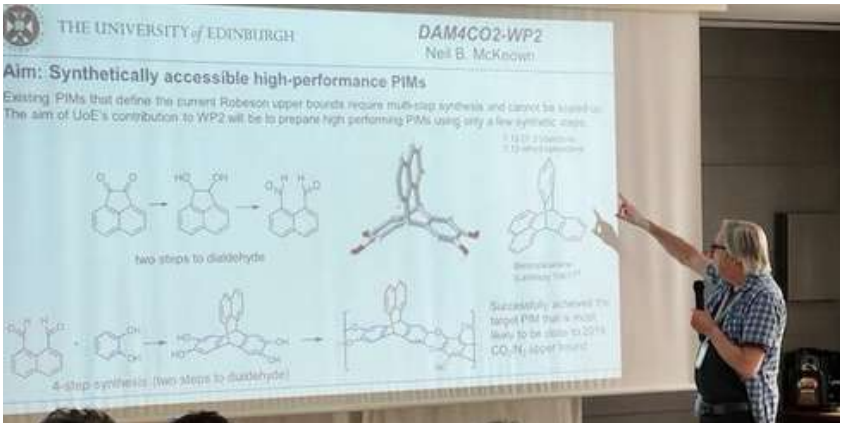
Instead, some traditional polymers successfully yielded highly spongy membrane morphologies, for instance the Matrimid solution in γ-Valerolactone, which are most likely to be suitable for supporting the catalyst and CO_2 - selective membranes. The latter polymers also have the advantage that they can be cross-linked to provide increased thermal and chemical stability. The use of pore-forming additives such as polyvinylpyrrolidone was also studied to further tailor the membrane properties. Permeability measurements and the analysis of the pore size distribution are still in progress, while the first experiments to form thin film composite membranes are planned and approaches to extend this work to hollow fibre membranes are under investigation.

- "Workshop on renewable energy and catalysis", Perugia – Italy, 3th May 2024.
- XIV INSTM Conference on Materials Science and Technology, Cagliari - Italy, 9th - 12th June 2024.
- 39 EMS Summer School 2024, Louvain-la-Neuve, Belgium, from June 10th - 14th 2024.
- "Polymers of Intrinsic Microporosity (PIMs): versatile materials for gas separation and catalysis", CNRS in Dijon (France), 28th June 2024.
- EUROMAR – The 20th European Magnetic Resonance Congress, Bilbao (Spain) 30th June - 4th July 2024.
- 9th international conference on "Metal-Organic Frameworks and open frameworks compounds", Singapore, 15th - 19th July 2024.
- 16th Magnetic Resonance in Porous Media Conference, Tromso (Norway), 26th - 30th August 2024
- SCI 2024 Chemistry: Elements of future. XXVIII National Congress of Società Chimica Italiana, Milano – Italy, 26th - 30th August 2024.
- 51st National Conference on Magnetic Resonance, Florence (Italy), 4th - 6th September 2024.
- Euromembrane 2024, Prague, 8th - 12th September 2024.
- Polycondensation 2024, Lyon 15th - 18th September





2° project meeting



The 6 months DAM4CO$_2$ meeting was held on May 8th - 9th, in Valencia, Spain, organized by Primalchit. The meeting was a hybrid event to allow also the participation of the consortium member that were not able to physically reach the location for personal or business reasons.

The first day was devoted to the discussion on Materials preparation and characterization, while the second day was more focused on their implementation as membranes and on the modelling tools that are helping in the development of the DAM4CO$_2$ concept.

Primalchit also organized a team-building activity with a visit to the Albufera Natural Park, to glue the consortium members.

People hired on the project

Dr. Kamran Ghasemzadeh, who holds a Ph.D. in Chemical Engineering, is a highly accomplished researcher and author. He has published 70 peer-reviewed papers, 8 books, and 40 book chapters with prestigious international publishers. His primary research expertise is in the areas of membranes and membrane reactors, where his work has significantly advanced both knowledge and practical applications in the field of chemical engineering. Since 2015, Dr. Ghasemzadeh served as an Associate Professor in the Department of Chemical Engineering at Urmia University of Technology in Iran. Simultaneously, between August 2022 and 2024, he held the position of Senior Research Associate at the Manchester University, where he contributed to several cutting-edge research projects.
Currently, Dr. Ghasemzadeh is a Senior Research Associate at the Institute for Materials and Process at the University of Edinburgh, where his research continues to drive innovation in membrane technology in the EIC $DAM4CO_2$ project.

