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Editorial

Damos la bienvenida al 2025 con un Boletín cargado de noticias, artículos y reseñas.

Comenzamos el número con la fantástica noticia del premio a la trayectoria profesional en Ingeniería Química otorgado por el Grupo Especializado de Ingeniería Química de la RSEQ a nuestro compañero Tomás Cordero, Catedrático del Departamento de Ingeniería Química de la Universidad de Málaga. Desde el Boletín nos sumamos a las felicitaciones a Tomás por este reconocimiento tan merecido.

Continuamos con dos artículos científicos: el primero, liderado por el Instituto de Energías Renovables de la Universidad Nacional Autónoma de México, trata sobre la valorización mediante procesos de secado solar y pirólisis de *Sargassum fluitans III* procedente del Caribe Mexicano; el segundo, del Grupo de Procesos Termoquímicos de la Universidad de Zaragoza, estudia el impacto del pretratamiento hidrotermal en medio ácido sobre el rendimiento de carbones derivados de biomasa en el almacenamiento electroquímico de iones de sodio.

En este número contamos también con tres reseñas de Tesis Doctorales: Cristian Daniel Jaimes-Páez (Universidad de Alicante), Raúl Llamas-Unzueta (INCAR-CSIC) y Laura Gómez Alonso (ICB-CSIC).

Continuamos con una reseña del proyecto BIVALIA-CM, recientemente concedido, en el que participan nuestros compañeros del Departamento de Ingeniería Química de la Universidad Autónoma de Madrid. El proyecto tiene como objetivo la integración de distintas tecnologías para el desarrollo de procesos que permitan la valorización de residuos procedentes de la industria alimentaria.

Cerramos este número con dos reseñas de actividades organizadas por el GEC que tuvieron lugar en Baeza (Jaén): el II Curso del GEC, titulado "*El carbono y sus materiales, caracterización y aplicaciones*", así como las II Jornadas

de Jóvenes Investigadores del Grupo Español del Carbón, celebradas a continuación. Ambas actividades tuvieron un gran éxito de participación y destacaron por la alta calidad de las ponencias impartidas en el curso y las contribuciones presentadas en las jornadas.

Por último, tenemos el placer de anunciar las comunicaciones que han resultado premiadas en las distintas categorías:

- **Artículo científico:** "Influence of temperature on the char produced through solar pyrolysis of Agave Angustifolia leaves", cuyo autor de correspondencia es Alejandro Ayala-Cortés, y corresponde a una colaboración entre el Instituto de Carboquímica-CSIC y el Instituto de Energías Renovables, UNAM (Mexico).
- **Artículo de divulgación y/o investigación docente:** "Promoting scientific culture and sustainable development among secondary students", cuyo autor de correspondencia es J. Torres-Liñán, del Departamento de Ingeniería Química de la Universidad de Málaga.
- **Contribución de un artículo científico en la sección "Nuevos Grupos":** "Engineered Carbon-based Nanomaterials for Energy, Biomass Valorization, Photocatalysis & Water Remediation" cuyo autor de correspondencia es Diana M. Fernandes, del REQUIMTE/LAQV, Departamento de Química e Bioquímica, Faculdade de Ciências, Universidade do Porto (Portugal).

¡Enhorabuena a todos los premiados!

Covadonga Pevida García
José Luis Pinilla Ibarz

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El grupo especializado de Ingeniería Química de la RSEQ concede a Tomás Cordero el premio a la trayectoria profesional en Ingeniería Química

María José Valero Romero

Universidad de Málaga.

Estimados amigos del Grupo Español del Carbón, es un honor para mí redactar esta reseña para compartir con vosotros el merecido **Premio a la Trayectoria Profesional en Ingeniería Química** que fue otorgado al Profesor Tomás Cordero por el Grupo Especializado de Ingeniería Química de la Real Sociedad Española de Química (RSEQ), en la convocatoria de 2023. Este reconocimiento será otorgado públicamente durante la XL Reunión Bienal de la RSEQ, que tendrá lugar en Bilbao, entre el 30 de junio y el 3 de Julio de 2025.

La concesión de este premio, junto con otros conseguidos anteriormente por miembros del grupo, demuestra, una vez más, el gran nivel científico del GEC. Estoy convencida de que todos los miembros del Grupo Español del Carbón se suman a mi felicitación y comparten mi satisfacción al ver cómo un investigador de destacada excelencia científica y humana, con una notable dedicación al avance de la Ciencia y la Tecnología desde diversos enfoques, tales como la investigación y la gestión, ha sido merecidamente reconocido por su trayectoria profesional.

A continuación, os menciono algunos detalles de este premio y del Profesor Tomás Cordero.

Cada año, la RSEQ organiza los premios de investigación del Grupo Especializado de Ingeniería Química con el objetivo de reconocer las contribuciones más destacadas en esta disciplina. En esta ocasión, el Profesor Tomás Cordero fue merecedor del Premio a la Trayectoria Profesional en Ingeniería Química, un reconocimiento que pone en valor sus aportaciones al campo de la Ingeniería Química a lo largo de su carrera investigadora y profesional. Este premio representa, además, un reconocimiento profesional de compañeros del misma área ya que su candidatura estuvo propuesta por numerosos socios activos del Grupo Especializado de Ingeniería Química de la RSEQ.

En cuanto a su trayectoria profesional, Tomás Cordero es Catedrático en el Departamento de Ingeniería Química de la Universidad de Málaga desde 2006. Se licenció en Ciencias Químicas en 1983 y obtuvo su Doctorado en Ciencias (Ingeniería Química) en 1987, ambos en la Universidad de Málaga. Fue beneficiario de dos becas postdoctorales: Universidad-Empresa (1988) y PFPI (1989). En 1990 realizó una estancia postdoctoral en la Pennsylvania State University (EE.UU.).

Desde 1997, es responsable del grupo de investigación TERMA (TEP-184, <http://www.grupoterma.uma.es/>), especializado en Ingeniería Química y Tecnología Ambiental. Su investigación

se ha centrado en el estudio de los procesos de valorización de biomasa y residuos industriales, enmarcados en la sostenibilidad, para la obtención de biocombustibles, productos químicos y materiales avanzados de carbono, con relevancia científica y técnica en procesos de adsorción y catálisis aplicados a la protección ambiental y aplicaciones energéticas.

Entre sus trabajos de investigación destaca la preparación y caracterización de carbones activados mediante gasificación parcial de lignina Kraft o madera de eucalipto, y activación química por pirólisis catalítica, logrando altos rendimientos debido a la reducción de alquitranes durante el proceso. Además, ha estudiado la preparación de carbones activados mediante activación química de lignina Kraft con $ZnCl_2$ y H_3PO_4 , aplicados a ligninas Alcell® y otros residuos lignocelulósicos, generando carbones en distintas morfologías: polvo, formas granulares, fibras de carbono y monolitos, con alta resistencia a la oxidación y elevada acidez.

Algunos de estos materiales de carbono han sido utilizados como catalizadores ácidos o soporte de catalizadores metálicos para procesos de biorrefinería (como las reacciones de metanol a DME, MTO y ODH) y, más recientemente, el uso de gas de síntesis ($CO + CO_2 + H_2$) derivado de la gasificación de residuos de biomasa y/o de CO_2 e H_2 obtenidos mediante captura de carbono y fuentes renovables, para su conversión en biocombustibles. Por último, hay que destacar sus trabajos de investigación relacionados con la eliminación de contaminantes en fase gaseosa y líquida mediante adsorbentes y catalizadores heterogéneos basados en carbono.

Esta investigación la ha desarrollado gracias a la obtención de numerosos proyectos de investigación regionales, nacionales e internacionales, así como a través de contratos de investigación con empresas. Como resultado, ha publicado más de 160 artículos en revistas científicas de alto impacto, en áreas como Ingeniería Química, Tecnología Ambiental y Ciencia de Materiales. Además, ha sido reconocido por el "Ranking" Stanford como uno de los investigadores más citados en su área a nivel mundial durante su carrera científica. Ha dirigido cerca de una veintena de tesis doctorales, demostrando así una excelente capacidad formativa. Cuenta además con varias patentes y capítulos de libros y ha presentado más de 300 comunicaciones en congresos nacionales e internacionales y varias conferencias plenarias y Keynote, como la que impartió en la X Reunión del GEC en Girona.

Tomás ha desempeñado también diversos cargos de gestión científica. Fue miembro del equipo de

coordinación del área de Tecnología Química de la Agencia Nacional de Evaluación y Prospectiva (ANEP) entre 2010 y 2015, y ha formado parte de varios paneles de evaluación nacional en temas relacionados con la Ingeniería Química.

Entre otros cargos, ha sido secretario del Grupo Español del Carbón (1997-2005) y Vicepresidente de la sección de Ingeniería Química de la Real Sociedad Española de Química (2016-2022). Además, ha sido director de la Escuela de Doctorado (2020-2022) y Vicerrector de Personal Docente e Investigador (PDI) (2022 – 2023), ambos cargos en la Universidad de Málaga.

No deja de ser menos importante destacar su contribución a la formación de varias generaciones de Ingenieros Químicos en la Universidad de Málaga. Muchos de los que hemos asistido a sus clases coincidimos en que Tomás es un profesor excepcional, no solo por su profundo conocimiento en el área, sino también por su calidad humana y su habilidad para transmitir los fundamentos y conceptos de la Ingeniería Química de manera clara y accesible. Además de su labor docente en la titulación y grado de Ingeniería Química, Tomás ha impartido clases en otras titulaciones, como Ingeniería Industrial e Ingeniería en Organización Industrial y, actualmente, en el Máster Interuniversitario en Ingeniería Química, del cual fue coordinador por parte de la Universidad de Málaga durante 5 años. También mencionar que participó de forma activa en la adaptación de la titulación de Ingeniería Química, en la Universidad de Málaga, al Espacio Europeo de Educación Superior, tanto a nivel de Grado como de Máster.

esta oportunidad para mostrar mi admiración por su perseverancia y compromiso en todas las tareas que realiza. Además, destaco su capacidad para enseñar y transmitir valores esenciales como la honestidad y la constancia, los cuales han dejado una huella imborrable en mi formación y en la de muchos otros.



Este resumen breve de su trayectoria profesional muestra la gran cantidad de trabajo y el enorme esfuerzo que ha tenido que dedicar en el desarrollo de su carrera como investigador, y aún más destacar su contribución a la Ingeniería Química y a la formación de Ingenieros Químicos y doctores. Como su alumna y doctoranda, quiero aprovechar

Valorization of *Sargassum Fluitans III* from the mexican caribbean: solar drying and pyrolysis

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Abstract

Since 2014, the Mexican Caribbean has received more than 2360 m³ of *Sargassum* per km of coast. This sargassum is gathered with any treatment, and in the high temperatures it decomposes rapidly, generating pollution on the seashore and health problems in the local community. This work proposes a train of solar treatment for the valorization of *Sargassum fluitans III*, which consists of drying and pyrolysis for the production of biochar. Findings show that the drying time is around 90 min, independent of the type of solar dryer (i.e., greenhouse solar dryer (GHD) or open-sun drying). Results from the pyrolysis process show that the main characteristics of the biochar are affected by the temperatures of the process. At 800 °C, an amorphous char is obtained, with some inorganic compounds, such as potassium, sodium, phosphorus, sulfur, and chlorine, characteristics that are attractive for its use in energy storage, or as a biofertilizer.

Keywords: Concentrated solar energy, solar pyrolysis, biochar, solar drying

Resumen

Desde el 2014 el Caribe Mexico recibe mas the 2360 m³ de *Sargassum* por km de costa. Este sargassum es almacenado sin ningún tratamiento, y con las altas temperaturas se descompone rápidamente, generando contaminación en la costa y problemas de salud en la comunidad local. Este trabajo propone un tren de tratamiento solar para la valorización de *Sargassum fluitans III*, el cual consiste en el secado y la pirólisis para la producción de biocarbón. Los resultados muestran que el tiempo de secado es de aproximadamente 90 min, independientemente del tipo de secador (i.e. secador solar invernadero (GHD) o un secador solar abierto. Los resultados del proceso de pirólisis muestran que las principales características del biocarbón dependen de la temperatura del proceso. A 800 °C, se obtiene un carbón amorfo, con algunos compuestos inorgánicos, como potasio, sodio, fósforo, azufre y cloro, características que son atractivas para su uso en almacenamiento de energía o como biofertilizante.

Palabras clave: Energía Solar Concentrada, Pirólisis solar, biochar, secado solar.

1. Introduction

Since 2014, the Mexican Caribbean has received more than 2360 m³ of *Sargassum* per km of coast [1]. The massive influxes are believed to be linked to human activities that have affected ocean temperatures. In 2018, the amount of *Sargassum* reported on the Caribbean coast was 20 million metric tons [2], which then increased, although the amount varies depending on ocean currents and on seasonal conditions. At its peak (March to August, 2018) 4500 m³/km was being beached per month [2]. The main species are *Sargassum fluitans III*, *Sargassum natans I* and *Sargassum natans VII*, with *Sargassum fluitans III* (*S. Fluitans III*) being the dominant species, accounting for around 60% of the total biomass [3]. A range of solutions to clean up the beaches and seashore have been implemented, with mixed results. Unfortunately, sand is often removed during cleaning, and beach fauna is destroyed with severe consequences for ecological systems. Once the seaweed is collected and taken from the beach, it is usually left to accumulate in huge mounds of untreated biomass in nearby communities, where it decomposes rapidly, resulting in land and water contamination, and in health problems for the local population [4]. In order to mitigate these problems, several potential uses for the sargassum have been proposed that take advantage of it as a resource, rather than simply view it as a waste product. Examples include construction materials, such as MDF panels, adobe, and concrete [5], biofilters and adsorbents [6] and textile and pharmaceutical products [2].

All of these valorized waste products use dry biomass, which is produced via a costly, energy-intensive drying process. To reduce these costs, a solar treatment, followed by a pyrolysis process is proposed. This low-cost means of preserving the *Sargassum* reduces its volume and allows it to be accumulated safely, before pyrolysis is carried out, to give biochar. *S. Fluitans III* was dried in a greenhouse solar dryer (GHD), and thus, the kinetic parameters were obtained in order to design a large-scale *Sargassum* solar dryer. Pyrolysis was performed in two different pyrolysis reactor to valorize the *Sargassum*. The pyrolysis process was performed at different temperatures to evaluate the impact of temperature in the char properties.

