Laboratory of catalysis and materials

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LSRE-LCM, Laboratory of Separation and Reaction Engineering – Laboratory of Catalysis and Materials, is an R&D Unit leader in Chemical Engineering research, focusing on separation, reaction engineering, environmental technologies, catalysis, and materials science. LSRE-LCM's main headquarters is at the Department of Chemical Engineering of FEUP - Faculdade de Engenharia da Universidade do Porto, Porto - Portugal, with an external branch - the IPL branch at Instituto Politécnico de Leiria. The LSRE-LCM team consists of nearly,160 persons, including permanent professors and researchers, post-doctoral researchers, PhD students, project researchers, administrative staff, and visiting researchers.

The designation of LSRE-LCM R&D Unit was adopted in 2013 from the merger of the two research units: LSRE - Laboratory of Separation and Reaction Engineering, led by Professor Alírio Rodrigues; and the LCM - Laboratory of Catalysis and Materials, led by Professor José Luís Figueiredo, that started the partnership in 2002 and in 2004 awarded the status of Associated Laboratory LSRE-LCM.

More recently, in 2021, the LSRE-LCM R&D Unit in articulation with two more R&D Units based at the Department of Chemical Engineering at FEUP (LEPABE - Laboratory for Process Engineering, Environment, Biotechnology and Energy; and, CEFT - Centro de Estudos de de Fenómenos de Transporte, stands for Transport Phenomena Research Center in Portuguese) created the Associate Laboratory in Chemical Engineering – ALiCE, the largest Portuguese Associate Laboratory in the area of Chemical Engineering, with more than 450 researchers, 40 % of which with PhD, who over the past 20 years have contributed to consolidate a relevant international position in the Chemical Engineering field at the University of Porto (top-1, 20 and 100 at national, European and World level, respectively, according to well-known and different rankings).

LSRE-LCM R&D Unit is organised into five research groups (RG): RG1 - Cyclic Adsorption/Reaction Processes; RG2 - Product Engineering; RG3 - Environmental Engineering; RG4 - Carbon Materials, Catalysis and Environmental Assessment; RG5 - Photo-Electro-Chemistry and Nature-Inspired Systems. LCM includes mainly RG4 and RG5. Both groups work in the intersection of the fields of Catalysis and Carbon (as LCM – Laboratory of Catalysis and Materials in the past), extended to adjacent scientific domains that include three major research areas: Nanostructured Carbon Materials, Environmental Catalysis and Technologies, Energy, Fuels and Chemicals.

Nanostructured Carbon Materials

Nanostructured Carbon Materials is a transversal area of research in the LCM working on the development of carbon materials (nanotubes/nanofibers, graphene derivatives, graphitic carbon nitride, carbon dots, carbon gels, ordered mesoporous carbons, graphene derived, among others) with tuned textural and surface chemical properties. The tuning of textural properties and surface chemistry of carbon materials is a major research area of LCM, allowing these functionalized materials to be used as catalysts, adsorbents, material in membranes, supercapacitors, sensors, functional textiles, and biomedical devices.

The team has a lengthy background in the modification of textural properties and surface chemistry of carbon materials [1-3]. Functional groups containing O, N, S, B or P (Figure 1) can be incorporated on the surface of carbon materials [4] either by in-situ doping during synthesis [5, 6], or by post-doping in the presence of heteroatom-precursors [7-9]. Current developments focus on solvent-free methodologies using ball-milling mechanical treatments and thermal treatments to incorporate different heteroatoms (N, S, P, and B) [9-13]. The LCM is actively involved in synthesising biomass-derived carbon materials [13,14] and producing carbon nanotubes from simulated and solid plastic waste precursors [15].

