



Foreword/Avant-propos

International Renewable Energy Congress 2015: Focus on biomass energy, environment and sustainable development



This thematic issue of *Comptes rendus Chimie* is devoted to the 6th International Renewable Energy Congress – IREC'2015 – held from 24 to 26 March 2015 in Sousse (Tunisia). Eighteen papers have been selected and, after peer-reviewing, fourteen were accepted for publication, which cover important subjects such as biomass thermochemical conversion, synthesis of micro- and mesoporous materials, wastewater, and exhaust gas treatments. They are focusing on recent development on waste minimization and the impacts of pollutants. The minimization techniques covered thermal and plasma gasification, fast pyrolysis, CO₂ supercritical extraction, absorption, membrane filtration, and catalytic oxidation.

The International Renewable Energy Congress (IREC) provides a forum for researchers and practitioners around the world on recent developments in the fields of renewable energy and sustainable development. This conference is organized every year in Tunisia; it was initiated by Prof. Maher Chaabene in 2009. This congress covers eight different topics in the field of energy and sustainable development: solar thermal energy, photovoltaic energy, wind energy, biomass energy, hybrid energy, hydrogen and fuel cells, energy management and storage, sustainability and environment. The IREC'2015 attracted 336 papers from 24 countries. Of these, 210 papers were selected by an international scientific committee for oral presentations and 76 for poster presentations. All papers have undergone a formal peer-reviewing procedure to ensure the highest possible technical quality with the aid of more than 130 reviewers [1]. The Biomass Energy, Environment and Sustainable Development (BE2SD) session has attracted 57 scientific papers. During the BE2SD session, 24 oral communications and 15 posters were presented. Dr. Amhamed Assanosi has received an award for the best oral presentation of the BE2SD session; his contribution *Fructose dehydration to 5HMF in green self-catalysed DES composed of N,N-diethylethanolammonium chloride and p-toluenesulfonic acid monohydrate (p-TSA)* was selected by the scientific committee for publication in this thematic issue.

1. Selected contributions

For this thematic issue of *Comptes rendus Chimie*, 14 manuscripts have been selected. The aim of the first one, entitled *The nature of the deposited carbon at methane cracking over a nickel loaded wood-char* [2], was to investigate the catalytic properties of raw biomass chars and Ni-loaded biomass chars prepared at a high-heating rate for the methane decomposition reaction. The raw chars exhibited a moderate catalytic activity in methane cracking, while the Ni-loaded ones showed a catalytic activity 10 times higher than that of the raw chars. The deposited carbon was a highly ordered one, as evidenced by XRD, Raman analysis, and oxygen reactivity tests. The activation energy in the combustion reaction was estimated to be 300 kJ/mol. These results indicate that biomass chars can be effective low-cost and active supports for metal impregnation to be used in the catalytic cracking of hydrocarbons for hydrogen production.

The second paper, *Biomass conversion to hydrogen-rich synthesis fuels using water steam plasma* [3], studies an experimental plasma chemical reactor equipped with an arc discharge water steam plasma torch used for biomass conversion into hydrogen-rich synthesis fuels. Glycerol and crushed wood were used as biomass sources. The effects of different conversion parameters, including the water steam flow rate, treated material flow rate, and plasma torch power, were studied. The experimentally obtained results were compared with the model based on thermodynamic equilibrium. Additionally, the quantification of the plasma conversion system in terms of energy efficiency and specific energy requirement was performed. It has been found that the synthesis gas can be effectively produced from biomass using water steam plasma.

The third paper, *A robust numerical model for characterizing the syngas composition in a downdraft gasification process* [4], proposes a numerical model developed to characterize the chemical composition, the heating value, and the temperature of the syngas produced by using a downdraft gasifier fueled with residual biomasses. The

process of gasification is essentially described through a global reaction that includes all the gaseous species and the yields of char and tar. The syngas chemical composition has been obtained by solving a set of equations that are of mass and energy balances, methanation and the water-gas shift reactions, which govern the gasification process. The proposed model was calibrated and validated through the comparison with two sets of experimental data. The comparison between the results of the simulation and the experimental data has shown a very good agreement, which allows pointing out the capability of the model to characterize the syngas composition and the temperature of the producer gas. Moreover, the performed sensitivity analysis shows the influence of the moisture content and equivalent ratio on the chemical composition, equilibrium temperature, and heating value of the producer gas.

The fourth paper is entitled *Fructose dehydration to 5HMF in green self-catalysed DES composed of N,N-diethylethanolammonium chloride and p-toluenesulfonic acid monohydrate (p-TSA)* [5]. Due to the increasing concerns about the availability and accessibility of fossil fuel reserves, and the subsequent effect of using them on climate change, production of green energy has recently become a hot area of interest in the research field. As a renewable energy source, biomass conversion to biofuels has shown great potential towards green fuel production; particularly fructose conversion to 5-hydroxymethylfurfural (5HMF) as a building-block material and source of green fuels and other high-value chemicals. Herein, the authors investigate fructose dehydration to 5-hydroxymethylfurfural (5HMF) as a green fuel precursor, using a green self-catalyzed environment-friendly Deep Eutectic Solvent (DES), composed of inexpensive N,N-diethyl ethanol ammonium chloride as an organic salt and p-toluenesulfonic acid monohydrate (p-TSA) as a hydrogen bond donor (HBD) and a novel medium for the fructose dehydration reaction. The advantage of using this DES is its ability to act as a solvent and a catalyst simultaneously. It has been shown to actively catalyze the dehydration reaction of fructose under moderate reaction conditions with a high 5HMF yield of 84.8% at a reaction temperature of 80 °C, a reaction time of 1 h, a DES mixing ratio of 1:0.5 salt to p-TSA (w/w), and an initial fructose ratio of 5.