2. Methodology

2.1 Solar drying system and process

Fresh sargassum samples were collected in Puerto Morelos, Quintana Roo, Mexico, and later classified by morphotypes, to perform a detailed evaluation of the solar pyrolysis process with different sargassum species (Figs. 1a and b). Drying tests were carried out in a greenhouse-type solar dryer (GHD) with spectrally selective films. This type of system integrates the solar and thermal drying in a closed environment with several advantages, including: reduction of product contamination, and energy consumption.

The GHD is 5 m x 6 m in area, and 4 m high, as even span greenhouse geometry. A steel structure, external and internal cover was used. The external walls are made of solid polycarbonate (PC) and consist in rear wall, front wall, and side walls. The internal walls are composed of cellular polycarbonate with spectrally selective film coating (SFC), and consist in rear internal wall, front internal wall, and side internal walls (Fig 1c). The lateral spaces between the external and internal walls prevent heat losses and to produce the airflow pattern. The spectrally selective film has two functions, the first to block

UV-blue light, helping to preserve sensitive UV-light compounds, and the second to absorb the radiant energy from the sun. The drying chamber consists of three stations where the drying trays can be laid on five levels. Each station is 4 m long, and was placed 0.5 m from the front and back walls. The GHD has three inlets and three outlets for air flow (Fig.1 d). Three fans were placed 1 m from the rear wall and at a height of 2.50 m, to promote the recirculation of the air, as previously reported in Román-Roldán et al. [7]. The energy absorbed by the spectral selective film is recovered with an air flow driven by fans placed above the false ceiling. Subsequently, air is conducted into the drying chamber because the side and front walls act as deflector plates. This innovative system avoids the use of solar collectors for air heating, reducing the land area occupied and the investment cost.

The air temperature, relative humidity (RH%) and air velocity inside the dryer were continuously measured. (Fig.1 e) The air velocity was set at 1.7 ± 0.2 m/s. The environmental parameters $HR_{air}\%$, environmental temperature (T_{amb}), global irradiance (I), and wind speed (v_w) were measured continuously using the data provided by the IER-UNAM meteorological station. Drying tests were performed at constant temperature for comparison.

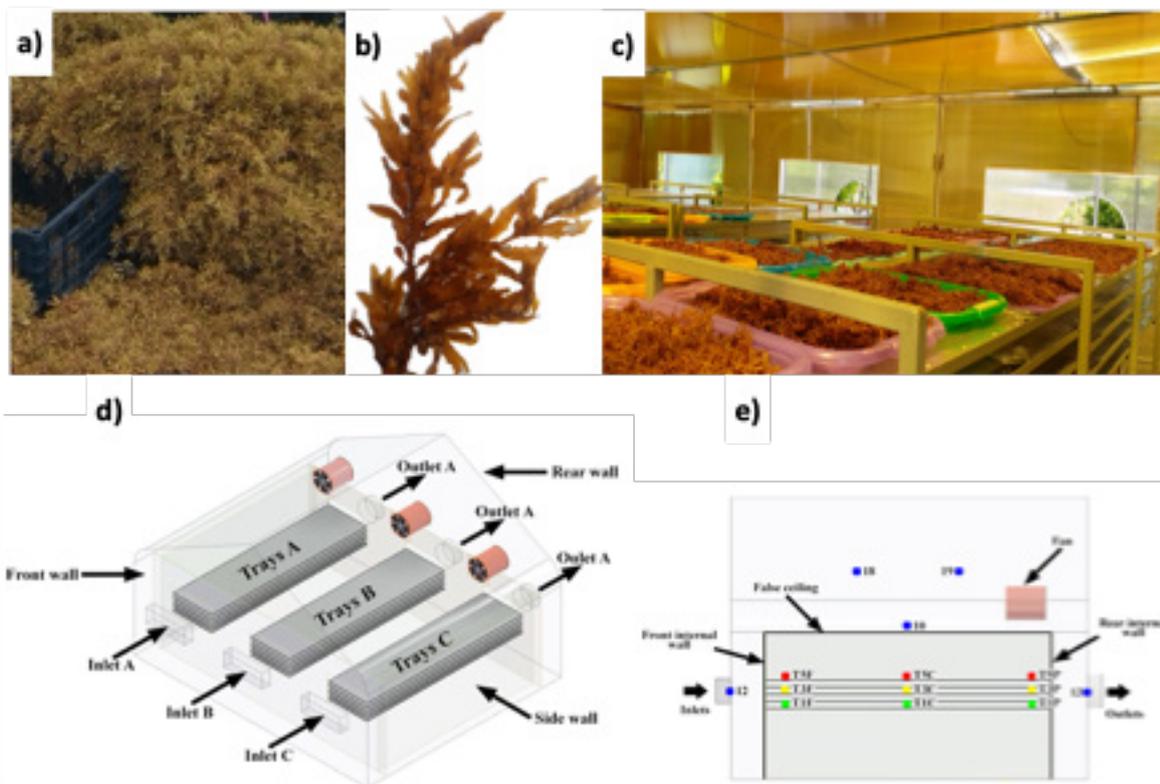


Figure 1. a) Sargassum collection stage in Puerto Morelos, Quintana Roo. b) Photograph of *S. Fluitans III*. c) New greenhouse-type solar dryer (GHD) with sargassum samples, d) Scheme of the GHD with the 3 drying stations, e) temperature, velocity and RH sensors located inside the GHD.

Two materials with specific optical properties were used as internal and external covers of the GHD. The external cover is solid polycarbonate that allow to transmit 95% of the energy of the entire solar spectrum [8]. The SFC material of the rear internal wall, front internal wall, and side internal walls allows

to transmit 40% of the sun energy, from 290 nm to 1600 nm. The solar rays that fall on the internal walls and the rear internal wall absorbs 50% of the solar energy, and 10% is reflected. The solar spectral irradiance measured under the PC and with the combination of PC and SFC is shown in Figure 2.

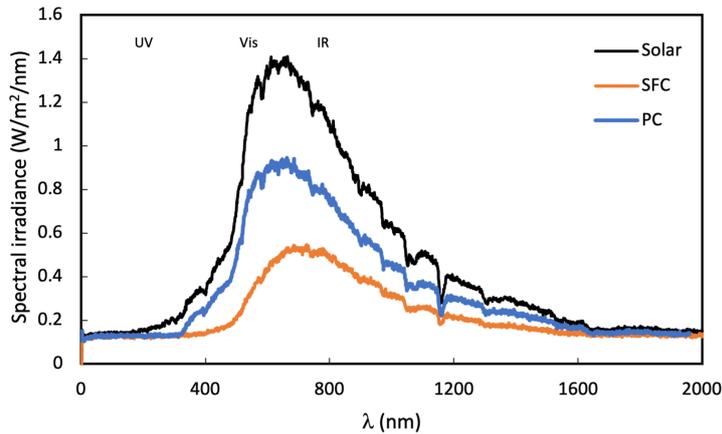


Figure 2. The spectral solar irradiance measured outside the solar dryer (solar) inside the solar dryer below the solid polycarbonate (PC), and the selective conductive film (SFC).

The weight loss of the sargassum on three drying stations of the GHD was measured every 30 minutes. In addition, open-drying tests were carried out simultaneously, for comparison. The drying kinetics was calculated using the normalized moisture content (MR) as follows:

$$MR = \frac{X - X_e}{X_o - X_e} \quad (1)$$

Where X is the moisture content as a function of time, X_o is the moisture content at the beginning of the drying process, and X_e is the moisture content at the end of the drying.

The Arrhenius equation is used to calculate the activation energy to drive drying.

$$D_{eff} = D_o e^{\frac{E_a}{RT}} \quad (2)$$

Where: D_o is a constant (m^2/s) that describes a constant diffusivity equivalent to the diffusivity at an infinitely high temperature, E_a is the activation energy (kJ/mol), R is the general constant of the ideal gas ($8.314, J / mol K$), and T is the absolute drying temperature (K).

2.2 Physicochemical characterization of *S. Fluitans* III and biochar

Physicochemical characterization was carried out on the sample using different techniques to evaluate the chemical composition and the morphology of the raw biomass and biochar. CHNS-O elemental composition was performed on the dry *S. fluitans* III biomass using a Perkin Elmer PE2400 analyzer. On the other hand, a thermogravimetric analysis (TGA) of the raw biomass and biochar was carried out to analyze its decomposition in a temperature range of 50 to 900 °C in a nitrogen atmosphere (TA, Q500). The morphology of both *S. fluitans* III and biochar was analyzed using a scanning electron microscope (S-5500 Hitachi). The biomass, biochar, and remaining ash were characterized using energy dispersive spectroscopy (EDS) coupled with a scanning electron microscope (SEM). The methodology for chemical characterization by EDS was carried out in 3 different areas of the sample, and an average of the results

was obtained for each element. Finally, an X-ray diffraction (XRD) analysis of the amorphous structure of the biochar was carried out (Rigaku Ultima IV).

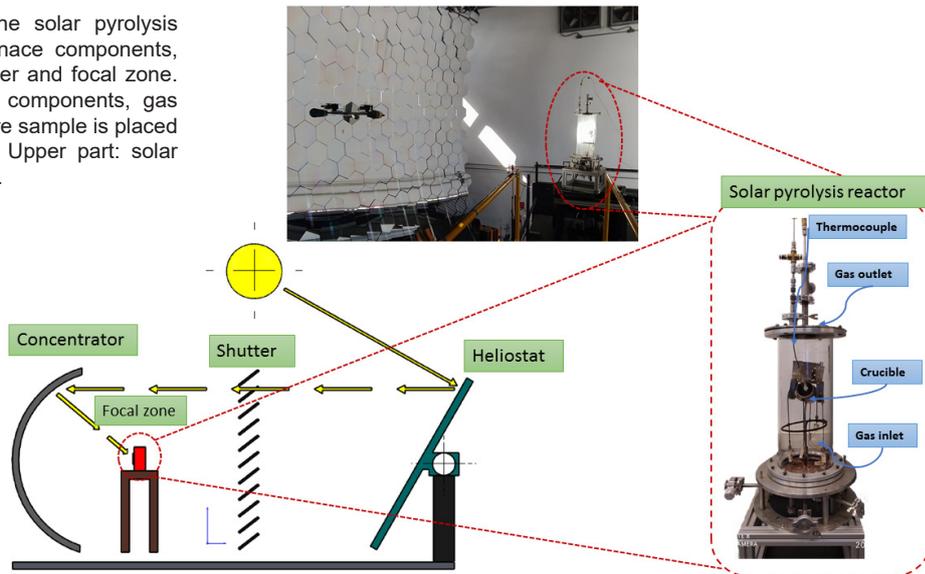
2.3 Solar pyrolysis

The pyrolysis process is often used to transform biomass into a variety of products. There are several ways of providing heat to the pyrolysis reactor, most of which involve the consumption of conventional fuels. This implies that a high-quality fuel is used to obtain a low quality fuel, therefore a pyrolysis process that obtains bio-oil using conventional fuels is not economically viable. Consequently, using concentrated solar energy to heat a reactor was proposed. After drying, solar pyrolysis was performed in a solar tubular reactor of 20 cm-diameter, 46 cm height. Inside the reactor there is an alumina crucible into which 2 g of *S. Fluitans* III sample was placed (Figure 2).

The pyrolysis reactor was heated using a concentrating solar system of the IER-UNAM (Solar Furnace). This furnace is composed of an heliostat of 81 m^2 that follows the sun and redirects the solar rays to the concentrator. The concentrator has 409 spherical mirrors that concentrate the solar rays at a focal distance of 3.68 m in a spot diameter of 8-9 cm. Between the solar concentrator and the heliostat, is a shutter that allows the amount of incident solar energy to be controlled and thus the temperature in the focal zone. (Fig. 3) The reactor is placed in the focal zone of the IER-UNAM Solar Furnace to reach reaction temperatures. At the beginning of the experiment, an Argon flow of 4 L/min was used for ten minutes to obtain a free-oxygen atmosphere. This flow was later reduced to 2 L/min during the experiment. The temperature was continuously monitored with a type K thermocouple. Solar pyrolysis experiments were performed at temperatures ranging from 250 °C to 800 °C for 1 hour with a heating rate of 20 °C/min to evaluate the impact of temperature in the process.

To perform a comparison of the char obtained with concentrated solar energy a pyrolysis in a tubular reactor heated by electric resistance was performed. The pyrolysis was conducted at 800 °C for 1 h at 22 °C/min heating rate. After pyrolysis, the biochar was washed with 1 M HCl to eliminate impurities, excess salts, and ashes. Subsequently, it was washed with distilled water until a pH of 7 was reached.

Figure 3. Schematics of the solar pyrolysis experiments. Left: Solar furnace components, Heliostat, concentrator, shutter and focal zone. Right: solar tubular reactor components, gas inlet and outlet, crucible where sample is placed and type "K" thermocouple. Upper part: solar pyrolysis reactor in operation.



3. Results

3.1. *S. Fluitans III* characterization

The CHNS-O elemental composition of *S. Fluitans III* indicates that this alga has the following composition: 26.83% carbon, 4.36% hydrogen, 1.17% nitrogen, 0.92% sulfur and 66.7% oxygen. *S. fluitans III* is 31.84% ash. The chemical EDS characterization shows the following composition: chlorine 29.76%, potassium 17.15%, calcium 10.78%, oxygen 28.09%, sodium 9.54%, sulfur 1.89% and magnesium 3.58%. The TGA analysis (Fig. 4) shows some relevant decomposition peaks: The first mass loss (14.24 %) corresponds to water and volatiles, observed from 61 °C to 221 °C. The largest mass loss (34.99 %) occurs from 221 °C to 580 °C and corresponds to the decarbonization and degradation of polymers. The last mass loss (28.94 %) is observed from 580 °C to

889 °C, and it is attributed to the decomposition of organic matter and the elimination of some metals and salts [9].

3.2 Solar drying

Figure 5a shows the kinetics of the sargassum drying. The drying time is 90 minutes for all cases. There are no significant differences in the drying time of open-drying (DSD) and greenhouse drying (GHD). Nevertheless, in the middle stage of the drying, between 30 - 60 min, there are significant differences in the drying times of the two treatments. Moisture loss in trays SF1 and SF3 was faster than in tray SF2 due the reduction of air velocity at this point in the GHD. Also, the moisture loss in trays SF1 and SF3 was faster than in the DSD due the temperature and wind speed.

