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Proceedings of the 3rd International Conference on Water Energy Food and Sustainability (ICoWEFS 2023)

Springer

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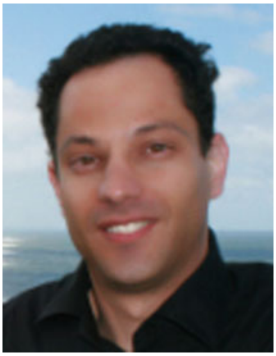

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Keynote Speakers

Eduardo Zarza-Moya
Plataforma Solar de Almería—CIEMAT—Spain

Eduardo Zarza-Moya is a researcher with 37 years of experience in the field of concentrating solar thermal (CST) technologies. At present, he is working at the Plataforma Solar de Almería (PSA) as Technical Coordinator and Head of the R+D Unit online-Focus CST technologies. He has coordinated several national and International R+D projects related to CST technologies (the projects: DISS, Solar Thermal Desalination, CAPSOL, PREDINCER, DETECSOL, etc.). He is a member of the AEN206/SC117 and IEC/TC117 standardization committees for solar thermal power plants, the Scientific and Technical Committee of the European Association of Solar Thermal Electricity (ESTELA), and the Spanish representative at the Executive Committee of SolarPACES.

Concentrating Solar Thermal (CST) Technologies for Sustainability

Abstract

It is estimated that the global energy consumption will reach 18,608 Mtoe by 2035, and it has become evident that fossil fuels must be replaced by renewable energies in order to reduce the global warming and the climate change that are already underway and are clearly endangering the future of the mankind. Due to its unlimited nature, solar energy must play a significant role in this effort to replace fossil fuels. At present, there are different technologies to make use of the solar radiation and some of these technologies are already well known because they have achieved a wide commercial deployment worldwide. Photovoltaic (PV) systems are a good example of these successful solar technologies. However, Concentrating Solar Thermal (CST) technologies are also a very promising option to achieve a fully decarbonized energy sector. The commercial deployment of CST technologies is still small, with a total installed power of less than 8 GW if both power and heat applications are taken into account, and less than 7 GW if only CST power plants are considered. Nevertheless, CST technologies have many socio-economic and technical benefits that must be considered (i.e., they are suitable for both power and heat generation, there are many countries with a high level of direct solar radiation, excellent dispatchability due to their thermal energy storage systems, significant job creation in places where they are implemented, high local content and excellent complementarity with PV plants), and these benefits altogether make CST technologies an excellent option for clean and sustainable energy supply. The reasons why CST technologies must play a significant role in the decarbonization of the energy market will be further explained in this invited talk.

Fernando José Cebola Lidon
Faculty of Sciences and Technology of Universidade Nova de Lisboa (FCT/UNL)—Portugal

Fernando José Cebola Lidon is a full professor at the Faculty of Sciences and Technology of Universidade Nova de Lisboa (FCT/UNL) and a coordinator of the Research Center "Geobiosciences, Geoengineering and Geotechnologies"—GeoBioTec—Polo FCT/UNL and graduated in Biology and Geology from the University of Évora and in Biochemistry from the Faculty of Sciences of the University of Lisbon, with a Ph.D. in Biology—specialty Plant Biochemistry from FCT/UNL and recognition by ECE/USA. Develops is work in the field of Agroindustrial Production and Transformation Technologies, focusing its activity on the development of functional foods following sustainable perspective, considering principles of eco-efficiency with the adoption of last generation technologies and aiming at the promotion of economic added value. Thus, articulation with the agro-industrial industry is developing techniques for the biofortification of edible parts of foods of vegetable origin, equating with the introduction of technical itineraries to increase productivity indices, adoption of crop monitoring techniques with the introduction of edaphoclimatic sensors and definition of food and nutritional quality standards.

Biofortification of Pear Rocha with Calcium: Production Workflow and Quality Definition of the Final Product

Abstract

Low dietary intake of Ca in humans has been epidemiologically linked to various diseases (such as osteoporosis), which can have serious health consequences over time. Accordingly, the development of an agronomic workflow for Ca biofortification of Rocha pears (Pyrus communis L.) and the assessment of physicochemical deviations was carried out in an orchard located in the West region of Portugal. During fruits development, leaves were sprayed a total of seven times with $Ca(NO_3)_2$ (with concentrations between 0.1–0.6 kg ha^{-1}) and/or $CaCl_2$ (with concentrations between 0.4–8 kg ha^{-1}). During fruits development, chlorophyll a parameters, as well as leaf gas exchange parameters such as net photosynthesis, stomatal conductance, transpiration rate, instantaneous and water use efficiency, only showed minor deviations, which indicated that the threshold of toxicity was not surpassed. Calcium contents varied during fruits development and at harvest the average biofortification index varied between 47 and 63%. Besides, the equatorial region of the fruits showed for all treatments (substantially in Ca treated samples) higher Ca contents in the epidermal and in the central regions. It is concluded that, although prevailing a heterogeneous distribution of Ca in fruit tissues, high indexes of biofortification in Rocha pears can be prompt in the orchards, without substantial physicochemical changes. Accordingly, agronomic biofortification with Ca can be used as a strategy for benefiting consumer's health.