Carmen Rizzuto, got her Ph.D. in Physical, Chemical, and Materials Sciences and Technologies PON R&I Scholarships for the industrial characterization of the theme at the University of Calabria in June 2022.
Since October 2022, she has been involved in the Prin-doMino 2022 project, and she focused on the preparation, characterization, and modeling of efficiently Mixed Matrix Membranes based on (per-)fluorinated metal-organic frameworks for the treatment of large amounts of CO_2 under the scientific supervision of Dr. Alessio Fuoco and Dr. Elena Tocci. In October 2024, she joined the European project "Double Active Membranes for a Sustainable CO_2 Cycle" ($DAM4CO_2$), supervised by Dr. Alessio Fuoco and Dr. Elena Tocci. Nowadays, she is focused on preparing, characterizing, and modeling membranes for CO_2 capture and catalysis.

Dr. Giulio Bresciani received his Master's degree in Chemistry from the University of Pisa in 2015. In 2016, he was admitted to the Doctoral School in Chemistry and Material Science at the same university. During his PhD, he spent six months as an exchange student in Prof. Timo Repo's group at the University of Helsinki. He completed his PhD in 2020 under the supervision of Prof. Guido Pampaloni, focusing on the synthesis of novel metal carbamates and their catalytic applications in CO_2 activation. Dr. Bresciani worked for 18 months on the "doMino" project (PRIN2020) as a research fellow. Since May 2024, he has been part of the European project "Double Active Membranes for a Sustainable CO_2 Cycle" ($DAM4CO_2$), under the supervision of Prof. Marco Taddei and Prof. Marco Lessi. His research interests center on the synthesis and characterization of metal-organic frameworks (MOFs), with a particular focus on X-ray diffraction techniques.

People hired on the project

Dr. John Tobin , obtained his PhD in polymer and organic chemistry at Heriot-Watt University in 2018, focusing on the development of conjugated porous polymers as heterogeneous photocatalysts under continuous flow conditions. Following this, he was involved in several industrial projects funded through OGIC (Oil & Gas Innovation Centre) to develop new functional materials to help improve the health, safety, and environmental protections required on deep sea oil rigs. In June 2019, John joined the McKeown group in the School of Chemistry at University of Edinburgh to continue development of microporous polymer membranes for gas separation applications. This work has focused on the synthesis of novel monomers and polymers, characterisation, and determining routes for upscaling procedures. Since August 2024, he has continued and expanded this work through "Double Active Membranes for a Sustainable CO_2 Cycle" (DAM4CO_2), supervised by Prof. Neil McKeown.

Communication activities

Destination Terra 2100 - DT2100

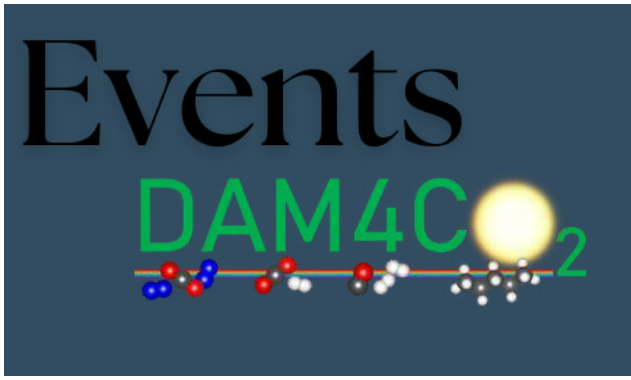
On September 20th, 2024, Valentina Crocella and Matteo Signorile (INSTM unit) participated as speakers to the dissemination event “Destination Terra 2100 - DT2100” (Sangano, Italy). The event involved a non-specialized audience of ca. 50 participants. They introduced the overarching issues in carbon capture technologies and conversion, and how these are addressed within DAM4CO_2 .