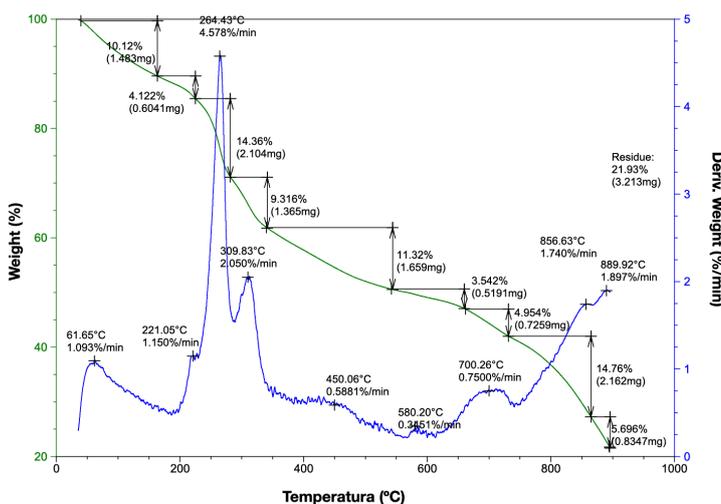


Figure 4. TGA analysis of *S. fluitans III*.

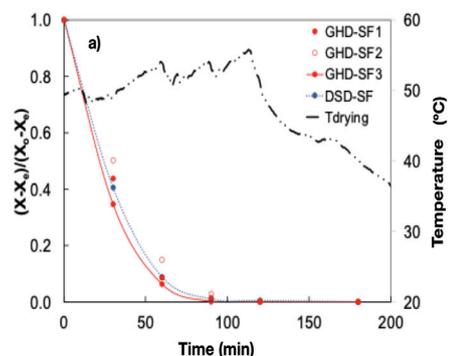


Figure 5. Drying kinetics of *S. Fluitans*

The average air velocity inside the GHD dryer was 1.7 m/s and the wind speed on the samples exposed directly to the sun was 2.1 m/s. The average of the relative humidity inside the solar dryer was 12.38% and outside was 32.83%. The drying of the sargassum exposed directly to the sun was favored by the speed

of the wind and the concrete on which the metallic trays were placed. The effective diffusivity of the GHD was greater than that of DSD ($1.82 \times 10^{-7} \text{ m}^2/\text{s}$, $1.413 \times 10^{-7} \text{ m}^2/\text{s}$, respectively) and both values are within the range reported for liquid diffusion. Therefore, the water migration occurs faster with GHD.

3.3 Pyrolysis

Figure 6 shows different magnifications of the micrographs of the biochar obtained at 800 °C in the electrical tubular reactor. They show a disordered surface with visible white spots, which could be attributed to the presence of inorganic elements. Magnification to 100k shows that the biochar also contains pores and slits. These channels favor biochar for a wide variety of applications, such as catalyst or adsorbent of contaminants, thanks to the favorable channels for ion transport [10]. The X-ray diffraction analysis of *S. fluitans* III biochar is shown in Figure 7a. There are two peaks, confirming that the material obtained is a carbon structure. The largest diffraction peak is around $2\theta = 25^\circ$, while the second is observed at 45° , corresponding to

graphite (PDF 00-041-1487). The amplitude of the two peaks confirms that the carbon obtained is amorphous. Thermogravimetric analysis (TGA) and differential thermal analysis (DTA) of *S. fluitans* III biochar are shown in Figure 7b. The first mass loss, around 50-150 °C (16.73%), can be attributed to the evaporation of moisture from the material. This mass loss shows an endothermic reaction as seen in the long? peak at this temperature. The second (3.97%) and third (2.47%) mass losses correspond to the decomposition of volatile materials. In Figure 6b the endothermic peaks corresponding to these losses can be seen.

Further mass loss above 400 °C did not produce endothermic reactions. Therefore, it can be concluded that a stable carbon material was obtained [11].

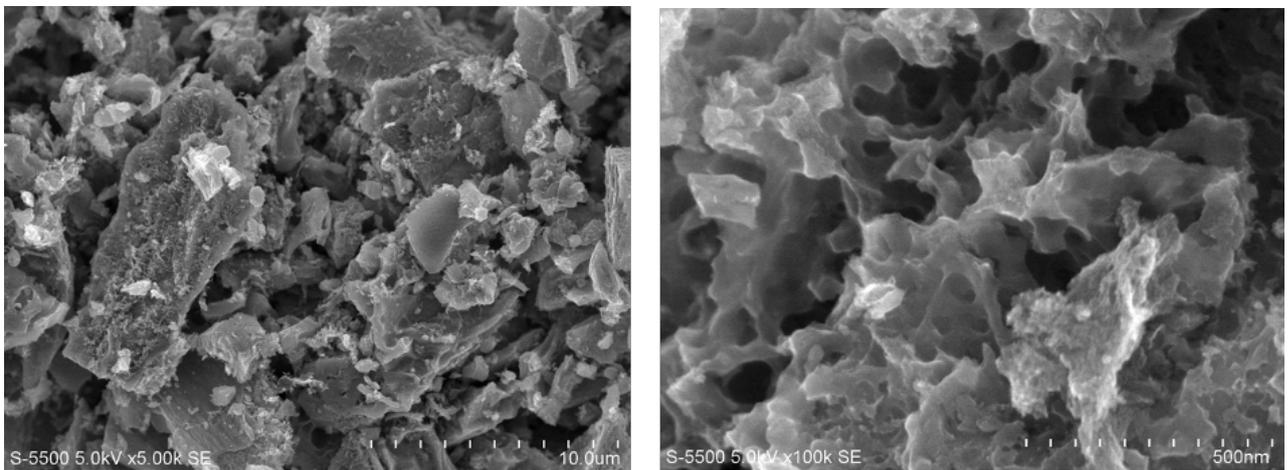


Figure 6. Micrographs of *S. fluitans* III biochar obtained at 800 °C with different magnifications.

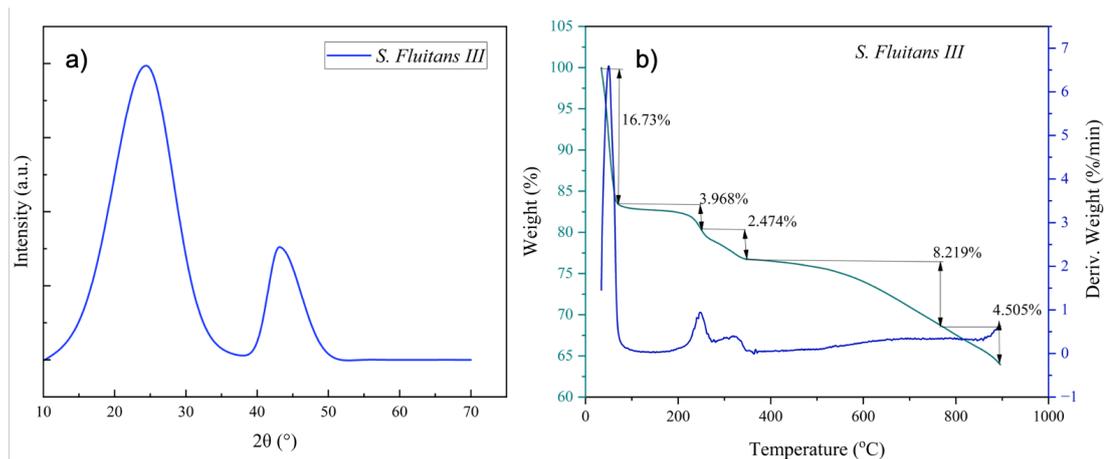


Figure 7. a) X-ray diffraction analysis of *S. fluitans* III biochar, b) TGA/DTG analysis of *S. fluitans* III biochar.

Solar pyrolysis was performed at different temperatures to assess the impact of temperature in the char yield and its main characteristics. Figure 8 shows the char yield and chemical conversion of each experiment. In this figure, it can be observed that an increase of temperature lowers the char yield from 77.2% to 29.5%, due to the higher decomposition of *S. fluitans* III. At 800 °C, there are more cracking reactions, increasing the gas production and reducing the char yield from 50.3% to 29.5%. The metals found in the *S. fluitans* III, such as

calcium and potassium, also favor tar disintegration reactions and char reforming, which increase the gas yield and reduce the char yield [12].

The results found in this work are consistent with those found in the literature. Farobie et al. [12] investigated the effect of temperature on the product distribution, where it is reported that at 400 °C, the the char yield is 55 %. When the temperature is 600 °C, the char yield falls to 30 %. In our experiments, the char yield at 400 °C is 60%, and when the temperature increases to 650 °C, the car yield decreases to 50%.

Two similar experiments were performed at 350 °C (Fig. 8). In this figure, minor differences, of 2% in both cases, in the char yield and chemical conversion are seen. These differences can be attributed to the minor variations in temperature that are obtained in the solar reactor, which are between 30 and 50 °C.

The micrographs of solar chars obtained at different pyrolysis temperatures are shown in Fig. 9. The raw biomass has a smooth textured wall. When the pyrolysis process is performed at 350 °C, the wall starts to break down, producing fissures. As the pyrolysis temperature increases, the structure of the raw biomass breaks more easily, resulting in a porous, amorphous structure. In the micrographs, from the EDS analysis, it is also possible to observe the inorganic compounds in the char surface (white zones of Fig. 9): chlorine, potassium, calcium, magnesium and sulfur.

The TG analysis of solar biochar is shown in Figure 10. The first mass loss at around 50-100 °C (9.47%) can be attributed to the evaporation of moisture from the material. The following mass losses (7.25 %, 5,72%, and 6.21 %) correspond to the decomposition

of volatile materials. However, above 700 °C, a larger mass loss is seen (33.43 %), which may be due to a catalytic effect of alkali (K and Na) and alkaline earth metals (Ca and Mg) remaining in the char structure [13]. It can therefore be concluded further studies to evaluate the catalytic effect of metals on the physical activation of *S. fluitans* III char are required.

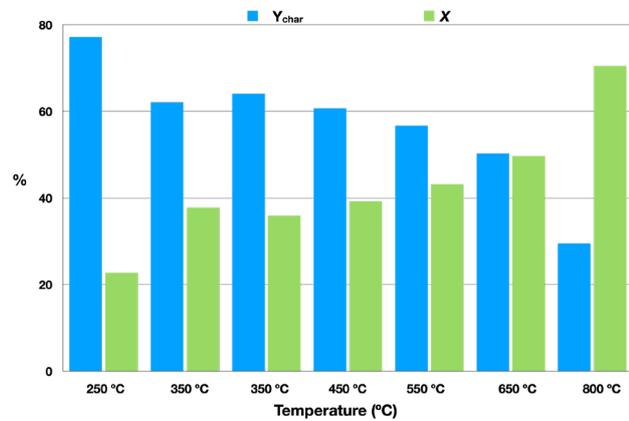


Figure 8. Solar pyrolysis experiments: temperature, char yield and chemical conversion.

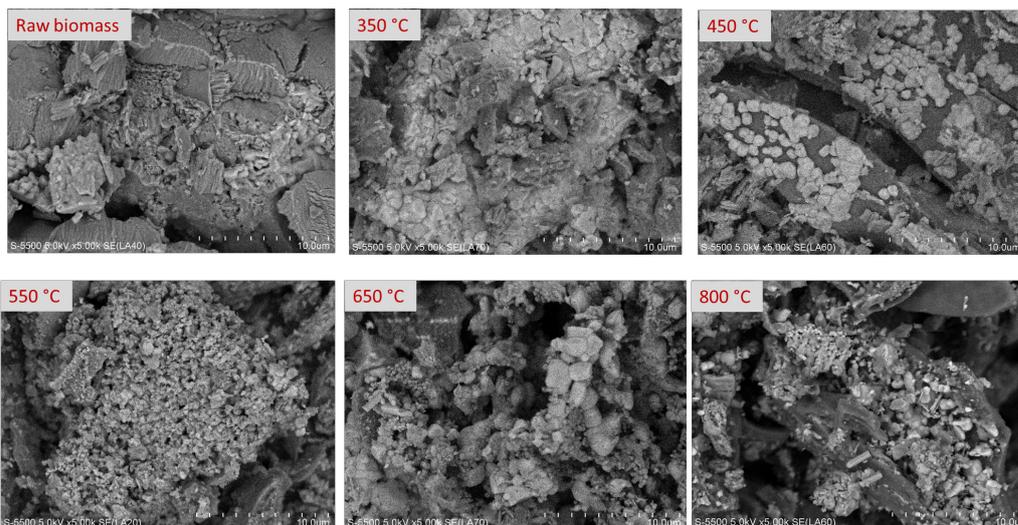


Figure 9. Micrographs of raw biomass (dry *S. fluitans* III) and solar biochar obtained at different temperatures.

The biochar obtained by both methods is amorphous and porous. Although the pores do not have a well-defined geometry, they can function as channels for ion transport or as active sites for energy storage. Inorganic components such as chlorine, potassium, calcium, sodium, magnesium and sulfur are also present. It is worth mentioning that at 800°C, a white deposit forms on the walls of the solar reactor. EDS analysis of this white deposit showed it to be composed of Cl, K, and Na, indicating that high temperatures promote the separation of some of the inorganic components of the biochar. This could be of interest for specific biochar applications. For instance, its porous, amorphous structure makes it ideal for water and air filtration systems, where the pores can adsorb contaminants. Biochar could also be used in energy storage technologies, such as supercapacitors or batteries, where its surface characteristics and ion adsorption capacity are crucial. Separating inorganic components,

such as chlorine, potassium, and sodium, at high temperatures could produce purer carbons, allowing advanced materials to be manufactured, or catalysts for industrial processes.

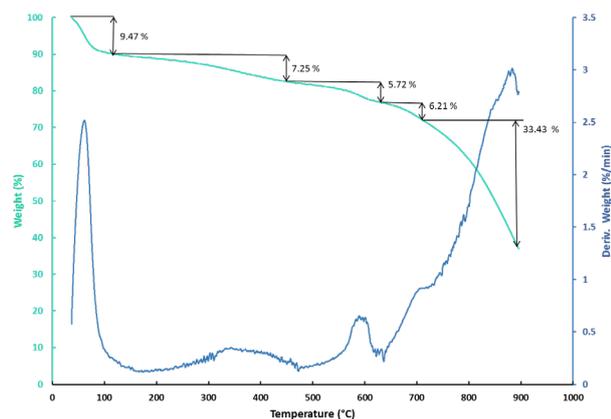


Figure 10. TGA/DTG analysis solar obtained biochar from *S. fluitans* III.