The fifth paper bears the title *CO₂ gasification of woody biomass chars: the influence of K and Si on char reactivity* [6]. Although the influence of metallic and alkaline elements on biomass char reactivity is well known, a quantitative assessment of this catalytic effect is hard to obtain because of the chemical and textural complexity of biomass. The effect of two elements, K and Si, on the CO₂ gasification reactivity of a biomass char was studied using thermal gravimetric analysis. A beech sample was pyrolyzed at 800 °C under a nitrogen atmosphere and then impregnated with known amounts of silicon or potassium, allowing one to obtain a wide range of K/Si ratios. The reactivity of the impregnated samples was studied under a CO₂ (20% vol.) atmosphere. The results show that at low conversion ratios, char reactivity depends mainly on its textural properties, with strong diffusional limitations. When conversion reaches 60%, the presence of a catalyst (K) and an inhibitor (Si) becomes the major parameter influencing reactivity. From

these experiments, a general trend was obtained between the K/Si ratio and reactivity as a function of conversion.

In the sixth paper, *Simulation of the fast pyrolysis of Tunisian biomass feedstocks for biofuel production* [7], an optimized model was developed for the production of biofuels from biomass using the SuperPro Designer tool. Four types of Tunisian biomass feedstocks, including date palm rachis, olive stone, vine stems and almond shell were selected for the fast pyrolysis process simulation. Simulation tests were performed at different temperatures ranging from 450 to 650 °C, at residence times ranging from 0.1 to 10 s, and then the product yields were determined. The obtained results indicated that a temperature of 575 °C and a vapor residence time of 0.25 s were the optimum parameters to maximize the bio-oil yield. A comparison between the different feedstocks indicated that a higher bio-oil fraction was obtained from the date palm rachis and vine stem. However, the difference between the samples was not significant and further investigations on the bio-oil properties are requested to select the suitable biomass for bio-oil production in Tunisia.

The seventh paper, *Synthesis of EMT/FAU-type zeolite nanocrystal aggregates in high yield and crystalline form* [8], focused on different ways to improve the yield and/or the crystalline quality of EMT/FAU-type zeolite nanocrystal aggregates obtained in the presence of an organic additive triethanolamine (TEA). The increase in the amount of the aluminum reagent enhanced the synthesis yield by a factor of 2.5 without affecting the crystallization rate and the microporous volume. On the other hand, the increase in the thermal treatment time allowed increasing the synthesis yield, the crystallization rate, and the microporous volume. Furthermore, the addition of EMT zeolite seeds into the starting reaction medium improved the crystallization rate and the microporous volume.

In the eighth paper, *Synthesis of mono and bi-layer zeolite films on alumina substrates* [9], bilayer zeolite films ZSM-5/ZSM-5, ZSM-5/EMC-1 and NaY/ZSM-5 were synthesized on α -alumina plates. The bottom ZSM-5 or faujasite Y (NaY) zeolite layers were obtained by direct hydrothermal synthesis or by using a seeding step followed by a secondary growth method, respectively, while the secondary growth method was used for the synthesis of all top zeolite layers. A complete characterization of the obtained materials was proposed using various techniques, such as X-ray diffraction, scanning electron microscopy, X-ray fluorescence and nitrogen sorption measurements. Continuous and highly crystallized bilayer zeolite films with thicknesses around 11–18 μm were obtained, and this protocol was selected to prepare low cut-off membranes.

The ninth paper was *Design and characterization of flat membrane supports elaborated from kaolin and aluminum powders* [10]. Porous flat ceramic-metal composite (Cermet) membrane supports were elaborated from kaolin and aluminum powder mixtures by the press drying-sintering process. The evolution of structure and surface properties was followed by water permeation, tensile strength, mercury porosimetry, surface charge, and contact angle measurements. These characterizations have demonstrated that the addition of aluminum to the kaolin matrix has a beneficial effect on the membrane support

properties. In particular, the water permeability and mechanical strength increased gradually in the presence of aluminum. In addition, the hydrophilicity of the cermet supports was found to increase gradually with the aluminum load. However, the surface charge was not affected by the aluminum introduced into the cermet composition. Filtration experiments were carried out with the support containing 4% wt of aluminum. These results indicated that this support could be used to purify dye-containing water.