EU - Researchers Night

On 27th September 2024 we took part in the SuperScienceMe – Research is your Elevation event. We have been active by showing the scope and preliminary results of DAM4CO_2 in the EU Space. We also foreseen our engagement in the “Researchers at Schools” activities also for the next years, to further communicate the DAM4CO_2 results and to promote the scientific culture at high school level by usinf the “Citizen Science” approach.

Accademia dei Lincei

On October 3rd and 4th, The Accademia Nazionale dei Lincei, one of the oldest Italian cultural institutions, organized a conference on “Synthetic Fuel”. Silvia Bordiga, a member of the DAM4CO_2’s INSTM unit, acted as a co-chair. Marco Taddei (INSTM), Carmen Rizzuto (CNR), and Alessio Fuoco (CNR) participate to the event in different moments, also presenting the DAM4CO_2 activities. The event was broadcasted at national scale on Rai Cultura, the cultural division of RAI, the national public broadcasting company of Italy.



On June 19th, a workshop titled "**Towards Carbon Neutrality - New frontiers in carbon dioxide capture and valorization**" took place in Torino, focusing on carbon dioxide (CO_2) capture and reuse. Organized by researchers from the University of Turin's Department of Chemistry and supported by the NIS (Nanostructured Interfaces and Surfaces) Interdepartmental Center, the event represents a relevant dissemination action in the context of DAM4CO_2.

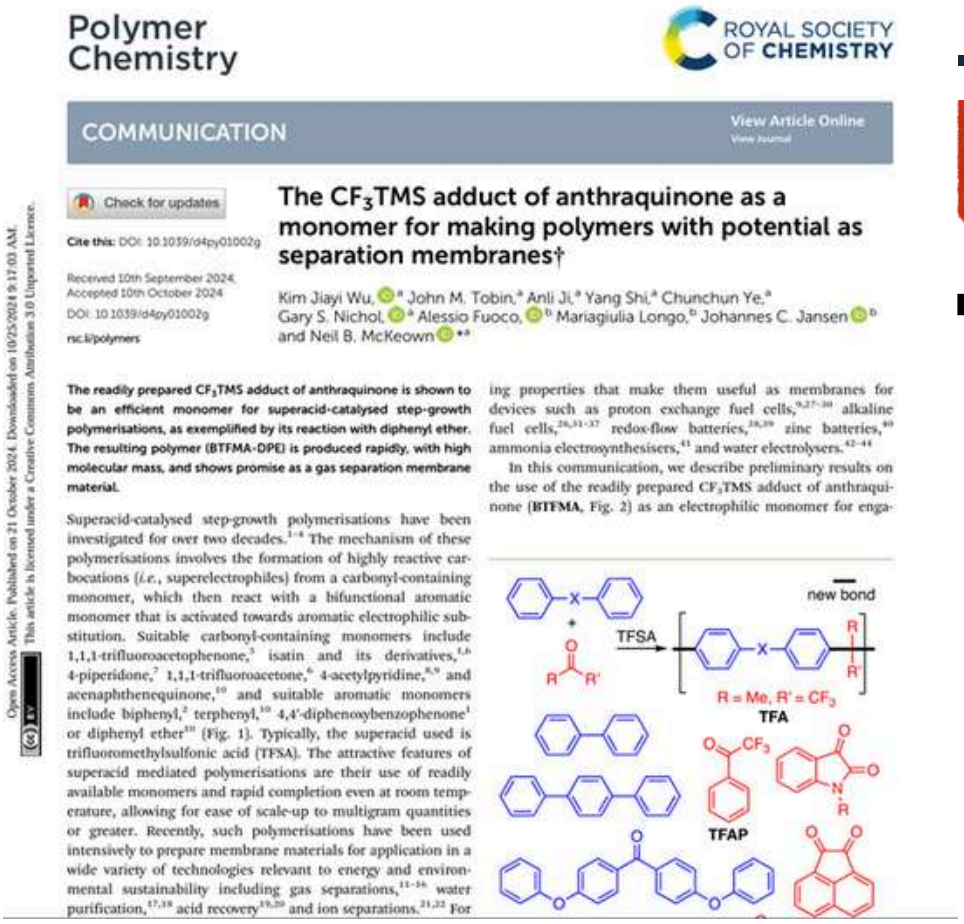
The workshop gathered 130 participants from academia and industry, including PhD students, and featured 16 international speakers who addressed CO_2 management from various perspectives, including research, engineering, and regulatory aspects. The opening remarks were delivered by Prof. Gabriele Ricchiardi, NIS President, and Dr. Alessio Fuoco, the main coordinator of the DAM4CO_2 project. Dr. Francesco Matteucci from the EIC introduced funding opportunities and the council's initiatives.