4. Conclusions

This work describes a solar treatment train for *S. fluitans* III, which consists of a two-step process: solar drying using a greenhouse-type solar dryer and pyrolysis of dried sargassum to produce biochar. This treatment train reduces the risk of sargassum decomposition and produces a value-added product, biochar, which could be used in energy storage technologies, such as supercapacitors or batteries or as biofertilizers due to the porosity and inorganic components present. The drying time was 90 minutes, whichever type of dryer is used for the conditions analyzed. In the case of pyrolysis, it was seen that increasing the temperature impacts the surface characteristics, and the carbon yield decreases, but a better quality biocarbon is obtained. The carbon obtained is amorphous, with inorganic components such as chlorine, potassium, sodium, magnesium, and sulfur. At 800 °C, the evaporation of some inorganic components is also observed, such as Cl, K, and Na, so there is a separation process at these temperatures.

Acknowledgements

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Impact of hydrothermal pretreatment in acidic medium on the performance of biomass-derived carbons for sodium-ion electrochemical storage

Impacto del pretratamiento hidrotérmico en medio ácido sobre el rendimiento de carbones derivados de biomasa en el almacenamiento electroquímico de iones de sodio

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Abstract

Hard carbons (HCs) have emerged as promising anode materials for sodium-ion batteries (SIBs) due to their ability to store sodium ions in surface functionalities, structural defects within the amorphous region, pseudographitic domains, and micropores. In this study, HCs were synthesized from vine shoots (VS) and waste hemp hurd (WHH) using a two-step process involving HCl-assisted hydrothermal pretreatment followed by carbonization at 800 °C or 1 000 °C. The resulting HCs demonstrated remarkable electrochemical performance in sodium-ion half-cells, achieving reversible specific capacities of up to 368 mAh g⁻¹ at a current density of 0.1 A g⁻¹. This excellent behavior is attributed to the appropriate physicochemical properties of the prepared HCs, particularly their hierarchical pore size distribution and tailored carbonaceous structure.

Resumen

Los carbones duros (HCs) han surgido como materiales prometedores para su uso como ánodos en baterías de iones de sodio (SIBs). Esto es debido a la capacidad de los HCs para almacenar iones de sodio en grupos funcionales en superficie, defectos estructurales dentro de la región amorfa, dominios pseudo-grafíticos y microporos. En este estudio, se sintetizaron HCs a partir de sarmientos de vid (VS) y residuos de cáñamo industrial (WHH) mediante un proceso en dos etapas que incluye un pretratamiento hidrotérmico asistido con HCl seguido de una carbonización a 800 °C o 1000 °C. Los HCs obtenidos demostraron un notable desempeño electroquímico en semiceldas de iones de sodio, alcanzando capacidades específicas reversibles de hasta 368 mAh g⁻¹ a una densidad de corriente de 0,1 A g⁻¹. Este excelente comportamiento se atribuye a las propiedades fisicoquímicas de los HCs producidos, particularmente a su distribución de tamaños de poro jerarquizada y a su apropiada estructura carbonosa.

1. Introduction

Sodium-ion batteries (SIBs) are emerging as leading candidates for the next generation of large-scale

electrochemical energy storage systems, essential for integrating intermittent renewable energy sources into the grid [1]. However, their successful deployment hinges on the development of high-capacity anode materials, as traditional graphite anodes exhibit limited sodium-ion storage capacity [2]. Hard carbons (HCs) have gained prominence as promising anode materials for SIBs, thanks to their ability to store sodium ions in surface functionalities, defects in the amorphous region, pseudographitic domains, and micropores [3]. Notably, the production of HCs from biomass waste is gaining attention due to its environmental benefits, cost-effectiveness, and potential to enhance reversible capacities, aligning with circular economy principles.

Sodium insertion into hard carbons primarily occurs in two stages during galvanostatic discharge (see Figure 1): (1) a slope region at relatively high voltage (above 0.15 V) and, (2) a plateau region at low voltage (below 0.15 V) [4]. While the precise mechanisms underlying these regions still remain under scientific debate, a growing consensus suggests that the low- and high-voltage regions are primarily attributed to adsorption-driven storage processes (involving surface charge storage, heteroatoms, and defects) and intercalation and pore filling processes, respectively. However, biomass-derived HCs still present challenges that limit their practical application, including low initial Coulombic efficiency (ICE), limited rate capability, and relatively poor cycling stability. The low ICE is commonly attributed to the formation of a thick solid electrolyte interphase (SEI), particularly when carbonates-based liquid electrolytes are employed, leading to substantial irreversible trapping of sodium ions [5]

Transforming a given biomass precursor into a high-value carbon material can be effectively achieved through hydrothermal pretreatment followed by high-temperature annealing. This approach has been proposed as a facile and environmentally friendly route for synthesizing carbonaceous materials from lignocellulosic feedstocks [6]. Hydrothermal pretreatment utilizes subcritical water as both a solvent and catalyst, enabling acid- and base-catalyzed reactions in biomass conversion. During hydrothermal treatment, cellulose and hemicellulose can hydrolyze above 180 °C into oligomers and

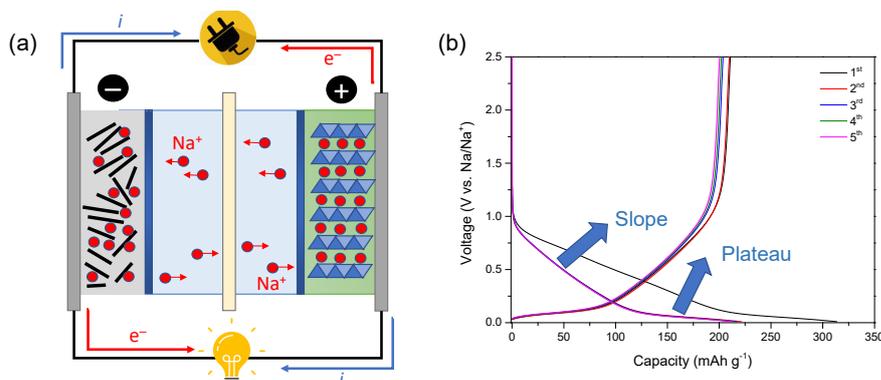


Figure 1. Simplified scheme of a sodium-ion battery (a) and typical galvanostatic charge-discharge curves for a HC-based electrode in a half-cell setup (b).

glucose, followed by dehydration, isomerization, and polymerization to form soluble products and hydrochar. Lignin, which is more resistant, partially degrades to phenolic compounds or undergoes solid-state transformations to polyaromatic hydrochar.

Compared with the direct carbonization synthesis pathway, the hydrothermal process fosters pore enlargement and the creation of nanosphere domains, which could enhance the reversible capacity of the resulting HC-based electrodes [7]. Furthermore, using an acidic medium can further catalyze the above-mentioned reactions in biomass precursors, leading to hydrochars with notable chemical, morphological, and structural changes. This opens the door to producing engineered HCs to be used for sodium-ion storage purposes.

This article summarizes the research activities conducted within our research group, focusing on the synthesis of hard carbons derived from two residual biomass sources (vine shoots and waste hemp hurd) via hydrothermal pretreatment in acidic medium (HCl) and their subsequent physicochemical characterization and electrochemical performance measurement in sodium-ion half-cells.

2. Experimental section

2.1. Production of hard carbons

Two biomass sources were used as carbon precursors: vine shoots (VS) and waste hemp hurd (WHH). VS (*Vitis vinifera* L) were collected in the wine region of Somontano (Huesca, Spain). The raw VS, generated during winter pruning, were sun-dried and crushed to achieve a particle size smaller than 0.7 mm. WHH (*Cannabis sativa* L.), produced in Narlisaray (Black Sea region of Turkey) as a by-product of the hemp defibration process, was sieved to particles approximately 5 mm in length. Results from proximate and elemental analyses, along with the determination of the main biomass constituents (extractives, hemicellulose, cellulose, and lignin) and the primary species present in ash are available in previous publications for VS [8] and WHH [9] samples.

Hard carbons were produced via a two-step carbonization approach. First, 15 g of VS or 5 g of WHH were hydrothermally treated in 50–60 g of a 2 mol dm⁻³ aqueous solution of HCl, for 12 h at 180 °C, using a PTFE-lined stainless-steel autoclave (Huanyu ZHT-172C, 100 mL volume). The material

was then collected via vacuum filtration and dried at 100 °C for 12 h. The resulting hydrochar was placed in a ceramic boat and carbonized at a heating rate of 5 °C min⁻¹ to final temperatures of 800 °C or 1 000 °C. A tubular reactor made of mullite, inserted into a furnace (Carbolite TF1 16/60/300), was used under an Ar atmosphere. The resulting HCs were ground into powder (particle size below 90 μm), washed with a 2 mol dm⁻³ HCl solution for 2 h at room temperature, and rinsed with DI water until neutral pH was achieved [10,11].

2.2. Physicochemical characterization of hard carbons

The morphologies of the HCs were examined by an Inspect-F50A scanning electron microscope equipped with energy dispersive X-ray spectroscopy (SEM-EDX; FEI, The Netherlands). Further analysis of the HCs structure was conducted with a Tecnai F30 high-resolution transmission electron microscope (HR-TEM, FEI) operated at 300 kV. Interplanar distances were determined from HR-TEM images using Digitalmicrograph® software.

Structural properties were analyzed via X-ray powder diffraction (XRD; Empyrean, Malvern Panalytical, UK, $\lambda = 0.154$ nm) and Raman spectroscopy (Alpha 300, WITec, Germany, $\lambda = 532$ nm). The interlayer spacing between graphene layers (d_{002}), the apparent thickness of crystallites along the c-axis (L_c), and the apparent width of crystallites along the a-axis (L_a) were calculated from XRD spectra utilising Bragg's law and Scherrer's equation. Raman spectra were deconvoluted into five peaks, comprising one Gaussian-shaped band (D3) and four Lorentzian-shaped bands (G, D1, D2, and D4). The peak area ratio (A_{D1}/A_G) was calculated as a measure of structural disorder.

Specific surface areas and pore size distributions were estimated from N₂ and CO₂ adsorption isotherms at -196 °C and 0 °C, respectively, following outgassing under vacuum at 150 °C for 8 h. Measurements were conducted using an Autosorb-iQ analyser (Quantachrome, Germany). Specific surface areas (SSA) were calculated using the Brunauer-Emmett-Teller (BET) equation, while pore size distributions were determined using Non-Local Density Functional Theory (NLDFT) models for slit pore geometries. All the calculations were conducted with QuadraWin 6.0 software.

2.3. Electrochemical measurements

The electrochemical performance of HCs was evaluated using three-electrode Swagelok t-cells. The working electrode (WE) comprised the respective HC, carbon black Super P™ as a conductive agent, and sodium carboxymethyl cellulose (Na-CMC) as a binder, with mass fractions of 0.8, 0.1, and 0.1, respectively. A uniform slurry was prepared by mixing these components with DI water under magnetic stirring. The slurry was then uniformly applied onto a high-purity aluminum current collector (16 μm thickness) using a baker applicator to form a composite electrode with a thickness of 100 μm . Finally, 12 mm diameter electrodes were punched and dried under vacuum at 120 $^{\circ}\text{C}$ for 12 h [12].

The Swagelok t-cells were assembled in an Ar-filled glovebox (Mbraun, Germany) with O_2 and H_2O contents below 0.5 ppm. Two sodium metal discs, with diameters of 12 mm and 5 mm, were used as the counter and reference electrodes, respectively. Two glass fiber filters (190 mm thickness, Prat Dumas, France) served as separator. The electrolyte (200 mL) consisted of a 1 mol dm^{-3} NaTFSI solution in a 1:1 (vol. ratio) mixture of dimethyl carbonate (DMC) and ethylene carbonate (EC).

Electrochemical measurements were performed using a potentiostat-galvanostat (SP-200, Bio-Logic, France) at room temperature. Galvanostatic charge discharge (GCD) measurements were carried out within a potential window of 0.01–2.5 V (vs. Na^+/Na).

3. Results and discussion

3.1. Physicochemical properties of hard carbons

Figure 2 displays SEM images of the produced hard carbons, which have been denoted as function of the precursor (VS or WHH), hydrothermal medium (water, *w*, or *HCl*), and highest annealing temperature (800 or 1 000 $^{\circ}\text{C}$). As observed in Figure 2, all the produced HCs preserved the biomass' structural voids and channels, likely due to the relatively high lignin contents (20.3 % [8] and 23.8 % [9] by mass for VS and WHH, respectively). This preservation may enhance electrolyte penetration and sodium-ion diffusion, potentially improving electrochemical performance. Additionally, HCl-assisted hydrothermal pretreatment led to carbon surfaces with rougher textures and smoother contours, attributed to the promoted hydrolysis of soluble compounds. When HCs were produced at a highest temperature of 1 000 $^{\circ}\text{C}$, randomly distributed microsphere domains—particularly in WHH-HCl-1000—were observed.