José Luís Figueiredo (former LCM leader) and Fernando Pereira (LCM leader) co-authored the development of a reliable method for quantification and identification of oxygenated functionalities on carbons, providing a major asset to correlate the catalytic properties of the carbon materials with their surface chemistry. The technique was in the paper Modification of the surface chemistry of activated carbons [16], in Carbon journal in 1999. At the time, the article presented a novel method for quantitatively analysing the oxygenated groups on the surface of carbon materials released under heating during temperature-programmed desorption (TPD). The work has been used around the world by several researchers working on the subject (~2800 citations -Scopus, Feb.25) and, more recently, the basic principles required to perform an adequate analysis, allowing the correct assessment (qualitatively and quantitatively) of the oxygenated groups on the surface of carbon materials were revised and a set of "best practices" for the TPD analysis of carbon materials, in general, was established [17].

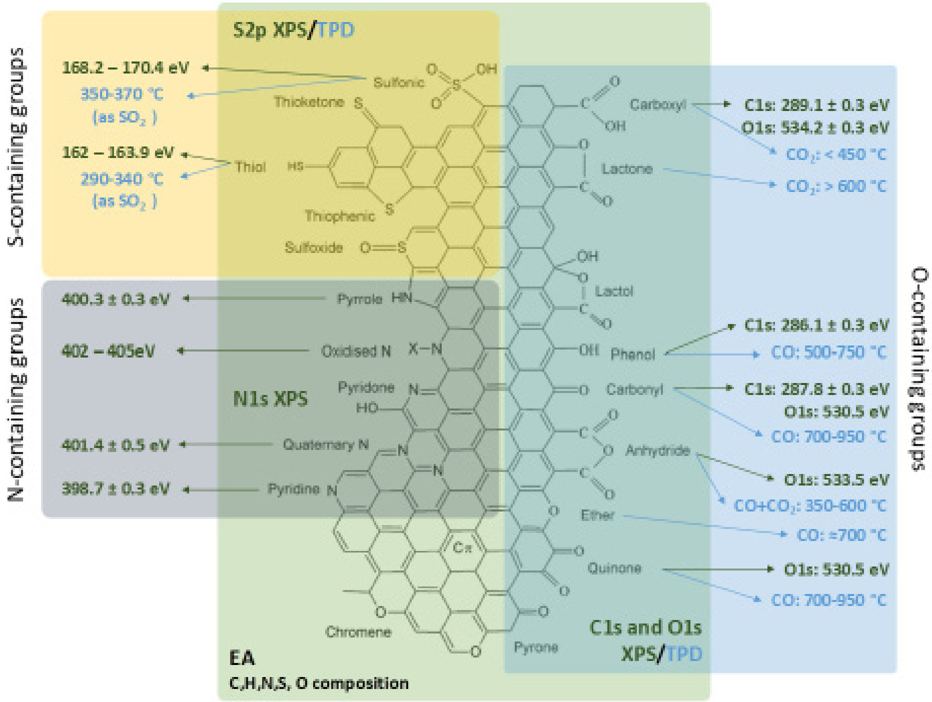

Figure 1. Oxygen, nitrogen and sulphur surface groups incorporated on carbon materials and techniques for their identification/quantification (reprinted from [33]).

Carbon materials can act as supports for several active and stable mono and bimetallic catalysts, incorporating different metal active centres on adequate carbon supports or hybrid materials [18, 19]. However, carbon materials can also function as catalysts on their own. Novel metal-free catalysts include acidic carbon xerogels/nanotubes for acid reactions like esterification of acetic acid [20], hydrolysis of cellulose and hemicelluloses, and the production of acetins via transesterification [21]. In addition, heteroatom-doped carbon nanotubes have been employed for the oxidation of organic compounds [12, 22].

Besides catalytic and adsorption applications, carbon materials have been investigated under the groups in the development of functional textiles. Photosensitive nanoparticles, led by titanium dioxide (TiO2) were investigated in cotton textile cleaning, replacing harsh industrial bleaching methods [23]. Graphitic carbon nitride, a metal-free photocatalyst, introduces eco-friendly self-cleaning and antimicrobial qualities through budget-friendly LED setups in textiles [24]. Innovative clothing designs achieving over 30 dB shielding effectiveness for electromagnetic interference were developed using carbon nanotubes, TiO_2, Fe_2O_3, and PEDOT:PSS [25]. Textiles coated with 70 wt.% Bi_2O_3 dispersed in a polymeric matrix surpassed heavy lead-based solutions in flexibility and efficacy for high-frequency radiation protection.