Participants and some of the speakers of the workshop.

The first scientific session began with Dr. Alexis Dunan from Carbon Gap LTD, who provided insights into current and future European CO_2 emission legislation, emphasizing the growing role of regulation in CO_2 capture processes. Prof. Susana Garcia from Heriot-Watt University discussed the "PrISMa" project, which integrates chemistry, materials science, and data science to create CO_2 capture materials. Virginia Guiotto (University of Turin) presented the progress on developing perfluorinated Metal-Organic Frameworks (MOFs) for CO_2 separation using Mixed Matrix Membranes (MMM).The second session highlighted various innovative approaches to CO_2 capture and energy storage. Dr. Alexander Forse from the University of Cambridge discussed using electrochemical processes to modify carbon materials for CO_2 capture. Prof. Matteo Romano (Polilteсnico di Milano) presented the "Cleanker" project, which implemented a pilot plant for carbon looping in cement production. Prof. Angiolina Comotti (Università degli studi di Milano Bicocca) showcased advanced synthesis and characterization of MOFs for CO_2 capture. Dr. Sergio Bocchini, from Politecnico di Torino, discussed the advantages of using ionic liquids for CO_2 capture over traditional amine-based solutions.

The third session, focused on MMM's role in CO_2 separation and conversion, started with Prof. Rocio Semino (Sorbonne Universitè) illustrating the use of simulations to understand interface phenomena in polymer-based MMMs containing MOFs. Dr. Carmen Rizzuto from CNR-ITM discussed experimental and computational aspects of using perfluorinated MOFs in MMMs. Dr. Grazia Bezzu (Swansea University) explored the development of microporous polymers for CO_2 capture, while Dr. Adele Brunetti (CNR-ITM) presented the use of MMM-based reactors containing carbon nitride for photoreduction of CO_2 into synthetic fuels.

The final session covered CO_2 conversion methods. Prof. Roberto Gobetto (University of Turin) introduced recent results in electrochemical CO_2 conversion using Re- and Mn-based catalysts. Dr. Nataly Carolina Rosero (Instituto de Cerámica y Vidrio - CSIC) presented the use of hydrotalcite-based electrocatalysts for CO_2 reduction. Prof. Francesca Valetti (University of Turin) discussed biological CO_2 capture and conversion processes, highlighting microbial metabolism as an alternative to photosynthesis. Prof. Harash Manyar (Queen's University Belfast) reviewed various methods for converting CO_2 into high-value chemicals. The event was concluded by the talk of Prof. Berend Smit (EPFL), exploring the use of generative AI in the "PrIsMa" project to design new CO_2 capture materials.



Polymer Chemistry

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rsc.li/polymers

The CF_3TMS adduct of anthraquinone as a monomer for making polymers with potential as separation membranes†

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The readily prepared CF_3TMS adduct of anthraquinone is shown to be an efficient monomer for superacid-catalysed step-growth polymerisations, as exemplified by its reaction with diphenyl ether. The resulting polymer (BTFMA-DPE) is produced rapidly, with high molecular mass, and shows promise as a gas separation membrane material.