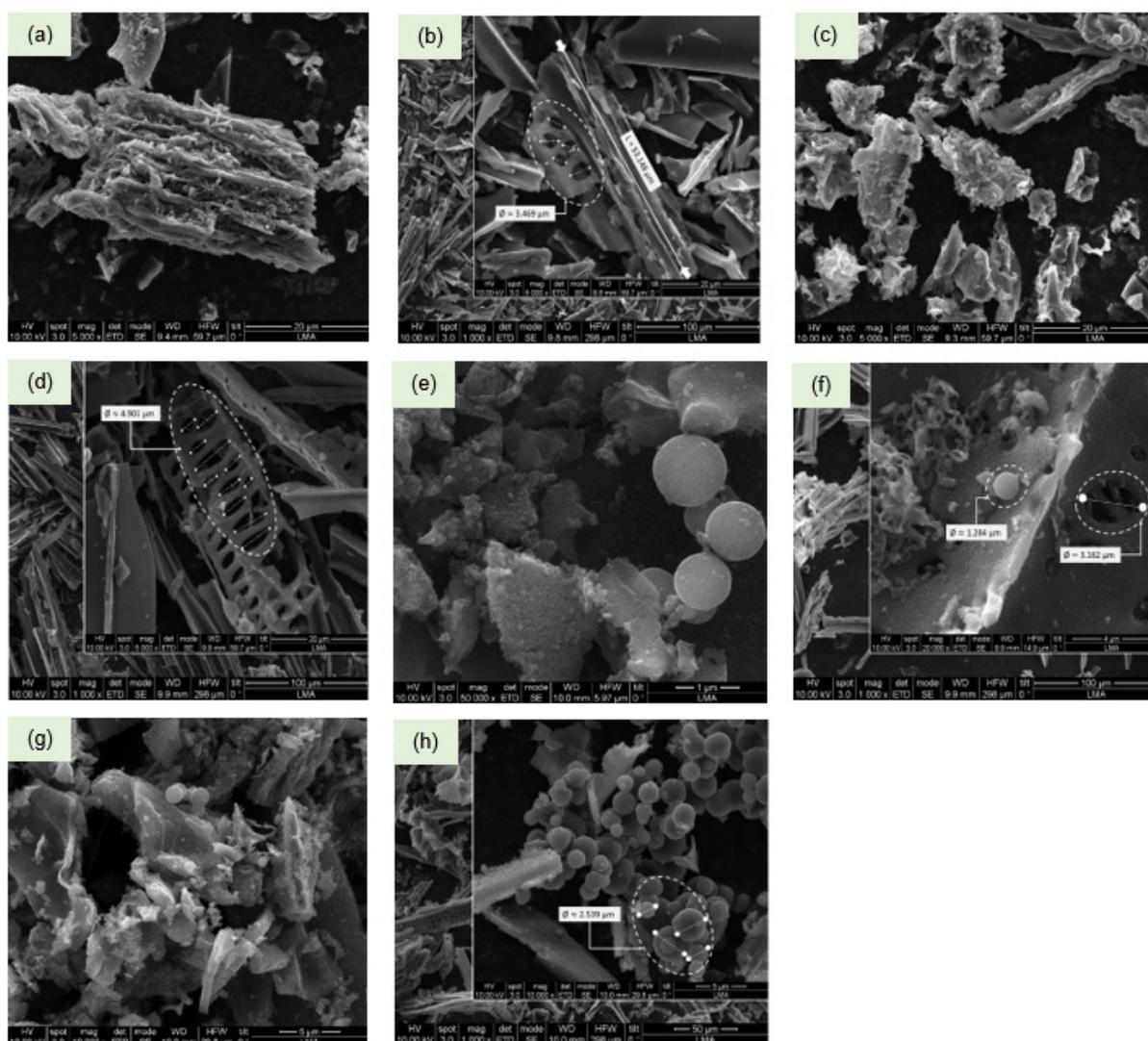


Figure 2. SEM images of VS-w-800 (a), WHH-w-800(b), VS-w-1000 (c), WHH-w-1000 (d), VS-HCl-800 (e), WHH-HCl-800 (f), VS-HCl-1000 (g), and WHH-HCl-1000 (h).

The acquired HR-TEM images (see Figure 3) revealed that the hard carbons obtained through hydrothermal pretreatment with pure water exhibited a predominantly amorphous structure with randomly oriented pseudographitic domains. Furthermore, d_{002} values exceeding 0.36 nm were observed, enabling Na-ion intercalation. An increase in the temperature from 800 °C to 1000 °C enhanced the formation of curved graphene-like layers around closed or quasi-closed pores, which are hypothesized to improve the

specific capacity of the active material [13]. When HCl was employed during the hydrothermal pretreatment, similar or even wider interlayer spacings—ranging from 0.41 nm to 0.49 nm (as shown in Figures 3e, 3f, and 3h)—were observed. Following annealing at 1 000 °C, while amorphous regions with turbostratic domains persisted ($d_{002} \approx 0.40$ nm), other local domains underwent significant ordering, resulting in fully graphitic zones with d_{002} values below 0.30 nm (see Figure 3g).

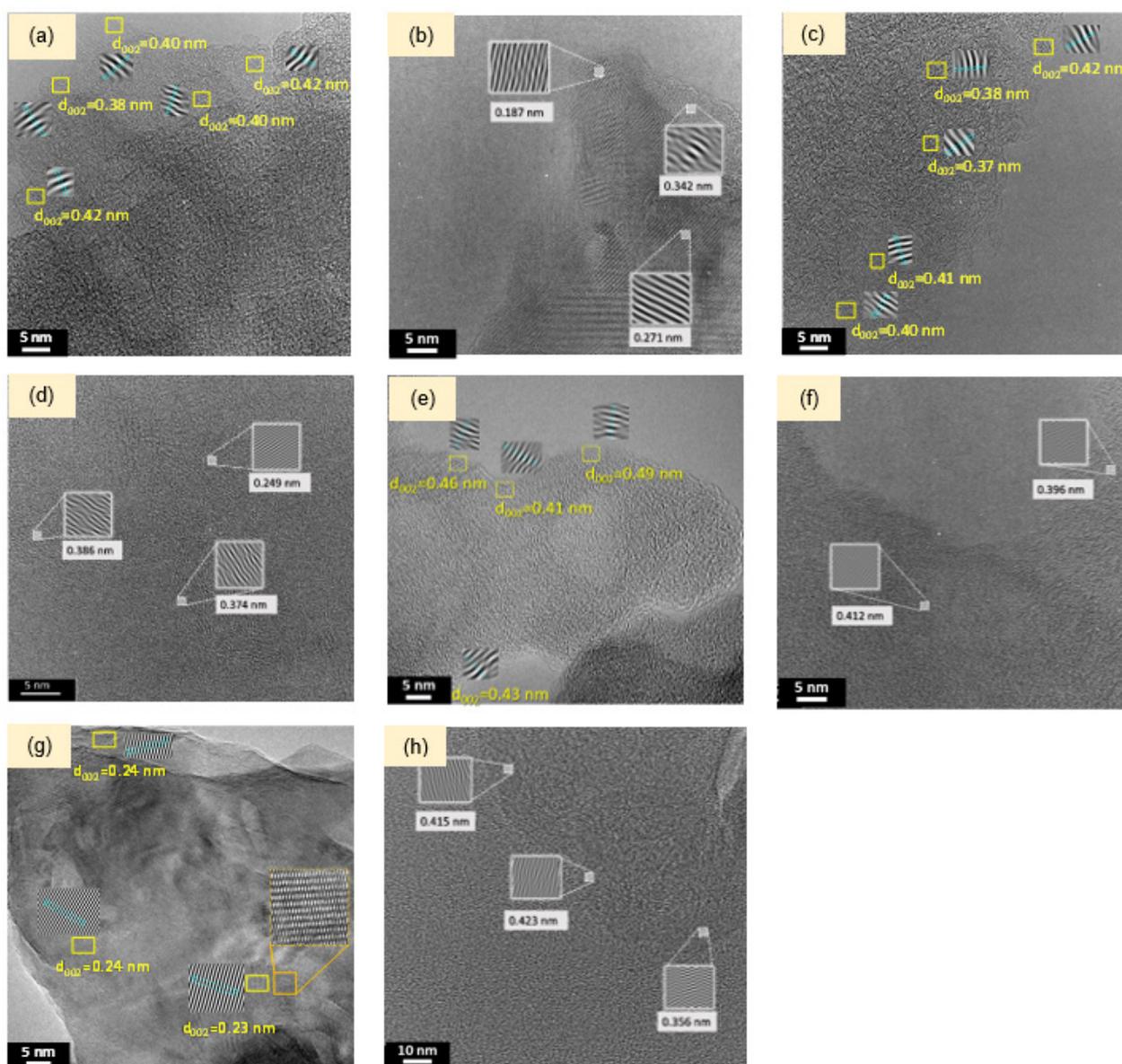


Figure 3. HR-TEM images of VS-w-800 (a), WHH-w-800(b), VS-w-1000 (c), WHH-w-1000 (d), VS-HCl-800 (e), WHH-HCl-800 (f), VS-HCl-1000 (g), and WHH-HCl-1000 (h).

Despite the differences observed in the structural ordering on surface, results from XRD and Raman spectroscopy (see Table 1) indicate that the degree of disorder for bulk materials was relatively similar among them, except for VS-w-800, which appears to exhibit a high degree of disorder. For VS-derived HCs, however, a slight increase in structural order is deduced in carbons annealed at higher temperature and/or hydrothermally treated with HCl instead of water.

Regarding the textural features of hard carbons, Table 2 lists the specific surface areas and pore volumes determined from N_2 and CO_2 adsorption isotherms. The specific surface areas significantly increased when the hydrothermal pretreatment was performed in an acidic medium. For VS-derived carbons, relatively high mesopore volumes were obtained at both annealing temperatures. These hierarchical pore size distributions could shorten the diffusion paths for Na-ions and enhance kinetics [14].

Table 1. Structural parameters of hard carbons deduced from XRD and Raman analyses.

Material	From XRD			From Raman
	d_{002} (nm)	L_c (nm)	L_s (nm)	A_{D1}/A_G
VS-w-800	0.385	0.887	2.875	8.05 ± 0.04
WHH-w-800	0.388	0.895	3.508	4.79 ± 0.30
VS-w-1000	0.381	0.874	3.822	5.69 ± 0.22
WHH-w-1000	0.381	0.874	4.009	5.22 ± 0.46
VS-HCl-800	0.374	0.922	3.334	5.44 ± 0.41
WHH-HCl-800	0.385	0.880	3.300	4.05 ± 0.55
VS-HCl-1000	0.373	1.011	3.784	4.76 ± 0.25
WHH-HCl-1000	0.383	0.868	3.451	5.52 ± 0.50

Table 2. Textural features deduced from N₂ and CO₂ adsorption isotherms.

Material	S_{BET}^a (m ² g ⁻¹)	S_{BET}^b (m ² g ⁻¹)	Micropore volume ^c (cm ³ g ⁻¹)	Mesopore volume ^c (cm ³ g ⁻¹)	Ultramicropore volume ^d (cm ³ g ⁻¹)
VS-w-800	82.0	397	0.080	0.025	0.122
WHH-w-800	61.7	388	0.031	0.007	0.180
VS-w-1000	17.0	445	0.062	0.030	0.127
WHH-w-1000	11.1	402	0.006	0.011	0.186
VS-HCl-800	659	575	0.295	0.240	0.157
WHH-HCl-800	462	423	0.178	0.030	0.196
VS-HCl-1000	127	440	0.079	0.212	0.127
WHH-HCl-1000	95.6	428	0.058	0.011	0.199

^a From N₂ adsorption isotherm at -196 °C. ^b From CO₂ adsorption isotherm at 0 °C. ^c From N₂ adsorption data using a NLDFT model. ^d From CO₂ adsorption data using a NLDFT model.

3.2. Electrochemical performance of hard carbon-based anodes

Table 3 presents the charge specific capacities and the corresponding ICE values derived from GCD measurements (over the first five cycles) at a relatively low current density (0.1 mA g⁻¹). The WHH-HCl-1000-based electrode exhibited excellent performance, achieving a specific capacity of 368 mAh g⁻¹ in the fifth cycle. This value is significantly higher than those reported at the same current density for other biomass-derived HCs prepared via hydrothermal pretreatment and thermal annealing

[15,16]. The remarkable capacities and relatively high ICE value achieved for WHH-HCl-1000 can be attributed to its structural and textural characteristics, which offer numerous active sites for reversible Na-ion storage through adsorption on defects, micropore filling, and intercalation. The low ICE values measured for the rest of carbons obtained via HCl-assisted hydrothermal pretreatment can be ascribed to the presence of relatively high volumes of mesopores and/or wide micropores, which promote the formation of thick SEI layers when carbonate-based electrolytes are employed [12].

Table 3. Charge specific capacities (mAh g⁻¹) and ICE values from the first five discharge-charge cycles at 0.1 mA g⁻¹.

Material	Capacity at cycle 1	Capacity at cycle 2	Capacity at cycle 3	Capacity at cycle 4	Capacity at cycle 5	ICE (%)
VS-w-800	162	160	157	153	149	56
WHH-w-800	101	99	98	97	96	48
VS-w-1000	246	243	239	237	236	71
WHH-w-1000	212	200	191	187	185	75
VS-HCl-800	192	188	187	186	183	34
WHH-HCl-800	162	161	160	159	158	39
VS-HCl-1000	167	164	161	159	158	39
WHH-HCl-1000	385	381	372	368	368	69

Regarding the rate capability of the HC-based electrodes, Figure 4 displays the charge specific capacities derived from GCD measurements conducted at current densities of 0.1, 0.5, 1, and 2 A g^{-1} . Among the carbons synthesized from VS, VS-HCl-800 exhibited the highest rate capability, with reversible capacities of 146, 126, and 102 mAh g^{-1} when cycled at 0.5, 1, and 2 A g^{-1} , respectively. This finding can be attributed to the hierarchical pore size distribution of this material, which includes a relatively high mesopore volume (see Table 2). In contrast, the best-performing material at 0.1 A g^{-1} (WHH-HCl-1000) showed a significant decline in charge capacity when cycled at higher current rates, delivering 156, 115, and 83 mA h g^{-1} at 0.5, 1, and 2 A g^{-1} , respectively.

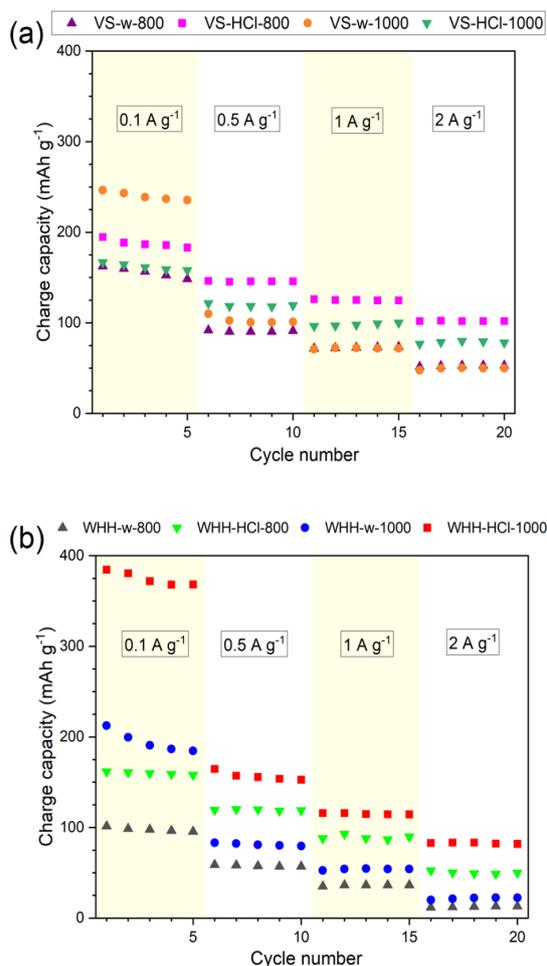


Figure 4. Charge specific capacities vs. cycle number at various current densities ranging from 0.1 to 2 A g^{-1} for (a) VS-derived HCs, and (b) WHH-derived HCs.