Wolf-Gerrit Früh
School of Engineering and Physical Sciences, Institute of Mechanical, Process and Energy Engineering, Heriot-Watt University, Edinburgh, Scotland

After completing a first degree in Physics at the University of Freiburg in Germany and a D.Phil. in Atmospheric Physics at the University of Oxford, Wolf-Gerrit Früh took up a position in 1997 teaching Mechanical and Energy Engineering at Heriot-Watt University in Edinburgh. His research covers a wide range from fundamental fluid dynamics experiments and simulations through wind turbine aerodynamics to energy systems modeling and the application of data science for prediction and optimisation for energy systems design and scheduling.

Recent work relevant to the energy-water-food nexus include a study to reduce the carbon footprint of whisky distillation through Renewable Energy and thermal storage, heat-recovery in commercial kitchen, aquaponics, vertical farming, and desalination through solar-powered reverse osmosis. Most of these projects included collaboration with industry.

The Role of Low-Carbon Energy Sources and Storage in the Water-Food-Energy Nexus

Abstract

Water, food, and energy are absolutely essential. Providing all three in a way where supply of one does not endanger any of the other is a key requirement for sustainable development. This contribution identifies key challenges of, and opportunities for, providing energy for water and food provision. A brief overview over the energy requirements for water treatment and food production will identify some of the main challenges, such as water purification to drinking water quality, or growing crops for the increasing population. Having identified some specific challenges, this contribution will illustrate opportunities for more sustainable water and food production through renewable energy systems with a particular focus on local solutions. Examples include water purification powered by solar energy, energy recovery from water

treatment, and design of renewable energy systems required for controlled agriculture or aquaponics. The examples will highlight that many opportunities exist but that the energy system to support the water or food production must be carefully designed and optimized to improve the sustainability of water, food, and energy provision. In many cases, the optimization of an energy system for a particular task in a specific location will point toward a hybrid system, combining a range of energy generation technologies as well as some form of energy storage.

Antje Disterheft
NOVA School of Science and Technology, NOVA University Lisbon, Portugal

Antje Disterheft is a postdoctoral researcher and an integrated member at Center for Environmental and Sustainability Research (CENSE), from NOVA School of Science and Technology, NOVA University Lisbon. She is enthusiastic about exploring transformative processes that can support keeping human-nature systems in balance. Her research focuses on capacity building and new approaches to collaboration and co-creation for sustainability in Higher Education Institutions. In the past years, she started to engage deeper with the lens of care when thinking about sustainability and founded The CareLab for People and Planet at her faculty. The CareLab is a new space of collaboration, co-creation, and transformative learning where the interlinkages of inner and outer sustainability are explored.

She holds a Ph.D. in Social Sustainability (Universidade Aberta) and is an alumni of the Postdoc Academy for Transformational Leadership. German by nationality, but living in Portugal for more than 16 years, she enjoys being in nature, biking, and cooking.

Sustainability and Care—Why Our Inner Worlds Matter for Transformative Change

Abstract

Sustainability is inherently linked to questions of relationship: How do we relate to ourselves and to the world around us, and how can we enhance our transformative

capacity to thrive within the planetary boundaries? In the current times of multiples crises, e.g., the climate crisis, the effects of the recent pandemic due to COVID-19, as well a global crisis of trust, the concepts of intertwined inner and outer care are highly relevant: Personal care for ourselves (physical, emotional, and mental care) will impact and reflect in care for our communities and environment and draws on the social-ecological system perspective. Besides progress and growing awareness of sustainability, the world continues on a rather unsustainable path. While the Sustainable Development Goals (SDGs) offer us a roadmap of sorts toward a more sustainable future by 2030, it is becoming more apparent that without deeper transformation the goals might not be reached. What is missing in a world of increasing knowledge and sophisticated technology, where knowledge and technology might not hold the only solutions to solve complex unsustainability?

A growing number of scientists regard the inclusion of inner transformation as a deep leverage point for our sustainability endeavors. In this speech, I will explore why sustainability and care start with us and how our inner transformation can contribute to achieving the SDGs and help implement transformative change.

Shaul Sorek
Ben-Gurion University of the Negev
Jacob Blaustein Institutes for Desert Research, Zuckerberg Institute for Water Research, Department of Environmental Hydrology and Microbiology, Sde Boker Campus, Israel

Graduated as B.Sc. in mechanical engineering at the Ben-Gurion University of the Negev and received his M.Sc. and Ph.D. degrees at mechanical engineering of the Technion—Israel Institute of Technology. His research interests are in the development of fundamental modeling for biomedical and environmental systems. These address theoretical formulation for macroscopic transport phenomena of multiphase and multi-components driven through heterogenous media by inertia to drag dominant forces, and related numerical methods implementing Eulerian and Lagrangian concepts. Concerning quantitative decision making for water related resources management, he aims on causality-driven directed information in addition to spatiotemporal measures.