Environmental Catalysis and Technologies

The LCM has given special effort to design innovative solutions and methods for environmental protection technologies. Research is being carried out for water characterization, treatment, and desalination, as well as removing pollutants from gaseous and liquid effluents. The design of carbon-based catalysts for the oxidation of organic compounds in water by different advanced oxidation processes (AOPs) is a consolidated research area within the groups; materials are applied in catalytic ozonation [26-28], catalytic wet (air/peroxide) oxidation [22, 29, 30], persulphate activation [31], and photocatalysis [32-34].

For the reduction of inorganic compounds, the team has consolidated work on catalytic reduction of different oxyanions in water (such as NO_3-, BrO_3- and ClO_4-) [35-37], allowing them to be converted into less toxic species and avoiding the use of "trapping" technologies. Carbon materials [18, 38] and metal oxides [39] (such as TiO_2, Al_2O_3, ZrO_2, CeO, and ZnO, among others) were used as support for the active metal phase in an attempt to synthesize more stable and active catalysts for the reduction of inorganic ions. In this context, several combinations of noble/promoter metals, as well as their rearrangement on the surface of the catalyst (in alloy form or not), were studied to achieve efficient conversion and selectivity in the catalytic process. Different strategies were established to support mono [40] and bimetallic [18] catalysts in macrostructured monoliths, which were then applied for the continuous catalytic reduction of inorganic ions. Integrated technologies for oxidation and reduction of pollutants in water have been studied to improve the efficiency of water and wastewater treatment [41], resulting in a Provisional Patent Application PT 118885 (Figure 2). These methodologies were recently extended for the degradation of per- and polyfluoroalkyl substances (PFAS), also known as the Forever Chemicals [42].

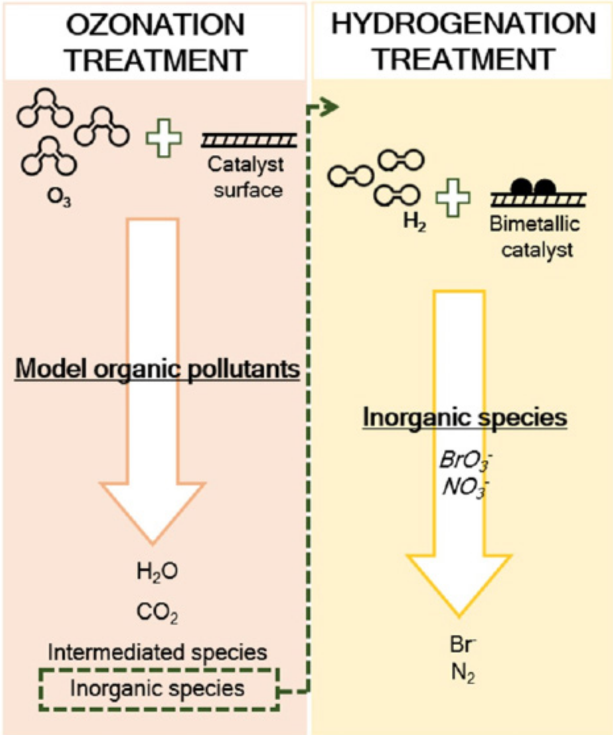

Figure 2

Concerning air pollution abatement, efficient metal oxide catalysts for the total oxidation of volatile organic compounds (VOCs) and carbon-based catalysts for NO reduction were developed. The team developed a cryptomelane-type manganese oxide synthesized by a novel solvent-free technique [22], and current research on VOCs abatement researcher is being made on the immobilization of powder catalysts on structured supports, like monoliths, to overcome the limitations of the use of powder catalysts [43]. In the selective catalytic reduction of NOx with carbon (SCR-C), different carbon materials (activated carbon, carbon nanotubes and carbon xerogel) functionalized with controlled physicochemical properties (texture and surface chemistry), as well as structured catalysts, have been investigated [44].