To evaluate the cycling stability of VS-HCl-800- and WHH-HCl-1000-based electrodes, 300 discharge-charge cycles were performed at 1 A g^{-1} , following an initial set of five cycles at 0.1 A g^{-1} . As shown in Figure 5, both electrodes exhibited a persistent decline in charge capacity over the cycles, likely due to the extent of parasitic reactions and/or the relatively thick and nonuniform SEI [17]. Nevertheless, upon restoring the current rate to its initial value (0.1 A g^{-1}), the WHH-HCl-1000 electrode demonstrated a satisfactory capacity retention of 91 % relative to

the values measured during the first five cycles at the same current rate. In contrast, the VS-derived electrode exhibited a lower retention of 79 % under the same conditions.

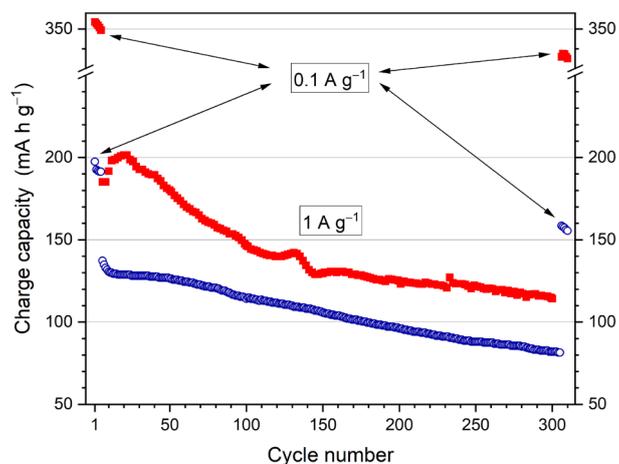


Figure 5. Cycling performance of VS-HCl-800 (blue circles) and WHH-HCl-1000 (red squares) at 1 A g^{-1} .

4. Conclusions

The results of this study confirm the potential of hard carbons derived from two waste biomass sources as anode materials for SIBs. Moreover, the proposed synthesis pathway presents a promising alternative to other approaches reported in the literature, offering advantages in terms of energy consumption and scalability.

The superior performance of the hard carbons pretreated hydrothermally in a hydrochloric acid medium can be attributed to their hierarchical pore size distribution, which facilitates Na-ion diffusion, and their appropriate carbon structure. This structure combines a highly disordered amorphous phase (with enlarged interlayer spacings) with highly graphitized microdomains, providing numerous active sites for Na-ion intercalation and simultaneously enhancing ionic conductivity.

Nevertheless, further research is still required to address the limitations identified in the present study, particularly the relatively poor cycling stability observed for the HC-based electrodes. In this regard, enhancing the performance of the electrode-electrolyte interphase is essential and could be achieved by developing more efficient electrolyte formulations.

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3D porous carbon structures derived from whey: manufacturing processes and applications

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Objectives and novelty

Producing Porous Carbon Structures (PCS) with complex designs and customized morphologies is not a straightforward task. Over decades of research on porous carbons, significant progress has been made in controlling nanoscale parameters such as porosity and surface chemistry. However, at the macroscopic scale, porous carbons are still presented in traditional formats such as powders, granules or pellets. Monoliths are also a recurrent form for porous carbons, but usually shaped in very simple geometries.

This doctoral Thesis aims to address three main objectives. The first one is the development of PCS with tailored morphologies that overcome current design and production limitations. Moreover, these structures must exhibit sufficient consistency to allow handling, storage, and transportation. In this work, the goal is to achieve this purpose through simple methodologies, using sustainable precursors and employing straightforward technologies mainly based on Additive Manufacturing (AM). The second objective is to utilize the fabricated PCS in applications where their geometric design provides advantages over the use of simpler morphologies. The ability to potentially obtain PCS with any morphology opens up possibilities for their use in applications previously unconsidered, such as tissue engineering or process intensification in chemical engineering. Finally, the third objective is to contribute to the valorization of whey and, consequently, reduce the environmental impact of this dairy by-product, which is generated in large quantities and poses significant management challenges.

Results

Whey possesses a set of characteristics that enable the fabrication of structures with controlled morphologies, either through the sintering of whey powder or from aqueous pastes [1]. For this purpose, different methods can be used, including molding [2], machining [3], and additive manufacturing techniques, such as Selective Laser Sintering [4] and Direct Ink Writing [5] (Fig. 1). These structures can then be transformed into PCS through carbonization and activation processes. During thermal treatment, whey exhibits thermally stable behavior, ensuring that the final PCS retain the same morphology as the original whey pieces. The only deformation experienced during this process is an isotropic

shrinkage of a known magnitude, allowing for precise control over the exact dimensions of the final product (Fig. 2a).

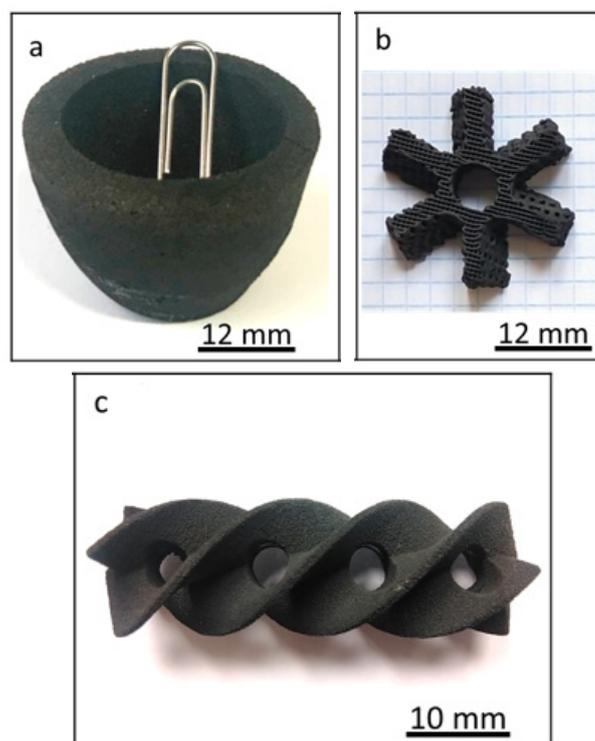


Figure 1. Examples of PCS obtained by casting and machining of whey powders (a), Direct Ink Writing (b) and Selective Laser Sintering (c).

PCS derived from whey are highly porous materials (ranging from 50% - 70% of porosity) with a hierarchical porosity that includes micropores, mesopores, and macropores (Fig. 2b), with BET surface areas up to 500 m²/g. This porosity can be increased by activation processes, obtaining as a result activated carbons with BET values higher than 1000 m²/g [6]. They also contain a significant amount of nitrogen in the range of 2% to 3%. Depending on the mineral content of the primary whey, the carbonized structures exhibit varying ash content, which can be easily reduced or removed through acid washing. However, their most notable property is the exceptionally high mechanical strength of the porous carbon components, as demonstrated by abrasion, flexural, and compression strength tests.

The freedom of design enables their use in innovative applications previously unexplored in the field of porous carbons. For instance, they can be employed in the development of bone scaffolds for tissue engineering [7]. In chemical engineering, they

can be applied to process intensification through the fabrication of catalytic agitators or continuous porous carbon reactors with optimized geometries [8,9].

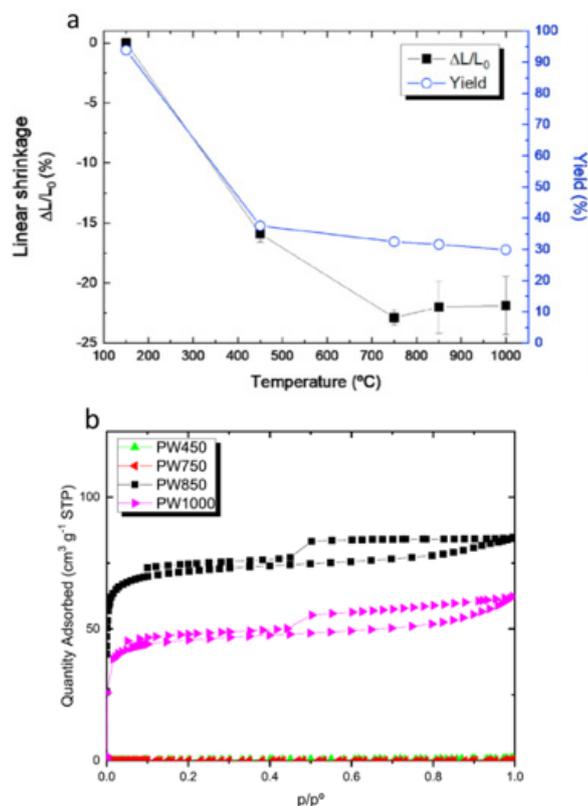


Figure 2. Shrinkage and yield (a) and Nitrogen isotherms (b) of PCS at different carbonization temperatures.

In addition to being used independently, whey can be combined with other powdered materials for the fabrication of composite structures, PCS or pellets [10]. This can be achieved via dry processing, by mixing powders followed by sintering, or via wet processing, by forming mixed aqueous pastes. Using the latter method, whey pastes can serve as binders for the production of activated carbon pellets. Specifically, the binder can be made directly from liquid whey. This provides a more sustainable and cost-effective alternative to traditional binders, mostly pitch and bentonite, as well as an opportunity for the industrial-scale valorization of liquid whey, processing thousands of tons.

Conclusions

- It is possible to obtain PCS with controlled morphologies using whey as a precursor. On one hand, its composition, primarily based on lactose and proteins, enables the formation of 3D whey structures through techniques such as molding or additive manufacturing, whether using whey powder or aqueous pastes. On the other hand, its thermally stable behavior allows pre-formed components to be carbonized without losing their shape, resulting in PCS with controlled and customized morphologies. Once fabricated, they can be easily machined, expanding the range of achievable geometries.

- Beyond morphological control, the most notable property of whey-based PCS is their high mechanical strength. Considering that they are porous materials, various mechanical tests (abrasion index, flexural strength, and compression strength) reveal greater consistency compared to other porous materials, including activated carbons, extruded cordierite, sintered hydroxyapatite, or trabecular bone. They also exhibit higher consistency than most carbons with similar porosities reported in the literature.
- The combination of whey with other powdered materials enables large-scale valorization of liquid whey, which poses significant environmental management challenges, offering a sustainable and cost-effective alternative to traditional binders such as pitch or bentonite.
- The ability to endow ECPs with complex, consistent morphologies—hard to obtain before the arrival of AM—allows their use in applications previously unexplored for porous carbons, such as the fabrication of bone scaffolds. Additionally, morphological control provides advantages in other applications, such as process intensification in chemical engineering through the development of catalytic agitators or the design of reactors with optimized geometries.

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Development of a sorption-enhanced methanation (SEM) process for the production of synthetic natural gas

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Objectives and novelty

The development of a sustainable energy system based on the efficient use of natural resources and a minimal carbon footprint has gained significant attention in recent years. This approach aims to replace fossil-based technologies with renewable material alternatives. In this context, the production of methane or synthetic natural gas (SNG) from renewable resources offers a viable alternative for energy storage while reducing emissions associated with fossil fuels. This thesis investigates the technological feasibility of a Sorption Enhanced Methanation (SEM) process for the production of high quality SNG. In the SEM process, the water produced as a by-product of the methanation reaction is removed from the reaction medium using porous materials such as zeolites, shifting the reaction equilibrium towards CH_4 production ($\text{CO} + 3\cdot\text{H}_2 \leftrightarrow \text{CH}_4 + \text{H}_2\text{O}$ and/or $\text{CO}_2 + 4\cdot\text{H}_2 \leftrightarrow \text{CH}_4 + 2\cdot\text{H}_2\text{O}$). When the adsorbent material becomes saturated and unable to adsorb further water, it is regenerated by variations in pressure and/or temperature for cyclic use. This method enables high CH_4 purity under milder temperature and pressure conditions and in a smaller number of stages than conventional methanation, simplifying the process and increasing overall efficiency. These features make the SEM process highly effective for SNG production, which could be stored without restriction in the current natural gas transportation network.

The objective of this thesis was to develop, investigate, optimise and scale up the SEM process using commercial catalysts and zeolites for different applications. Due to its versatility, the work explores the SEM process in two scenarios, investigating different feed gas mixtures: (i) $\text{H}_2/\text{CO}/\text{CO}_2$ mixtures simulating syngas from biomass gasification process, representing a biomass-to-SNG pathway; and (ii) H_2/CO_2 mixtures obtained from renewable H_2 via water electrolysis and CO_2 captured from industrial processes or air, representing an e-SNG production pathway. The SEM process was examined through two distinct technological developments: initial investigations were conducted in a micro fixed-bed reactor at lab scale (2 gr of material) coupled to a mass spectrometer [1], followed by testing in a pressurised TRL-3 fixed-bed reactor (100 gr of material), where the gas was analysed using a micro-chromatograph [2].

Results

Firstly, various catalysts and zeolites, as functional materials, were independently evaluated under relevant conditions for the SEM process to identify those with optimal. Ni, Rh and Ru-based catalysts were evaluated in a fixed bed microreactor at a laboratory scale on methanation activity over a temperature range of 200-300 °C for CO and CO_2 methanation at different gas space velocities. Ni-based catalyst demonstrated the highest activity achieving 80.1% of CO_2 conversion (X_{CO_2}) and 23.3 %vol. CH_4 purity at 250 °C with a gas space velocity of 1 $\text{kg}_{\text{CO}_2}/\text{kg}_{\text{cat}}\cdot\text{h}$. Concerning zeolites, zeolites 3A, 4A, 5A and 13X were texturally and physico-chemically characterised to evaluate their H_2O adsorption performance by thermogravimetric analysis (TGA). All the zeolites showed a higher affinity for H_2O than for CO_2 due to their hydrophilic nature. Zeolite 4A demonstrated the highest H_2O adsorption capacities (0.12 $\text{g}_{\text{H}_2\text{O}}/\text{g}_{\text{zeolite}}$ at 250 °C), which was attributed to its larger specific surface (S_{BET}) and micropore volume ($V_{\text{micropore}}$) [1].