Policy Directed Information Technology Governing Network Welfare by Balancing the Water-Energy-Food Nexus and Human Perceptions (WEFH) Measures

Abstract

The water-energy-food (WEF) nexus represents the most fundamental requirements for human existence. It is essential for social welfare and thus is a top strategic priority in the management of water related stakeholders, which enables security and sustainability of the WEF limited resources. In previous publications, following Boltzmann entropy, we prove that the WEF resources are linked by information, across the regional administrative nodal network (municipalities, districts, and states) system. We suggest the Volume (normalized WEF expenditures product) as the nexus holistic measure from high to low Volume nodes. The node's Volume with its household income combined density allows for a decision-making policy tool on a node's Welfare Mass (WM—product of Volume and density). This enables the aiming at balancing regulation of the system welfare. The intervention to balance household WEF consumption inequality, in conjunction with the subjective attitude uses big data mining and analytics approach vis a vis water availability, yields a computer-based decision-support system (DSS) for defunding/subsidizing actions to over/under respective consumption leads to activating regulatory policy instrument.

This WEFH input across a region welfare imbalanced nodal network is quantified and visualized to derive recommendations for policy-driven interventions to regional ascribed nodes' WM source/sink terms.

Our hypothesis of causality-driven directional information is exemplified by a sharp price increase in wheat and rice, for USA and Thailand, respectively, that manifests its impact on the temporal trend of Israel's administrative districts of the WEF expenditures.

Dr. Maria Dermiki
Atlantic Technological University, Ireland

Dr. Maria Dermiki has an interdisciplinary background in scientific research with a degree in Chemical Engineering from Aristotle University of Thessaloniki, M.Sc. in

Food Science and Nutrition from University of Ioannina, Ph.D. in Food Biosciences from the University of Reading, postdoctoral research experience in Sensory Science and Dairy Science and finally industrial experience in Food Product Development. She has a holistic approach in product development, starting from Food Chemistry, Food Processing, Sensory Evaluation and Consumer Science, Packaging and Legislation, while adhering to sustainable practices. She is interested specifically in understanding the food choices of different population groups to develop nutritious foods that address the three pillars of sustainability and contribute to sustainable diets. Finally, she applies the UN Sustainable Development Goals to all the modules she teaches at undergraduate and postgraduate level, with the aim to help students apply sustainable practices in their professional and everyday life.

"What Would You Eat to Save the Earth?," Challenges and Opportunities for Sustainable Food Product Development

Abstract

Nowadays, the inadequate natural resources, climate change, and the negative impact of food production on the environment call for more sustainable food production practices that will ensure there will be enough food to feed the growing world population. Numerous solutions are being explored to overcome the multiple challenges that current food production systems are facing. For example, researchers are exploring the application of protein sources alternative to animal, such as insects or plants that could have a lower environmental impact compared to livestock production. Adding to the growing challenge of food security, almost a third of food produced worldwide is lost or wasted along the supply chain. In order to achieve the UN Sustainable Development Goal 12.3 that aims to "halve global food waste at consumer and retailer level and reduce food losses at the primary and secondary production stages" multiple ways to reduce food waste are currently investigated. One way to achieve this and retrieve nutrients from food waste is through the concept of upcycled foods which is gaining research and commercial interest.

Despite the promising outcomes of the shift to more sustainable food products, there are numerous challenges still to be addressed. For example, there is a need for changes in the current legislation that hinders innovation toward the use of alternative ingredients such as insects or underutilized ingredients and food processing by-products. Another challenge is consumer acceptability of such alternative ingredients and products that contain them. This presentation will explore the potential of sustainable food product development while also discussing the challenges industry faces.

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The conference co-chairs and organizing committee wish to acknowledge the support and sponsorship given in the organization of the ICOWEFS 2023—International Conference on Water Energy Food and Sustainability, held at the Polytechnic Institute of Leiria, Portugal.

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Preface

The 3rd edition of **International Conference on Water Energy Food and Sustainability (ICoWEFS 2023)**, taking place in Leiria (May 10–12, 2023) Portugal, aims to be a major event to foster innovation and exchange knowledge in the water-energy-food nexus, embracing the Sustainable Development Goals (SDGs) of the United Nations, building a better future for all bringing together leading academics, researchers, and industrial experts.

Taking into account that the EU has announced several actions for the implementation of the SDGs, such as reducing emissions to achieve climate neutrality by establishing a goal of zero net emissions by 2050, but there will be an interim goal of reducing emissions by 55% by 2030. In addition, the EU required an increase to 45% of renewable sources in the energy mix by 2030, as well as that all new buildings in the EU produce zero emissions from 2030 linked to the plan that in the future, energy consumption should be smaller. The conference will also focus on interconnected areas such as waste and effluent recovery, renewable gases, renewable energies, carbon capture, efficient use of water, and sustainable agrifood technologies.

The conference expects to foster networking and collaboration among participants to advance the knowledge and identify major trends in the above mentioned fields.