Superacid-catalysed step-growth polymerisations have been investigated for over two decades.[1–4] The mechanism of these polymerisations involves the formation of highly reactive carbocations (*i.e.*, superelectrophiles) from a carbonyl-containing monomer, which then react with a bifunctional aromatic monomer that is activated towards aromatic electrophilic substitution. Suitable carbonyl-containing monomers include 1,1,1-trifluoroacetophenone,[5] isatin and its derivatives,[5,6] 4-piperidone,[7] 1,1,1-trifluoroacetone,[6] 4-acetylpyridine,[8,9] and acenaphthenequinone,[10] and suitable aromatic monomers include biphenyl,[2] terphenyl,[10] 4,4′-diphenoxybenzophenone[1] or diphenyl ether[10] (Fig. 1). Typically, the superacid used is trifluoromethylsulfonic acid (TFSA). The attractive features of superacid mediated polymerisations are their use of readily available monomers and rapid completion even at room temperature, allowing for ease of scale-up to multigram quantities or greater. Recently, such polymerisations have been used intensively to prepare membrane materials for application in a wide variety of technologies relevant to energy and environmental sustainability including gas separations,[11–16] water purification,[17,18] acid recovery[19,20] and ion separations.[21,22] For ing properties that make them useful as membranes for devices such as proton exchange fuel cells,[9,27–30] alkaline fuel cells,[26,31–37] redox-flow batteries,[38,39] zinc batteries,[40] ammonia electrosynthesisers,[41] and water electrolysers.[42–44]

In this communication, we describe preliminary results on the use of the readily prepared CF_3TMS adduct of anthraquinone (**BTFMA**, Fig. 2) as an electrophilic monomer for enga-

https://doi.org/10.1039/D4PY01002G

This article is Open Access

PRESS RELEASE

In an interview about how "deep tech" turns carbon dioxide and nitrogen compounds into assets, Alessio Fuoco talked about the DAM4CO$_2$ concept. Our coordinator shared how Double Active Membranes can be useful to produce renewable fuels through the conversion of CO_2 emissions directly at the sources.

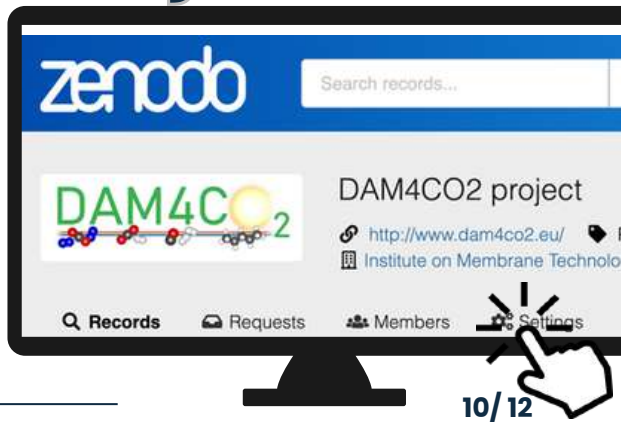
Read the full articles on **Deepsync** and **Youris**.

DEEPSYNC

youris.com
EUROPEAN RESEARCH MEDIA CENTER

Good News! We have a repository

We have enstablished the DAM4CO$_2$ community on Zenodo.
Zenodo is an open repository maintained by CERN and it is compliant with the data management requirements of Horizon Europe and EIC calls. It is supported by the European Commission to help the Open Data policy and a FAIR data management. By using Zenodo, the DAM4CO$_2$ consortium will help in the advancement of the FAIR principles of findability, accessibility, interoperability, and reusability in the field of carbon management and valorisation.