Based on this study, different zeolite/catalyst mixtures were evaluated under SEM conditions in a micro-fixed bed reactor. The SEM process was initially demonstrated using a commercial Rh-based catalyst combined with zeolite 5A in a 1/5 mass ratio (catalyst/zeolite). While the Rh catalyst showed good activity under conventional methanation, its performance improved significantly under SEM conditions for H_2/CO , H_2/CO_2 and $\text{H}_2/\text{CO}/\text{CO}_2$ feed gas mixtures. The best outcomes were obtained for a H_2/CO_2 mixture at 275 °C and 0.58 $\text{kg}_{\text{CO}_2}/\text{kg}_{\text{cat}}\cdot\text{h}$. Under these conditions, during the pre-breakthrough period, as the zeolite adsorbed the H_2O produced, complete conversion of H_2 and CO_2 was achieved, producing CH_4 with 100 vol%. purity compared to 22.9 vol%. for conventional methanation. The zeolite adsorbed 0.14 $\text{g}_{\text{H}_2\text{O}}/\text{g}_{\text{zeolite}}$ during this stage. Once the zeolite was saturated, the gas composition attained in the post-breakthrough period was the same as under pure methanation conditions. In addition, the performance of the SEM was tested using a $\text{H}_2/\text{CO}_2/\text{CO}$ mixture as feed gas, a novel approach in the literature. At 250 °C and 1.1 $\text{kg}_{\text{CO}_2}/\text{kg}_{\text{cat}}\cdot\text{h}$, the CH_4 content achieved during the pre-breakthrough period was 59.4 vol%, with H_2 being the main product impurity [3].

After demonstrating the potential of the SEM process, the performance of the most extensively studied zeolites in the literature (i.e. 3A, 4A, 5A and 13X) was assessed under SEM conditions using a Rh-based catalyst under CO_2 SEM conditions at 275 °C and 0.58 $\text{kg}_{\text{CO}_2}/\text{kg}_{\text{cat}}\cdot\text{h}$. The result showed a

significant improvement in CH₄ purity from 8.3 vol% under conventional methanation to around 70 vol% under SEM conditions, with XCO₂ increasing from 38.4 % to 90.2 %. However, the duration of the pre-breakthrough period varied significantly between the zeolites: 2.8 min, 17.3 min, 6 min and 16.2 min for the zeolites 3A, 4A, 5A and 13X respectively, with 3A and 5A exhibiting the shortest durations. In order to identify the optimum combination, Rh/4A and Rh/13X were tested at 275 °C and 0.45 kg_{CO₂}/kg_{cat}·h. Under these conditions the Rh/4A combination presented higher X_{CO₂} (94.5 %), CH₄ content (85 %) and H₂O uptake capacity (0.142 g_{H₂O}/g_{zeolite}) than the Rh/13X combination. These differences were attributed due to CO₂ adsorption on the zeolite 13X. Finally, long-term stability tests confirmed consistent Rh/4A performance over multiple SEM/ regeneration cycles, with effective regeneration achieved by heating until 400 °C [1].

In parallel, the reaction mechanisms for three commercial catalysts (Ru, Rh and Ni as active phases) were investigated under conventional methanation and SEM conditions using *in-situ* Fourier Transform Infrared (FTIR) technique. This analysis confirmed the superior catalytic activity of the Ni-based catalyst compared to Rh and Ru catalysts during CO₂ conventional methanation. Based on the detected surface intermediates, a reaction mechanism involving formate and CO as precursors for CH₄ formation was proposed. With Ni identified as the most active catalyst, SEM tests were carried out with Ni/4A and Ni/13X combinations. For Ni/4A, CH₄ and a small proportion of CO₂ were detected in the gas phase at the beginning of the SEM reaction, while H₂O appeared at a later event indicating its initial adsorption on the zeolite. In contrast, for Ni/13X, these phases were indistinguishable due to the chemisorption of CO₂ on zeolite 13X before the reaction started. This CO₂ adsorption on 13X was further confirmed during the regeneration stage by raising the temperature to 400 °C [4].

Based on the results obtained at the laboratory scale, the combination of zeolite 4A and Ni-based catalyst was identified as the most appropriate for evaluating the performance of the SEM process in a lab-scale TRL-3 fixed bed reactor. A comprehensive optimisation of the different process stages was carried out to maximise CH₄ productivity and purity and the H₂O adsorption capacity of the zeolite under moderate pressure and temperature conditions. Operating at 250 °C, 10 bar and 1.4 kg_C/kg_{cat}·h with a H₂/CO/CO₂ mixture, the process achieved pure CH₄ during 25 min. Different zeolite regeneration methods were evaluated to facilitate cyclic operation, with Pressure Swing Adsorption (PSA) selected as the most suitable and efficient method in terms of energy saving, allowing successive SEM/regeneration cycles to be performed [2]. In addition, a parametric study of the SEM process was carried out using both H₂/CO/CO₂ and H₂/CO₂ feed gas mixtures. In both cases, the influence of several process variables was determined, such as the effect of th

e H₂/CO/CO₂ and/or H₂/CO₂ ratio, pressure, temperature and the addition of light hydrocarbons such as CH₄ and C₂H₄. Finally, a synthetic syngas was used as a feed gas (including hydrocarbons up to C7). In this scenario, pure CH₄ streams were obtained for 16 minutes at 10 bar, 200 °C and a gas space velocity of 1.4 kg_C/kg_{cat}·h [5].

Finally, a comparative study was carried out to evaluate the performance of the SEM process and the conventional methanation process using H₂/CO/CO₂ mixtures as feed gas. Both studies were carried out in the same experimental setup, i.e. a TRL-3 scale fixed bed reactor. The results showed that under SEM conditions it was possible to obtain pure CH₄ operating at moderate pressure and temperature conditions. However, in the case of conventional methanation, the maximum CH₄ purity achieved was 78%vol. (dry basis) using a two cascade reactors scheme. Therefore, this work demonstrates the feasibility and versatility of the SEM process to produce high quality SNG under different feed gas scenarios [6].

Conclusions

This thesis demonstrates the potential of the SEM process for CH₄ production by combining zeolites as adsorbent materials with methanation catalysts to remove the H₂O from the gas phase, thereby achieving high CH₄ purities. The study started with a micro-fixed bed reactor and was scaled up to a TRL-3 fixed bed reactor based on the promising results obtained. Different catalysts (Rh-, Ru- and Ni-based) and zeolites (3A, 4A, 5A and 13X) were evaluated, demonstrating the flexibility of the process under different feed gas scenarios. The research focused mainly on two feed gas compositions: H₂/CO/CO₂ mixtures from sorption enhanced gasification and H₂/CO₂ mixtures where H₂ is produced from water electrolysis and CO₂ is sourced from industrial processes or direct air capture. By operating all stages at a uniform temperature, the SEM process reduces the need for high temperatures to enhance catalytic activity and low temperatures for H₂O condensation, offering significant reductions in both investment and operating costs. In conclusion, this work highlights the versatility and economic advantages of the SEM process for SNG production from diverse feed gas scenarios.

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BIVALIA-CM: Integrated biorefineries for the valorization of agri-food wastes into value-added products in the region of Madrid

BIVALIA-CM: Biorrefinerías integradas para la valorización de residuos de la industria agroalimentaria en productos de elevado valor añadido en la región de Madrid

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Con el nuevo año 2025, ha dado comienzo el proyecto BIVALIA-CM (TEC-2024/BIO-177), financiado en el marco de la *Convocatoria 2024 de ayudas a proyectos de I+D realizados en colaboración entre grupos de investigación pertenecientes a las universidades y organismos de investigación de la Comunidad de Madrid en la modalidad de programas de actividades de I+D en tecnologías*. Liderado por la Universidad Rey Juan Carlos (URJC), el consorcio que desarrollará el proyecto está formado por 8 grupos de investigación (uno de los cuales de carácter emergente), pertenecientes a dos universidades (URJC y Universidad Autónoma de Madrid, UAM) y tres centros de investigación (IMDEA Energía, y el Instituto de Catálisis y Petroleoquímica y el Centro de Investigaciones Energéticas, Medioambientales y Tecnológicas, pertenecientes al CSIC); esto supone la participación de 59 investigadores doctores, a los que se sumarán los investigadores pre y posdoctorales que se contraten con fondos del proyecto concedidos a tal efecto.

Con un presupuesto total de 1.020.000 €, el proyecto persigue la integración de distintas tecnologías para el desarrollo de procesos integrales que permitan la valorización de residuos procedentes de la industria alimentaria. En el contexto de la economía circular, se persigue el avance hacia la sostenibilidad en el sector mediante la valorización de residuos a bioproductos y bioenergía, en contraposición a las actuales vías de gestión, basadas en procesos de valorización energética de baja eficiencia, como la

incineración.

Dentro de la edición anterior de esta convocatoria de financiación, el mismo consorcio de grupos de investigación ya desarrolló una propuesta (proyecto BIOTRES-CM, <https://bio3project.ciberimaginario.es/>) para la valorización de diferentes fracciones residuales generadas en la fase final de la cadena alimentaria (distribución y consumo). El proyecto BIVALIA-CM aborda la valorización de residuos de fases iniciales (producción y distribución primaria), con el objetivo de plantear soluciones a toda la cadena de valor de la industria alimentaria (ver **Figura 1**). El objetivo es, en definitiva, establecer una “hoja de ruta” de valorización para cada tipo de residuo, garantizando la circularidad y la seguridad.

Para el desarrollo del proyecto se han considerado los sectores agroalimentarios más importantes en la Comunidad de Madrid (lácteo, fabricación de cerveza, vitivinícola y ganado porcino), y cuenta con el apoyo de diferentes empresas y asociaciones empresariales, como Lácteas del Jarama, Grupo Mahou, Cervezas La Cibeles, Bodega Bernabeleva, ASEACAM y Kerbest, que identificarán las corrientes residuales, suministrarán muestras de las mismas a los grupos investigadores y proporcionarán el apoyo necesario para la evaluación de las diferentes tecnologías de valorización.

Como puede observarse en la **Figura 2**, el carbón y los materiales de carbono representan un papel destacado, en diferentes formas, dentro de la propuesta tecnológica.

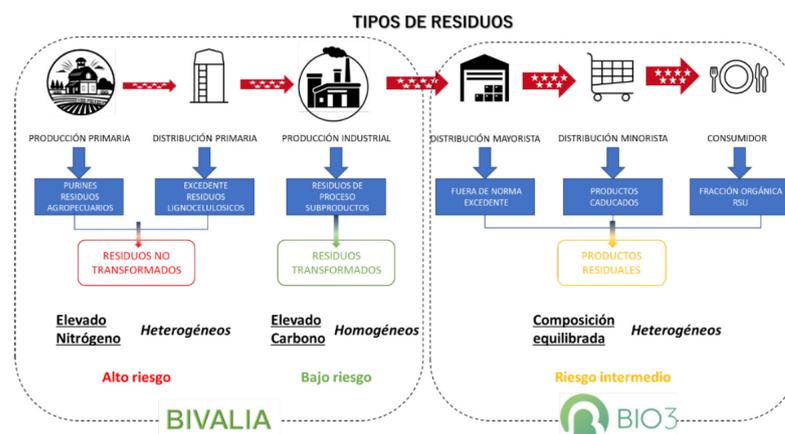


Figura 1. Tipos de residuos biogénicos producidos en los diferentes pasos de la producción de un alimento “de la granja a la mesa”.

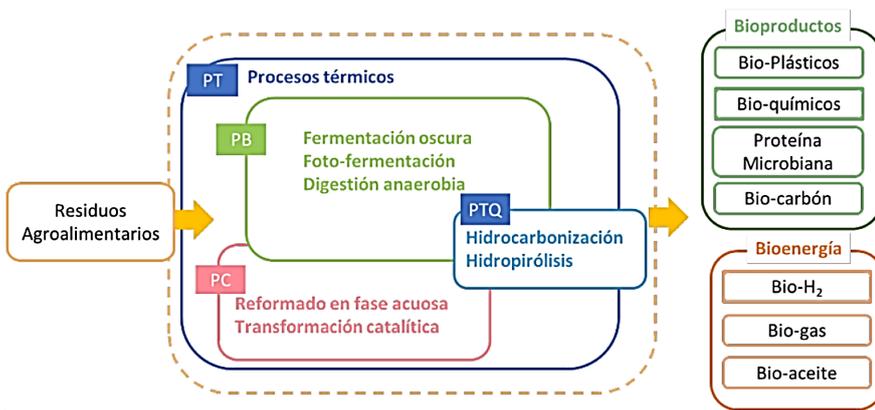


Figura 2. Integración de tecnologías para la producción de bioproductos y bioenergía en el marco del proyecto BIVALIA-CM.

El grupo Waste to Value de la UAM (W2V-UAM) estudiará la carbonización hidrotermal de diferentes residuos y mezclas de ellos para la producción de carbones de calidad adecuada para su posterior uso como biocombustible o para enmienda de suelos. Los resultados obtenidos mediante experimentación a escala de laboratorio serán finalmente validados en planta piloto. Además, diferentes fracciones carbonosas con potencial aplicación como biocombustibles también pueden obtenerse como subproducto en los procesos de hidropirólisis catalítica a baja presión, que serán estudiados por la Unidad de Procesos Termoquímicos del Instituto IMDEA Energía y el Grupo de Ingeniería Química y Ambiental de la URJC (UPT-IMDEA y GIQA-BIO-URJC, respectivamente).

Por otra parte, el grupo Tec4E-UAM (Tecnologías Catalíticas y de Separación para Medio Ambiente y Energía) empleará diferentes materiales carbonosos (negros de humo, carbones activados y carbones mesoestructurados, principalmente) como soporte catalítico para la valorización de diferentes residuos primarios y corrientes residuales secundarias mediante Reformado en Fase Acuosa (APR). Además de la descontaminación de las corrientes, este proceso transforma la materia orgánica presente en la corriente residual en una corriente compuesta, esencialmente, por H_2 y CO_2 . La propuesta investigadora incluye la colaboración con otros grupos participantes para el aprovechamiento de esta corriente en hidropocesos y como alimentación a cultivos mixtos de bacterias fototróficas púrpura

(empleados EN la producción de proteína para consumo, carotenoides, etc.).

Finalmente, el proyecto presta un especial interés a la divulgación de los resultados, a través de la participación del grupo Ciberimaginario de la URJC, de reconocido prestigio en el campo del desarrollo de soluciones de comunicación y formación en entornos digitales.