This conference is the 3rd co-organization between two Portuguese Polytechnics from Leiria and Portalegre and is open to new partners in the future events.

This edition counted with 87 presentations and 6 keynote speakers from 23 nationalities, that enriched the debate and evolution of these four scientific areas, namely AgriFood, Energy, Sustainability and Water. And as a result of the submitted papers, this book proceedings contains 70 papers.

Finally, the technical visits to a Smart Management Water System and a Biogas Power Plant in the surroundings of the conference city are contributors to promoting the dissemination of knowledge and the interaction among the academy, research centers, and the industry.

Welcome to this 3rd edition of the ICoWEFS 2023 Conference in Leiria, Portugal.

Leiria, Portugal
Portalegre, Portugal

João Rafael da Costa Sanches Galvão, Ph.D.
Paulo Brito, Ph.D.
Conference Chairs

Contents

Reactive Materials and Solutions Towards Treatment and Reuse of Waters with Contaminants of Emerging Concern

Adriano Santos Silva, Fernanda F. Roman, Ana P. F. da Silva, Jose L. Diaz de Tuesta, Marzhan Kalmakhanova, D. Snow, and Helder T. Gomes

Abstract Intense population growth has significantly impacted the quality and access to water, resulting in increased production and release of contaminants of emerging concern (CECs), such as pharmaceutical compounds. Catalytic wet peroxide oxidation (CWPO) is a promising technology for the removal of CECs that relies on the use of solid catalysts to accelerate the reaction, its reactivity and stability depending greatly on the catalyst used. This work aims to compare three typically studied catalysts: a clay-based, a carbon-based and a hybrid material, consisting in carbon-shell metal nanoparticle structure. Hybrid catalysts combine the benefits of metal-based catalysts (high activity) and carbon-based catalysts (low leaching), indicating to be a suitable choice. However, it is highlighted that the development of proper solutions for treatment and reuse of waters must pass through detailed identification and quantification of CECs, allowing better catalyst evaluations under real scenario conditions.

Keywords Water pollution · Remediation technologies · Heterogeneous catalysis · Fenton-like systems · Advanced oxidation processes

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1 Introduction

Intense population growth brought the need of solutions to various environmental problems directly related to the fast economic development over the years. The growing population also increased the demand for freshwater, a resource deeply affected by human activity [1]. Water is the most important resource for human life, but a significant population share still has no access to drinking water worldwide (*ca.* 34%) [2]. The number of people with access to drinking water has registered an increase since 2015, but it is not enough to achieve universal access to this resource by 2030, as targeted by the 17 goals of the UN agenda [3]. In this scenario, water reservoirs are under pressure due to contamination problems, soil erosion and inadequate sanitation [4]. Pathogens, detergents, chemicals, fertilizers, heavy metals and pharmaceuticals are examples of pollutants commonly found in water bodies, being a matter of public concern. In recent years, organic pollutants at trace levels have been identified in the environment, including in effluent streams of wastewater treatment plants, presenting new challenges for public authorities and environmental agencies [5]. This class of pollutants, known as contaminants of emerging concern (CECs), has been the focus of many studies [6].

The biggest problem related to the ubiquitous presence of CECs in water bodies is the lack of information on the effects caused by the presence of these compounds in water environments. Despite not being completely understood, some works reported that CECs presence in water directly affects human health and well-being. Damage to aquatic ecosystems and habitats were also reported in some studies [7]. This specific class of pollutants raises concern due to the expectation that pollution is likely to increase in the following years, caused by the predicted economic development. Aligned with the increasing pollution, there is a lack of evolution of mechanisms and regulations to address this environmental problem [8]. In this context, CECs pollution has become a matter of global concern for the water and wastewater treatment industry, public authorities, and society in general. Among CECs, pharmaceuticals have been a matter of special attention in the last years due to the proven negative effect these compounds have on aquatic life [9].

Several studies reported treatment options for water and wastewater contaminated with CECs, including adsorption, membrane filtration and electrochemical oxidation [10]. In addition, advanced oxidation processes (AOPs) have received great attention as suitable tertiary treatment alternative processes [11]. In recent years, studies exploring AOPs to treat water are focused on developing proper catalysts to promote robust oxidation power processes [12]. The mechanism behind AOPs action is related to the formation of highly oxidizing reactive oxygen species, such as hydroxyl radicals ($HO^{\cdot}$), capable of breaking down the contaminants into non-toxic simpler molecules. Among AOPs, catalytic wet peroxide oxidation (CWPO) has the potential to transform and remove organic pollutants from wastewater [13]. CWPO typically operates at mild conditions (25–130 °C and 1–4 bar), and a solid catalyst is used to promote decomposition of hydrogen peroxide into hydroxyl and hydroperoxyl radicals, which are highly oxidizing species [14]. The solid catalyst can be recovered and reused, which has been the main challenge for the validation of the feasibility of this technology. For instance, early studies demonstrated that a suitable catalyst for this process should be comprised of a support material (high surface area and mesoporous nature) and an active phase (transition metal) [12]. However, the need to solve iron leaching issues has led to new findings, showing that carbonaceous materials have a high potential to be used as catalysts due to their redox properties, allowing them to interact with hydrogen peroxide to form the oxidative radicals [15, 16].