The tenth paper, *Evolution of a fungal ecosystem in a water distribution system to a positive bacterial biofilm subsequent to a treatment using essential oils* [11], aimed at demonstrating the direct link between the microbial ecosystem of drinking water distribution systems and animal health in pig breeding. Based on a survey over 18 months, a treatment using essential oils proved to be efficient in increasing piglet health and zootechnical performance. Water pipe biofilms were monitored by laser scanning confocal microscopy, while zootechnical performance and health cost data were collected from professional organizations. In two representative monitored herds, it was observed that the drinking water distribution pipes, initially fouled by fungi, were replaced by a bacterial film while both veterinary costs and the total feed conversion ratio decreased. Essential oils may thus provide an efficient and sustainable alternative to the massive use of antibiotics for transforming an initial detrimental ecosystem to a positive biofilm.

The eleventh paper, *Effect of sludge pH and treatment time on the electrokinetic removal of aluminum from water potabilization treatment sludge* [12], deals with the treatment of industrial wastewater by electrocoagulation, which is well known to be very efficient and has been extensively studied. However, the generated sludge contained a huge amount of aluminum (when such a kind of electrode is used, several other metals are also present in the effluent). The objective of this paper was to study the possible removal of aluminum and chromium present in the sludge by electrochemical migration. This generated sludge contained high amounts of Cr (14,980 mg/kg of dry sludge), above the maximum level allowed by legislation, and Al (25,300 mg/kg of dry sludge). The potential toxicity of Al and Cr was discussed, as well as whether the efficiency of the EC process will be maintained if a proper treatment technology is followed to remove toxic metals from this sludge so that it can be safely used afterward without risk to the environment. The possibility of removing these pollutants from the resulting sludge by using an electrokinetic technique was tested. Acetic (3.6 M) and citric (1.3 M) acids were used as catholyte solutions to enhance the removal of aluminum and chromium (III).

The twelfth paper is entitled *Hydrogen sulphide removal from the effluents of a phosphoric acid production unit by absorption into chlorinated seawater under alkaline conditions* [13]. Atmospheric pollution during the manufacturing of Wet process Phosphoric Acid (WPA) is a critical environmental issue. The aim of this study was to examine the absorption of hydrogen sulfide (H₂S) emitted by this process into a chlorinated seawater solution under alkaline conditions. Parameters such as pH, sodium hypochlorite (NaOCl)

concentration, gas flow rate, operating time and volumetric ratio of the liquid as well as gas phases (Q/Q) were investigated for their effects on the H₂S absorption efficiency. It was found that sodium hypochlorite is an effective and economical H₂S oxidant. Moreover, according to the obtained results, a dose of 1 g Cl₂/L and a pH of 11 are recommended. The preferred gas flow rate in this study was equal to 15 L/min. Under these conditions, more than 98% of the hydrogen sulfide was removed, even at relatively high H₂S concentrations in industrial gases released from the plants of the Tunisian Chemical Group (GCT).

In the thirteenth paper, *Toluene total oxidation over Pd and Au nanoparticles supported on hydroxyapatite* [14], the catalytic total oxidation of toluene was performed on a palladium or a gold active phase on different stoichiometric, deficient, and carbonate-rich hydroxyapatite supports. Two preparation methods were used to introduce the active phase: conventional wet impregnation and nanoparticle deposition by impregnation of a colloidal suspension of a noble metal using the surfactant HEA16Cl. The obtained results showed that palladium was much more active than gold, the latter not being under a highly dispersed nanoparticle form. Moreover, a low palladium content (0.25 wt%) was enough to get high toluene conversion rates at low temperature. Introducing palladium by impregnation of the colloidal suspension or by conventional methods leads to similar catalytic efficiencies. Finally, systems corresponding to palladium supported on apatite supports, even with low palladium loading, were more catalytically efficient than palladium supported on classical alumina and could be classified among the most active systems for toluene total oxidation investigated in the literature.

The last paper, *Optimization of Algerian rosemary essential oil extraction yield by supercritical CO₂ using response surface methodology* [15], deals with the determination of optimal values of operating parameters such as temperature and pressure, leading to the best yield for supercritical CO₂ extraction of essential oils from the local rosemary plant, using the response surface methodology (RSM). The maximum of essential oil recovery percentage relative to the initial mass of leaf powder was 3.52 wt%, and was obtained at 313 K and 22 MPa. A second-order polynomial was used to express oil recovery, and the calculated mass of recovered oils using the RSM was very close to the experimental value, confirming the reliability of this technique. The chemical composition of the Algerian Rosemary oil obtained under optimal conditions (313 K and 22 MPa), determined by GC–MS analysis, revealed the presence of camphor (major compound) (52.12%), 1,8-cineole (9.65%), camphene (7.55%), α -pinene (6.05%), borneol (3.52%), aroma dendrene (2.11%), verbenone (1.97%), α -caryophyllene (1.71%), and others.

2. Conclusion and acknowledgements

This foreword to the thematic issue devoted to the IREC'2015 Conference provided an overview of the topics covered by the articles. The Guest Editors are thankful to the Editor-in-Chief of *Comptes rendus Chimie*, Prof. Pierre Braunstein (Strasbourg, France) for providing them with

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