In the field of water characterization, relevant advances were made in the environmental monitoring and risk assessment with several solid-phase extraction and liquid chromatography-tandem mass spectrometry (SPE-LC-MS/MS) methods being developed and optimized for the determination of more than 50 organic micropollutants in surface and wastewater matrices [45]. A new research topic on enantioselective analytical tools was started associated with an ERC granted to Ana Rita Ribeiro [46].

The increasing abundance and dispersion of microplastic particles (MPPs) in the environment and a better understanding of the resulting impacts have also been the focus of the LCM work, with some studies being carried out on the aging of MPPs under urban environment stressors and identifying the subsequent changes in their chemical structure [47]. Additionally, decontamination processes targeting microplastics and other water pollutants, such as adsorption, biocatalysis, and bioremediation, are being carried out [48].

The team employs comparative analysis of advanced wastewater treatment processes [49], identifying areas for improvement and assessing the environmental impacts associated with various oxidants used in decontamination procedures by the Life Cycle Assessment (LCA) methodology. Moreover, the LCA studies have been extended to the environmental performance of other technologies and products, such as packaging.

Energy, Fuels and Chemicals

This thematic area is focused on the development of new catalysts and technologies for the sustainable production of energy, fuels and chemicals, including photocatalysts for solar fuels and chemicals; electrocatalysts and carbon electrodes for energy conversion and storage; nanostructured catalysts for biomass conversion; and CO_2 utilization.

Photocatalytic technologies, including novel catalysts and innovative photoreactors, are being investigated: to produce renewable and sustainable hydrogen by visible-driven water splitting [50]; to the selective synthesis of aromatic aldehydes and imines [51]; to ammonia synthesis from water and nitrogen [52]; H_2O_2 production [53].

The team has been working on the continuous optimization of catalyst design, seeking effective yet less expensive electrochemical solutions for energy production and storage. From metal-free O-rich-carbon nanotubes [54, 55], to glucose-derived carbon materials [13], to their hybrids [56, 57], from cobalt and/or iron phthalocyanines [58-59] on CNTs, to the engineering of single atom Fe-N sites onto hollow carbon spheres [60] and carbon black [61], several materials have been deepen investigated in the oxygen reduction and evolution reactions with the support of the use of computational tools seeking for a better understanding of the processes/reactions involved.

Hierarchical carbons with different boron contents [62], glucose-derived carbons modified by introducing heteroatoms (O, P) and/or incorporating carbon nanotubes (CNTs) during the synthesis/activation procedure demonstrated promising results as supercapacitors [9, 56, 63]. The use of low-cost biomass-based materials (such as cork-based activated carbons) from eco-sustainable supercapacitors has been investigated. Carbon-based materials for flexible supercapacitors and wearable electronic gadgets have appeared as an interesting application [64], with some work being conducted in the lab.

The catalytic valorisation of biomass wastes into valuable chemicals is a promising technology that can link traditional refineries and renewable sources. At LCM, various carbon-supported noble (Ru-W-CNTs [65]) and low-cost metal catalysts (Ni-W bimetallic catalysts supported on glucose/carbon nanotube hybrid carbons [66]) have been developed

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The team has also been working on the development of technologies to transform excess CO_2 into valuable chemicals and fuels to protect the environment and reduce dependence on fossil fuels. Two front lines are under study: (i) utilization of CO_2 in the generation of C_1 products, and (ii) hydrogenation of CO_2 to C_2+ products (Figure 3). In the first, functionalized activated carbon (AC) and carbon nanotubes (CNTs) demonstrated to be high-performing supports for Ni-based CO_2 methanation catalysts, while the formation of a composite of AC and CeO_2 showed excellent performance in this application, with some property-performance relationships and reaction mechanisms being established by ex-situ and in-situ characterization [72]. For CO_2 hydrogenation to methanol and the reverse water-gas shift reaction, good performances were obtained using Cu-based catalysts supported on pristine CNTs and composites of pristine and functionalized CNTs : ZnO [73]. In the CO_2 hydrogenation to hydrocarbons through Fisher-Tropsch reactions, multimetallic catalysts (Fe, Co, K, Na) supported on carbon and aluminium oxide materials have been investigated, with the preliminary work showing that CNTs and Al_2O_3 are suitable catalytic supports for the reaction. Concerning the metallic phase, Na-containing catalysts promote the highest CO_2 conversion, whereas Fe catalysts have the highest selectivity in the C_2-C_4 products [74].