In this work, a comparison between the performance of different catalysts for the CWPO of paracetamol (PCM), chosen as a model pharmaceutical pollutant, will be shown. The catalysts used for this purpose are examples of typical materials (pillared clays), carbon materials (carbon nanotubes), and hybrid magnetic carbon-based materials (multi-core shell ferrite nanoparticles). The discussion will be presented on the performance of each catalyst, showing weaknesses of the traditional materials compared to the most advanced and recent catalysts used in CWPO. Last, based on the conclusions gathered, future studies will be briefly discussed in the quest to develop reactive materials and alternative solutions for the treatment and reuse of waters containing CECs, to contextualize the importance of the work that has been done so far.

2 Methodology

2.1 *Reactants and Materials*

Natural clay was supplied from Kazakhstan. Iron (III) chloride hexahydrate (99%, Aldrich) and cobalt (II) chloride hexahydrate (99%, Fischer Chemical) were used for the preparation of pillared clay. Alumina (99%, BASF), ethanol absolute (96%, Fischer Chemical), ethylene glycol (99%, Fischer Chemical), iron (II) chloride tetrahydrate (99%, Acros organics), iron (III) chloride hexahydrate (99%, VWR chemicals) and polypropylene (average Mw ~ 250,000, average Mn ~ 67,000, Sigma-Aldrich) were used to synthesize carbon nanotubes. Iron (III) chloride hexahydrate (99%, Aldrich), cobalt (II) chloride hexahydrate (99%, Fischer Chemical), ethanediol (99.8%, Fischer Chemical), formaldehyde (38% w/w), resorcinol (99%, Alfa Aesar), tetraethyl orthosilicate (TEOS) (98%, Fluka Chemika), sodium hydroxide (98%, Labkem) and ammonia solution (28–30%, Merck) were used to prepare the carbon-coated magnetic nanoparticles. Paracetamol (98%, Alfa Aesar), hydrogen peroxide (30% m/v, Fischer Chemical), titanium (IV) oxysulfate (99.99%, Aldrich), sulfuric acid (98%, Labkem), sodium sulfite anhydrous (98%, Panreac), acetonitrile (99.9%, Fischer Chemical), and ortho-phosphoric acid (85%, Riedel-de-Haen) were used for oxidation experiments.

2.2 Preparation of Catalysts and Characterization

Pillared clay (PCLAY) was prepared by mixing a pillaring solution with natural clay (2 wt%) previously washed with phosphate buffer solution to remove impurities. The pillaring solution was prepared by dropwise addition of 0.5 M NaOH solution, at room temperature, to an aqueous solution of 0.5 M $FeCl_3$ and 0.25 M $CoCl_2$ to obtain a final solution with a molar ratio OH/(Fe + Co) = 2:1. The suspension was stirred at room temperature for 3 h, and left aging for another 72 h (no stirring). In the end, the final solid was recovered by filtration and washed until rinsing waters reached pH 7, dried overnight at 60 °C, and calcined at 600 °C for 5 h, leading to PCLAY sample.

Carbon nanotubes (CNTs) were prepared by chemical vapor deposition using polypropylene (PP) as a carbon source, and iron oxide supported on alumina (IO/Al_2O_3) as substrate. The metal substrate was prepared by adapting a sol–gel procedure described elsewhere [17, 18]. In brief, a $FeCl_2{\cdot}4H_2O$ solution in ethanol and a $FeCl_3{\cdot}6H_2O$ solution in ethanediol were mixed (molar ratio 1:2) with 6.6 g of alumina. The amount of reactants was adjusted to obtain 20% iron oxide supported on alumina. The final powder was calcined at 300 °C for 12 h and 600 °C for 24 h. The CVD process was carried out in a one-chamber reactor under N_2 flow (50 mL min^{-1}) considering 1 g of catalyst to 5 g of carbon source. The synthesis was conducted by heating at 850 °C for 1 h. In the end, the CNTs were recovered and washed with a 50% vol./vol. H_2SO_4 solution at 140 °C for 3 h to remove metal impurities, leading to a PP-derived CNT (CNT@PP sample).