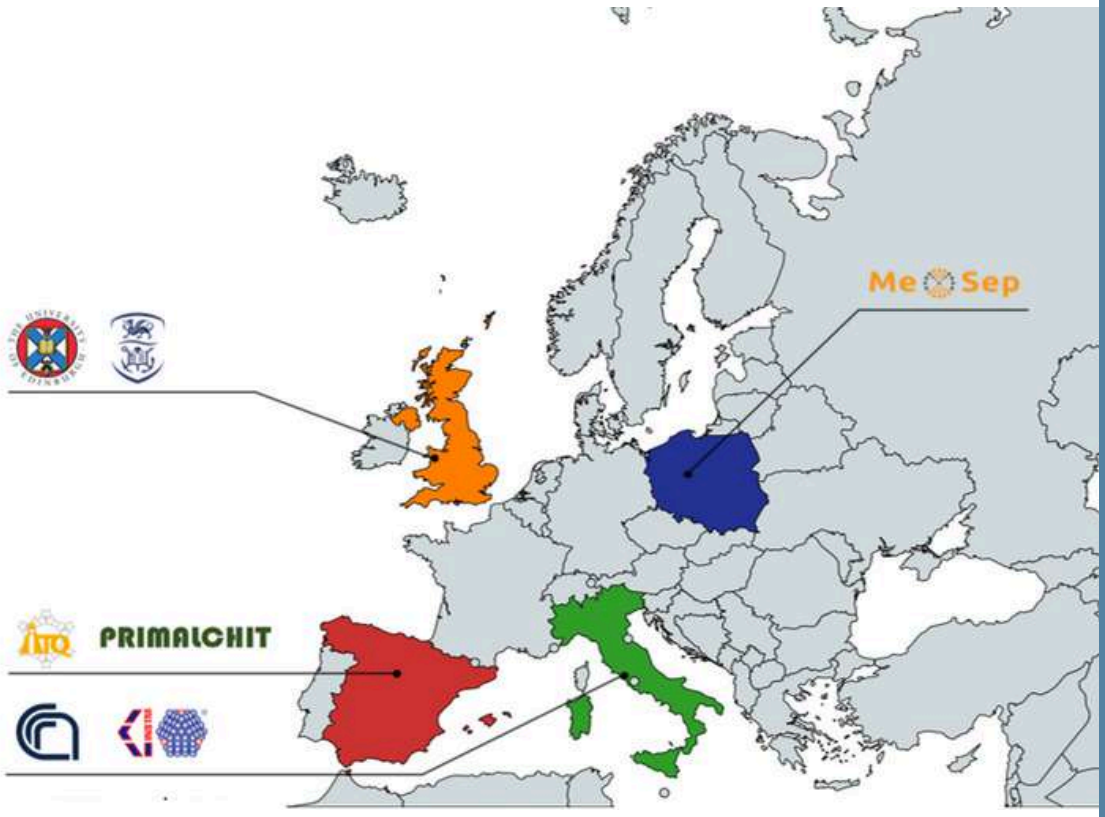
Portfolio Activities! »»

On June 17th and 18th, we have been in Helsinki for the European Innovation Council's (EIC) CO_2 and Nitrogen Portfolio Annual meeting. More than 30 representatives from all the 8 projects (CONFETI, DAM4CO$_2$, ECOMO, HYDROCOW, ICONIC, Mi-Hy, MINICOR, and SUPERVAL) have shared their progresses and discussed pioneering innovations on "Carbon Dioxide and Nitrogen Management and Valorisation".
You can find more information on the portfolio activities on the joint website.

The annual meeting gave also the opportunity to finalize the joint document "Carbon dioxide and nitrogen management and valorisation portfolio: strategic plan". This document defines the objectives of the portfolio, identifies the activities to reach those objectives and guides their implementation by establishing a governance structure. The Portfolio Strategic Plan is the product of a collaboration between the portfolio projects and EIC members. The Portfolio Strategic Plan will be revised once per year. It is freely available via the Publications office of the European Union

Finally, in Helsinki the premiere of the portfolio inaugural video took place. The video was recorded during the Kick-off Meeting in Bruxelles and it is a presentation of the portfolio. It is now available on the YouTube channel of the EU Innovation Council and SMEs Executive Agency (@EASMEExecutiveAgencyforSMEs)

Always as a portfolio activity, in June we organized the second session of the EIC portfolio webinars series on "Electrochemical methods for CO_2 and Nitrogen valorization: Electrocatalysis and Gas Diffusion Electrodes". This event was co-organized with the Confeti project, and the talks were given by Prof. Marta Costa Figueiredo (University of Eindhoven) and Dr. Jürgen Kintrup (Covestro Deutschland AG). More webinars will be organized in this year, and you can find the full list here:



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