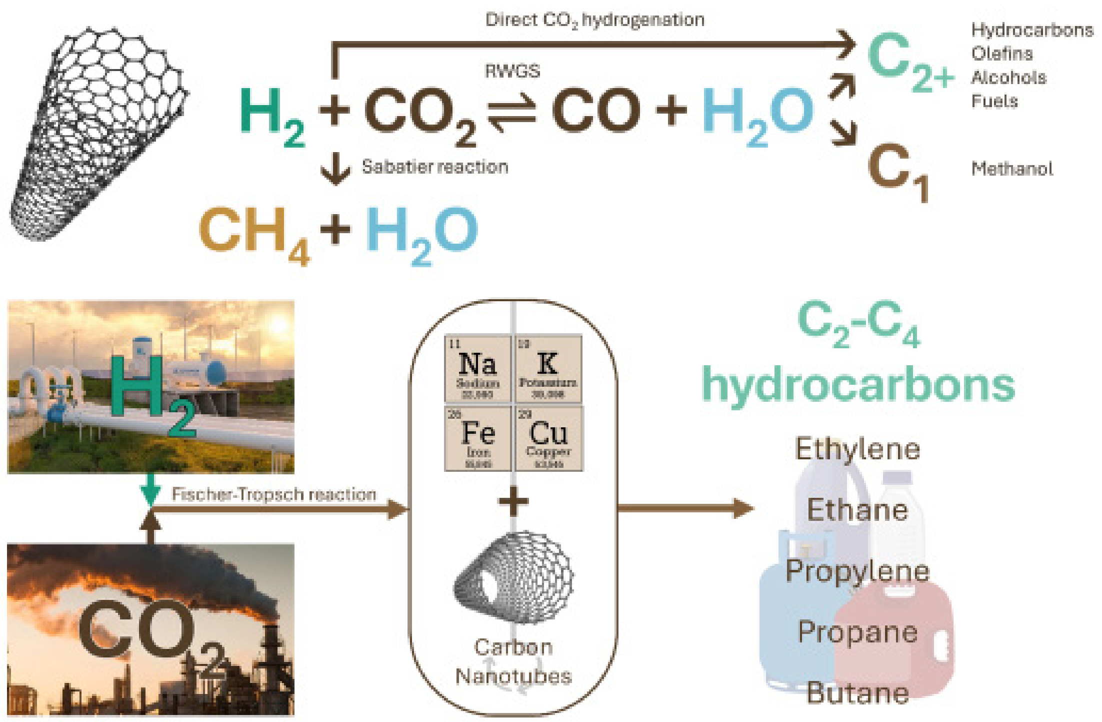

Figure 3

All these activities and discoveries are only possible due to the available facilities and collaborations of LCM (Figure 4). The lab is fully equipped to support advanced materials synthesis, modification, and characterizations. For synthesis, facilities include glass/quartz reactors; high-temperature ovens with controlled heating and atmospheres (inert, oxidant, reductive); high-pressure/vessel-pressure reactors; ultrasonic processors; microwaves, as well as several techniques for materials immobilization on macrostructured supports (dip-coating, spin-coater, viscosimeter). In-house, the materials characterisation can be assessed by N_2 adsorption-desorption isotherms; thermogravimetry analyses; temperature programmed desorption/oxidation/reduction (TPD/TPO/TPR), TPD of ammonia and CO_2, elemental analysis (CHNS/O); electrical conductivity (4-point-probe); contact angles; atomic absorption; and several spectroscopies: Fourier transform infrared spectroscopy (FTIR), spectrofluorimetry; Raman; UV-Vis spectroscopy; and inductively coupled plasma atomic emission spectroscopy (ICP).