Hybrid carbon-coated magnetic nanoparticles were prepared in a procedure divided into 4 steps: (I) synthesis of magnetic core, (II) carbon coating, (III) annealing, and (IV) etching. The synthesis of cobalt ferrite was performed by sol–gel, as described in other works [19]. Briefly, $CoCl_2.6H_2O$ solution in ethanol and $FeCl_3.6H_2O$ solution in ethanediol were mixed (molar ratio = 1:2) and stirred at 60 °C for 2 h. The resulting solution was stirred and the temperature was raised to 189 °C to remove all the solvent from the solid. The solid obtained from this step went through heat treatment at 300 °C for 24 h and 600 °C for 12 h in an oxidative atmosphere. The recovered material was washed to remove impurities and dried overnight in oven at 60 °C, leading to CoFe. For the carbon coating, the procedure involved the hydrolysis reaction of TEOS in the presence of ammonia and the polymerization reaction between resorcinol and formaldehyde. In brief, 0.25 g of nanoparticles were sonicated in 50 mL of water and transferred to a round bottom flask using 150 mL of ethanol. The flask was previously loaded with 0.1 g of resorcinol and 1.2 mL of ammonia solution. The mixture was stirred at 30 °C for 1 h when 0.15 mL of formaldehyde and 0.21 mL of TEOS were added to the flask. After 6 h of mixing, the temperature was raised to 80 °C, and the mixture was kept stirring for another 8 h. In the end, the nanoparticles coated with resin were washed several times until rinsing waters reached neutral pH. The last 2 steps involved the thermal treatment in N_2 atmosphere (1 h at 120 and 400 °C, followed by 3 h at 600 °C) and etching with NaOH 10 M solution for 16 h to remove the silica formed by TEOS hydrolysis, leading to production of CoFe@C.

Physisorption with liquid nitrogen and Fourier Transform Infra-Red (FT-IR) spectroscopy were performed to characterize the materials, as described in other works [12]. In brief, textural properties were gathered by analysis of N_2 isotherms at 77 K obtained in QuantaChrome NOVA TOUCH LX^4, equipped with long cells with a bulb and outer diameter of 9 mm. Prior to run N_2 adsorption analysis, samples were degasified at 120 °C during 16 h at vacuum. Specific surface area was determined by BET method and total pore volume at 0.98 of relative pressure. FT-IR analysis was carried out using a Perkin Elmer Spectrum Two FT-IR spectrometer at a resolution of 4 cm^{-1} and a scan range of 4000 to 450 cm^{-1}.

2.3 Liquid-Phase Experiments

Batch oxidation experiments were conducted in a well-stirred 250 mL round bottom flask equipped with a condenser and temperature controller. The initial concentration of PCM of 100 mg L^{-1} was used to evaluate treatment capacity of an organic pollutant in water. For the reaction, 100 mL of the model solution with pH adjusted to 3.5 using 1 M H_2SO_4 was loaded in the reactor, followed by the addition of the stoichiometric amount of H_2O_2 for complete mineralization of PCM. Once the temperature reached 80 °C, the catalyst was loaded, marking the beginning of the reaction (t_0). Non-catalytic and adsorption runs were performed under the same operating conditions in the absence of catalyst and H_2O_2, respectively.

PCM and H_2O_2 concentrations were monitored by periodically withdrawing aliquots from the reactor. H_2O_2 was determined based on a colorimetric method described in previous works [20], and PCM concentration was measured by a Jasco HPLC system at a wavelength of 277 nm (UV-2075 Plus detector) coupled with a Nucleosil 100-5C18 (15 cm × 2.1 mm id) column. The separation of the reaction products was obtained under an eluent flow of 0.300 mL min^{-1}, consisting of a mixture of ultrapure water with 0.15 wt%. of phosphoric acid (A) and acetonitrile (B) (A:B volume ratio = 90:10 v/v) for 6 min, followed by a gradient elution to reach A:B volume ratio of 65:35 up to 12 min (hold for 6 min). Leached iron was determined by atomic absorption spectroscopy (Varian SpectrAA 220) at the end of reaction.

3 Results and Discussion

3.1 Characterization

FT-IR spectra is shown in Fig. 1. The band observed at 780 cm^{-1} for PCLAY is related to quartz impurity in the material, commonly observed in materials originating from natural clays [12]. The band observed in 1025 cm^{-1} for PCLAY is ascribed to stretching vibrations of the Si–O group. For CNT, bands at 1636 and 1385 cm^{-1} can be attributed to C = C and carboxylate groups, respectively [21]. The band found in 3440 cm^{-1} is due to the presence of -OH groups of the water adsorbed in the CNT. For the CoFe@C nanoparticles, the band in 1632 cm^{-1} can be attributed to the bending vibration of the OH groups in the carbon-based shell. At last, both CoFe@C and PCLAY materials present a response around 450–500 cm^{-1}, ascribed to the presence of transition metals in their structure. These results confirm the majority carbon composition of CNTs, the metal-based composition for PCLAY and the hybrid nature of carbon-coated magnetic nanoparticles.

Regarding the textural properties (Table 1), it is possible to observe that PCLAY is the catalyst with the lowest BET-surface area (S_{BET}), which is related to the nature of the raw material used to prepare PCLAY. The CoFe@C catalyst has a lower surface area compared to CNT@PP, however, the hybrid nanoparticle possesses a higher surface area compared to the bare core (result not shown here). The same tendency observed for S_{BET} is repeated for the total pore volume (V_T) of the catalysts. The N_2 isotherms classification for the materials is typical of catalysts composed of mesopores for CNTs and CoFe@C, and macropores for PCLAY.