Tras el éxito alcanzado en BIOTRES-CM en lo relativo a producción científica (85 artículos científicos, unas 200 comunicaciones a congresos, 17 Tesis Doctorales defendidas y 4 patentes solicitadas), el proyecto BIVALIA-CM pretende potenciar al máximo la colaboración entre grupos, con el compromiso de los participantes en la difusión de los resultados exclusivamente a través de contribuciones coordinadas, es decir, en ningún caso firmadas por un solo grupo de investigación participante. Así, durante los cuatro años de duración del proyecto, se espera generar, al menos, 20 publicaciones científicas, solicitar 2 patentes y defender 5 tesis doctorales, todas ellas coordinadas por dos o más grupos de la red; además, está programada la realización de 8 actividades de formación (cursos, workshops, etc.) y la organización de 1 congreso internacional.

Toda la información del proyecto puede consultarse en la web <https://bivalia.ciberimaginario.es/>, a través de la cual también se puede solicitar la suscripción a la lista de distribución para conocer de primera mano todas las novedades, eventos y resultados científicos y tecnológicos de la red.



Biorrefinerías integradas para la valorización de residuos de la industria agroalimentaria en productos de elevado valor añadido en la región de Madrid



II Curso del GEC: El carbono y sus materiales, caracterización y aplicaciones

Celebrado en Baeza del 18 al 20 de noviembre de 2024

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El pasado 18 de noviembre se inauguró la segunda edición del Curso del GEC. Como probablemente recuerdan los lectores, este curso se dirige principalmente a los estudiantes de doctorado de los grupos de investigación que participan de nuestra asociación, brindándoles una formación básica en nuestro campo de investigación que es impartida por especialistas en las diferentes líneas que abarcan los grupos del GEC. Está abierto también a cualquier otro estudiante interesado en profundizar en el carbono, ya sea en sus materiales y usos, o en la gestión de los gases de efecto invernadero donde este elemento se encuentra presente. Nuevamente, el curso se impartió en Baeza, dentro de las instalaciones de la Sede Antonio Machado de la Universidad Internacional de Andalucía (UNIA). El organizarlo a través de la UNIA permite que se certifique su realización, haciéndolo más atractivo para participantes provenientes de otros ámbitos. En esta ocasión, el curso estuvo codirigido por Agustín F. Pérez Cadenas y por Ramiro Rafael Ruiz Rosas.

Esta edición ha disfrutado de un notable incremento de participación, contándose con 45 estudiantes matriculados, que incluyen algunos participantes de la Universidad de Jaén que confiamos que pasen pronto a formar parte de nuestro grupo. Agradecemos el esfuerzo de todos los grupos que participan de nuestra asociación en apoyar esta actividad enviando a sus miembros más jóvenes. El curso contó con una duración de 20 horas, repartidas a lo largo los días 18, 19 y 20 de noviembre. Se estructuró sobre un bloque de conferencias de 2 horas de duración en las que se mostró a los estudiantes los últimos avances sobre la producción de los diferentes materiales de carbono y en su caracterización de forma rigurosa, evitando caer en la determinación de forma acrítica de parámetros fundamentales. Se abordaron también las distintas aplicaciones tecnológicas de estos materiales, principalmente en los sectores medioambiental y energético. En consonancia con nuestro compromiso a nivel de sociedad en la lucha contra el cambio climático, en esta edición se prestó especial atención al papel que juega el carbono en procesos destinados a reducir las emisiones de gases de efecto invernadero. Destacó el interés de los ponentes en recordar a los estudiantes la importancia de conocer los orígenes y estudiar los trabajos fundacionales de nuestros campos de investigación, evitando que la rueda se redescubra en cada nueva generación, y apoyándonos en esa base de conocimiento para expandir realmente las fronteras del mismo.

Estas conferencias se impartieron por diez miembros del GEC, siendo figuras de referencia que sirven

de ejemplo de cómo desarrollar exitosamente una carrera investigadora en nuestros ámbitos de conocimiento. A continuación, se recoge el programa diario del curso, junto con los ponentes a cargo de cada conferencia.

Lunes, 18 de noviembre: *Evolución de los materiales de carbono como respuesta a los desafíos de nuestra sociedad.* Rosa Menéndez López. *Síntesis sostenible de materiales de carbono porosos.* Noel Díez Nogués. *Geles de carbono y sus aplicaciones.* Agustín F. Pérez Cadenas. *Caracterización de la Química Superficial de Materiales de Carbono.* Francisco Heras Muñoz.

Martes, 19 de noviembre: *Caracterización de la texturaporosa de materiales de carbono y aplicaciones en almacenamiento de energía.* Diego Cazorla Amorós. *Caracterización superficial de materiales de carbono mediante métodos electroquímicos y aplicaciones en sensores.* Emilia Morallón Núñez. *Opciones y oportunidades en tecnologías de captura de CO₂.* Alberto Abad Secades. *Materiales de carbono para pilas de combustible, electrolizadores y baterías.* David Sebastián del Río.

Miércoles, 20 de noviembre: *Aplicaciones catalíticas de los materiales de carbono.* Juana M^a Rosas Martínez. *Aplicaciones medioambientales de los materiales de carbono.* Tomás Cordero Alcántara.

Agradecer encarecidamente a todos ellos su contribución al éxito del curso. Esperamos que hayan disfrutado de su participación, como lo hicimos los directores de su compañía, ya fuese en las aulas, en nuestros paseos matinales o nocturnos por el hermoso casco histórico de Baeza, o en las sobremesas en la sede "chica" de la Xavi Taberna, trasladada el último día a El Pasaje. El curso finalizó con una mesa redonda, moderada por los directores, donde los estudiantes expresaron su satisfacción con el contenido, y expusieron posibles mejoras para futuras ediciones. A buen seguro que al alumnado en la próxima edición no le faltará papel para tomar notas.



Sede de la UNIA – Baeza



Salón de ponencias, UNIA – Baeza



Ayuntamiento de Baeza

El realizar consecutivamente al curso del GEC la jornada de jóvenes investigadores en la sede de Baeza brinda una oportunidad única para que nuestros miembros más recientes interactúen, compartan vivencias similares, y establezcan nuevas amistades que permitan aumentar las colaboraciones entre nuestros grupos, siendo esto otro de los objetivos de estas actividades. Más allá de asistir a las clases en las aulas, el alojarse juntos en la Residencia durante cuatro noches, compartir mesa en alguno de los restaurantes de los soportales de la plaza de la constitución, y de contar con atractivas distracciones nocturnas en el Café Teatro Central seguro que ha facilitado que se consiga este objetivo, parte importante de la formación y el desarrollo de su carrera investigadora. Dada la satisfacción mostrada por los estudiantes, el crecimiento del número de participantes, y las ventajas que presenta el contar

con el apoyo administrativo de la UNIA y una sede donde realizar eventos en un entorno monumental sin un elevado coste, confiamos en que estas actividades se afiancen y constituyan un nuevo pilar de nuestro Grupo que complemente a las Reuniones. Nuestros jóvenes, con toda seguridad, serán en un futuro piezas clave de nuestro Grupo, todo esfuerzo puesto en su formación y capacitación redundará en beneficio del mismo.

Nos despedimos animando a todos los jóvenes investigadores que todavía no lo hayan hecho a participar en las próximas ediciones de estos eventos. También invitando a nuestros socios no tan jóvenes del GEC a colaborar activamente en futuras ediciones como docentes, así como informando de su existencia y motivando a sus doctorandos y jóvenes doctores a participar en ellos. ¡Nos vemos en las próximas ediciones!



Asistentes al Curso

II Jornadas de Jóvenes Investigadores del Grupo Español del Carbón (GEC) - Baeza (Jaén) 20-22 de noviembre 2024

Nausika Querejeta Montes, Zoraida González Arias (INCAR-CSIC)

Las II Jornadas de Jóvenes Investigadores del Grupo Español del Carbón (GEC) se celebraron, del 20 al 22 de noviembre de 2024, en la Sede Antonio Machado de la Universidad Internacional de Andalucía (UNIA) en Baeza (Jaén). Este evento, que tuvo su primera edición en fechas similares dos años antes, se ha consolidado como un importante punto de encuentro para los jóvenes investigadores pertenecientes al GEC.

La elección de Baeza como sede del evento no fue casual, ya que esta ciudad, reconocida como Patrimonio de la Humanidad, ofrece un entorno único que combina la riqueza histórica del Renacimiento español con instalaciones modernas adecuadas para el desarrollo de actividades académicas. La Sede Antonio Machado de la UNIA, ubicada en el corazón del conjunto histórico de Baeza, proporcionó el escenario ideal para fomentar el intercambio de ideas y la colaboración entre los participantes.

El objetivo principal de las jornadas fue incentivar el intercambio de experiencias y promover una temprana colaboración entre los jóvenes investigadores, creando un espacio donde pudieran compartir sus trabajos y establecer colaboraciones, fomentando así la capacidad de innovación, el pensamiento creativo y la investigación de excelencia.

Estas segundas jornadas fueron organizadas por las Dras. Zoraida González Arias y Nausika Querejeta Montes del Instituto de Ciencia y Tecnología del Carbono (INCAR-CSIC). El comité organizador contó también con la importante participación de miembros de otras instituciones pertenecientes al GEC (María del Carmen Recio Ruiz -UMA, Jaime López de los Álamos -ICB-CSIC, Samuel Calabuig Mompó -UA, Gabriela Live Lozada -UAM y Esther Bailón García -UGR), quienes desempeñaron un papel fundamental tanto en la preparación y desarrollo del evento como en la moderación de las sesiones.

En esta nueva edición, cuyo tema principal giraba en torno a los *"Materiales de carbono en la vanguardia de la tecnología y la sostenibilidad"*, han participado 50 investigadores (provenientes de 8 instituciones investigadoras nacionales) de los cuales 24 presentaron comunicaciones orales y el resto trabajos en formato póster todos ellos agrupados en diferentes topics. Además, se ha contado con 4 charlas especializadas. Dos de ellas tuvieron como objetivo la orientación de futuras carreras investigadoras: *"Una carrera investigadora. ¿Qué opciones de financiación tengo?"*, impartida por María Ros (Vicerrectorado de Investigación, Universidad de Granada) y *"Carrera investigadora: de la academia al sector privado"*, impartida por Juan Pablo Marco (Gas to Materials Technologies, S.L). Por otro lado, se contó con la charla impartida por Iñigo Zabalía de IESMAT - Instrumentación Específica de los Materiales S.A. (*"Tecnología DVS*

-Dynamic Vapor Sorption- en estudios de captura de CO₂. Estudios de co-adsorción entre la humedad ambiente y el CO₂ atmosférico), empresa que también patrocinó la pausa café del segundo día de las jornadas. La cuarta de las charlas (*"Nanomaterials and C-Journal of Carbon Research: A Step-by-Step Guide Through the Editorial Process"*) corrió a cargo de Paulina Maziarz, directora editorial de la revista *Nanomaterials* de MDPI, editorial que patrocinó los premios a mejor comunicación oral y mejor póster.



Figura 1. Juan Pablo Marco de Gas to Materials Technologies, S.L durante la impartición de su charla. Baeza, 21/11/2024.



Figura 2. Iñigo Zabalía de IESMAT - Instrumentación Específica de los Materiales S.A. durante la impartición de su charla. Baeza, 21/11/2024.



Figura 3. Acto de apertura de las II Jornadas de Jóvenes Investigadores del GEC. Baeza, 20/11/2024.

Tras la entrega de acreditaciones, la tarde del 20 de noviembre tuvo lugar el arranque oficial de la programación científica de las jornadas con el correspondiente acto inaugural a cargo del Prof. Agustín Pérez Cadenas de la Universidad de Granada y presidente del GEC (cuya inestimable colaboración resultó fundamental para la buena marcha del evento).



Figura 4. Acto de entrega de los premios a mejor oral y mejor póster. Baeza, 22/11/2024.

Figura 5. Asistentes a las II Jornadas de Jóvenes Investigadores del GEC. Baeza, 21/11/2024.



Figura 6. Imágenes tour nocturno por Baeza y cena, 21/11/2024.

Las jornadas se desarrollaron con gran éxito, ofreciendo varias sesiones de excelente calidad que abarcaron los últimos avances en la síntesis, procesado y caracterización de materiales de carbono, así como sus crecientes aplicaciones que pretenden abordar importantes retos de la sociedad. No podemos olvidarnos de otro tipo de aplicaciones, como la producción de biocombustibles, la adsorción/ separación/almacenamiento de gases, el manejo de residuos de biomasa o la producción de productos de alto valor añadido. Las sesiones orales destacaron por el elevado nivel científico de los jóvenes investigadores, quienes demostraron un notable interés y participación. Finalmente, durante la sesión de pósteres se exhibieron trabajos de gran calidad por parte de los investigadores más noveles, en un ambiente distendido que fomentó el intercambio de ideas.

Este evento ha proporcionado una plataforma integral para la presentación de investigaciones de vanguardia, networking profesional y orientación en el campo de los materiales de carbono, respaldado por el apoyo de importantes patrocinadores. Los avances presentados demuestran que, dada su versatilidad y relevancia, los materiales de carbono continúan siendo objeto de investigación de vanguardia, evidenciando su capacidad para abordar desafíos tecnológicos y sociales y reafirmando así

su papel fundamental en el avance científico y el desarrollo sostenible.

Las Jornadas finalizaron el viernes 22 de noviembre con la entrega de premios a mejor comunicación oral para María Nerea Rivas Márquez de la Univ. de Málaga y mejor comunicación en formato póster para Luis La Calle Canelada del Instituto de Carboquímica de Zaragoza.

Como no podía ser de otra manera, el evento también contó con una programación de actos sociales aprovechando el encantador entorno en el que se desarrollaba. Así, el 21 de noviembre los participantes disfrutamos de un tour nocturno guiado por la hermosa ciudad de Baeza, descubriendo así su rico patrimonio histórico y cultural. Posteriormente, se celebró una cena que fomentó la interacción entre los jóvenes investigadores, permitiéndoles establecer conexiones y fortalecer los vínculos entre las distintas instituciones que conforman el GEC. Esta combinación de actividades académicas y sociales enriqueció la experiencia general del evento.

Finalmente, queremos dedicar unas líneas de agradecimiento a los integrantes del Comité Organizador, a la UNIA por la acogida, y a todos los participantes por su aportación. Nos vemos en el mismo enclave en las siguientes jornadas que tendrán lugar en 2026.

Socios protectores del Grupo Español del carbón



Química del Nalón