To evaluate the material's performances under catalytic and other applications, versatile in-house made/adapted reactor installations are available, as well as some commercial options of batch and continuous high-pressure/temperature reactors, some with coupled analytical techniques. Analytic facilities are available for liquids and gases samples, including: high liquid chromatography – HPLC; liquid chromatography–mass spectrometry – LC-MS; gas chromatography - GC; gas chromatography-mass spectrometry – GC-MS; ion Chromatography – IC; total organic carbon – TOC; spectrophotometry of liquids; NOx Analyser; CO/CO_2 Analyser. Electrochemical workstations (potentiostat/galvanostat) are also available for the electrochemical assessment of materials with different cell configurations (two electrodes, three electrodes, half-cells).

Over the years, LCM accumulated collaborations with researchers from other R&I units around the world. It has participated in consortia for EU and transatlantic project funding, and it is part of collaborative laboratory networks, namely *BIOREF* - focused on R&I activities for advanced biorefineries. LCM is making a great effort to actively contribute to training high-level researchers and professionals through post-graduate and post-doctoral programs aligned with sustainable and scalable solutions for global energy and environmental challenges. For further information, we invite readers to visit our website at *lsre-lcm.fe.up.pt*.

Research areas	Research concept/keywords	*Future research targets*	Facilities
Nanostructured Carbon Materials	Carbon Materials with Tuned Properties	O, N, S, P, B-doping and synthesis of activated carbon, carbon xerogels and nanotubes, graphene, etc.) by liquid/gas-phase, mechanical treatments	Synthesis: Glass/quartz reactors; high-temperature ovens with controlled heating and atmospheres (inert, oxidant, reductive); high-pressure/vessel-pressure reactors; ultrasonic processors; microwaves, dip-coating, spin-coater, viscosimeter
	Carbon Materials for Catalysis	Carbon-Supported Metal Catalysts	
	Metal oxides for Catalysis	Metal oxides (Mn, Ce, TiO_2, FeO, Co) and metal oxides carbon composites	Characterization techniques: N_2 Isotherms; TGA; TPD/TPO/TPR/TPD-NH3; Elemental Analysis (CHNS/O); Conductivity (4 Point Probe); Contact angles; Atomic Absorption; FTIR; Spectrofluorimeter; Raman; UV-Vis; ICP
	Advanced Functional Materials	Functional Textiles	
Environmental Catalysis and Technologies	Advanced Oxidation Processes	Catalytic ozonation, catalytic wet oxidation, photocatalysis	Reaction installations Analytic facilities: HPLC, LC-MS, IC, TOC, GC, GC-MS, Spectrophotometry Liquid Samples, NOx Analyser, CO/CO_2 Analyser
	Catalytic Technologies for Water and Air Cleaning	Catalytic reduction of inorganic ions and PFAS degradation	
		Total oxidation of volatile organic compounds (VOCs)	
		Carbon-based catalysts for NO reduction	
	Water Characterization, Desalination and Purification	Monitoring and risk assessment Enantioselective analytical tools	
	Lyfe Cycle Assessment		
Energy, Fuels and Chemicals	Electrochemical Technologies for Energy Conversion and Storage	Oxygen/hydrogen reduction and evolution reactions	Electrochemical workstations (potentiostat/galvanostat) 2/3 electrodes, half-cells
		Supercapacitors	
	Biomass Conversion into Fuels and Chemicals	Catalytic valorization of agro-forestry and urban biomass residues	Reaction installations Batch and continuous high-pressure/temperature reactors Analytic facilities: HPLC, TOC, GC, GC-MS
		Conversion of Biomass and Bio-Oils to Aviation Fuels and Chemicals	
		C6 sugars and sugarcane molasses valorization	
	Catalytic Technologies for CO_2 Conversion	CO_2 Methanation	Reaction installations coupled with GC
		CO_2 Hydrogenation to Methanol	
		Reverse Water-Gas Shift Reaction	
		CO_2 Hydrogenation to Hydrocarbons	

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