Fig. 1 FTIR results

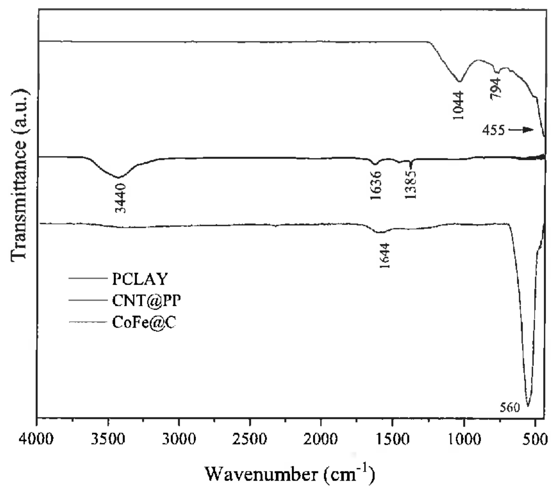

Table 1 Textural properties

Catalyst	S_{BET} ($m^2 g^{-1}$)	V_T ($mm^3 g^{-1}$)
PCLAY	19	72
CoFe@C	57	60
CNT@PP	94	247

3.2 *Liquid-Phase Reactions and Insights*

The results obtained for PCM and H_2O_2 concentrations upon reaction time in the CWPO of PCM are shown in Fig. 2. All materials display high catalytic activity, allowing to achieve more than 95% of PCM removal in 8 h of reaction. The catalytic activity towards H_2O_2 decomposition and PCM oxidation follow the same order: CoFe@C, followed by PCLAY and then by CNT@PP. PCM removal profile for CoFe@C is sharper compared to the other catalyst. For CoFe@C, the fast H_2O_2 decomposition observed in the beginning of the reaction slowed the performance, likely by allowing radical recombination, resulting into non-active species [22]. On the other hand, CNT@PP did not result in complete removal of PCM due to its inability to fully decompose H_2O_2.

Characterization results showed the presence of iron for CoFe@C and PCLAY, whereas CNT@PP did not display the presence of metal-based species, according to FT-IR spectra. This indicates that transition metals play a significant role in this process. Most likely, the iron present in those materials is the main characteristic in influencing the catalytic activity in a CWPO process. However, carbon-coated materials are preferred compared to PCLAY due to easy recovery and higher stability of the active phase arising from the protection of the core due to the carbon layer. Textural properties revealed a behavior previously reported in literature: higher surface areas do not necessarily lead to increased activity of catalyst in CWPO, as other parameters may be more relevant, such as the presence of metallic phases or carbon content. Tuning the metallic content may affect the stability of the catalysts, as increased leaching is expected, resulting in water pollution and loss of catalytic activity. On the

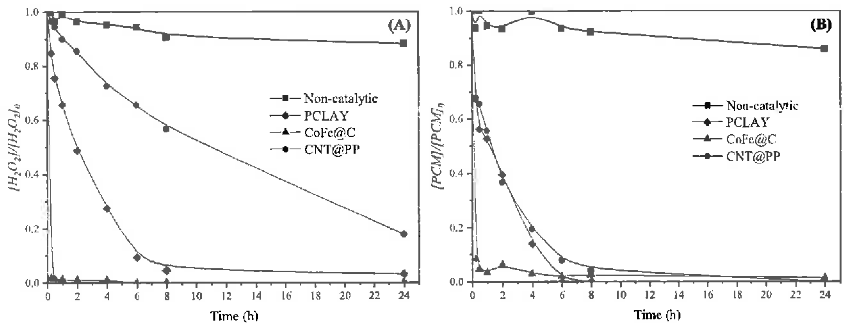

Fig. 2 Results of H_2O_2 **a** and PCM **b** concentrations through time for oxidation reactions

other hand, carbon-based materials do not have such high catalytic activity compared to metal-based ones, so surface modifications (e.g., doping) are required to increase the activity [16, 20].

The activity of PCLAY in the process is well-known and studied in the literature [12]. In brief, introducing transition metals in the clay structure during the pillaring process is the key feature ascribing activity for this material in H_2O_2 decomposition into hydroxyl radicals. The advantage of this material is related to its low-cost nature, which includes the use of a widely available resource (clay) with a low-cost modification process that can be performed at room temperature, not requiring energy to be carried out. However, the use of such material for liquid-phase reactions is complicated due to its hard recovery, which hinders the reutilization of the catalyst for other reactions. The reutilization feature is an important characteristic in CWPO studies because it is considered one advantage of this process compared to homogeneous Fenton reactions.

Carbon materials were already studied as catalysts for removing other organic pollutants by CWPO [23]. The catalytic activity of these materials for CWPO process is due to the electron donor–acceptor properties of the carbon structure, allowing the electron-transfer mechanism between the catalyst and H_2O_2. The study of non-supported carbon materials for CWPO is more recent than traditional metal-supported catalysts, and its application brings considerable advantages. For instance, non-supported carbon materials do not face iron leaching problems. Therefore, these catalysts do not face inactivation issues. It is important to highlight that carbon materials are stable for this application due to the mild temperature in which the process is carried out, which is the main issue related to the application of carbon catalysts in other fields. Herein, the CNT used to promote PCM degradation has yet one advantage compared to traditional carbon materials: the low iron content that cannot be removed from CNT (< 1%) confers magnetic properties to the nanostructured carbon-based material. This characteristic is useful for removing the magnetic CNT catalyst from the reaction medium, which increases the feasibility of using the same material several times as a catalyst for oxidation reactions.

Hybrid materials comprised of carbon and metal, such as carbon-coated magnetic nanoparticles, represent another material with potential applicability in CWPO [24]. These catalysts have several advantages, since the coating is responsible for protecting the magnetic core and promote the hydrogen peroxide decomposition into hydroxyl radicals, leading to increase the catalytic activity toward the removal of organic pollutants by CWPO. In this work, for example, the bare cobalt ferrite core can remove 43% of pollutants in 8 h of reaction (result not shown), which is a lower removal than the coated nanoparticle (> 95%). For the case reported here, the innovation lies in the synthesis method of the magnetic core, sol–gel, in this case. This method is known to result in nanoparticles with high purity, yield and monodispersibility of sizes.

Results of adsorption tests after 6 h of contact time performed with the tested materials are shown in Fig. 3, along with the concentration of iron determined at the end of experiments. Removal of PCM by adsorption did not reach more than 30% for all catalysts, with more expressive adsorption observed for CNT@PP due to its higher

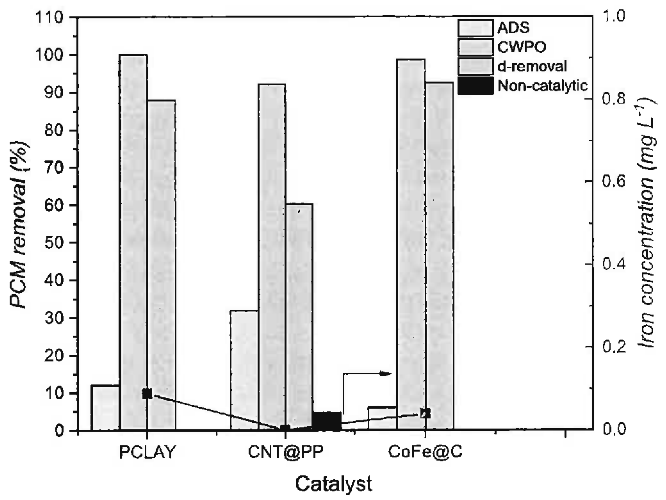

Fig. 3 PCM removal by CWPO and adsorption (left axis) and iron concentration at the end of the CWPO reaction (right axis)

surface area. The parameter d-removal calculated for all tests represents the difference between removal by CWPO and by adsorption, being useful to assess the catalytic efficiency of each material. For instance, according to this parameter, CoFe@C and PCLAY remove PCM with the highest efficiency compared to CNT@PP. However, CNT@PP is the catalyst with the lowest iron leached at the end of the reaction, which is related to the lower amount of iron in this material. Iron leaching for PCLAY was higher than CoFe@C, which could be expected due to the nature of the CoFe@C catalyst, where the carbon layer protects the magnetic core against leaching.

4 Final Remarks and Prospects

All materials used in this work showed catalytic activity towards the degradation of PCM, chosen as model CEC, by CWPO. Each catalyst was assessed in equal operating conditions and chosen to illustrate a specific case from literature. The work reported here shows the increased efficiency and feasibility of carbon-based magnetic nanoparticles over traditional metal-based catalysts (represented by PCLAY) and non-supported carbon catalysts (represented by CNT@PP). Nevertheless, carbon catalysts are still preferred over traditional metal-based materials due to iron leaching problems faced by the second class. All reactions carried out in this study were made in batch experiments, which is a useful tool for evaluating catalysts' performance. Still, continuous experiments should be considered in future works to evaluate the feasibility of the wastewater treatment studied here in real systems, including membrane-based systems and permeable reactive barriers incorporating novel reactive materials. Construction of permeable reactive barriers may provide a large-scale process for purification and storage of purified wastewater, but research needed to demonstrate this technology is in its infancy. Results presented here provide the basis for further investigation and the selection of catalysts for upscaling CWPO (or other AOPs) requires their evaluation under realistic, large-scale scenarios. Effective treatment of CECs necessarily passes through the proper knowledge of types of compounds, concentrations, and distribution. Identification and quantification of those CECs is an important step when considering development of remediation technologies, and future works should consider combining efforts to monitor pollutants in real wastewater to remediation technologies, aiming designing solutions that approximate to the real conditions observed in real effluents. We demonstrate here that selection of an appropriate catalyst for advanced oxidation technology will provide the means for construction of a complete treatment system for CECs and a scientific symbiosis since both fields should be studied simultaneously. This research show promise for a practical treatment of emerging pollutants under real scenarios and sustainable reuse of wastewater through both membrane and permeable reactive barrier systems.

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