



Elaboration of Activated Carbons from Lignocellulosic Residues (Saba and Palm Tree Husks) for the Adsorption of Ibuprofen

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Abstract

The management of agricultural waste and untreated wastewater, particularly those containing pharmaceutical pollutants, is a significant challenge, especially in West Africa. A possible solution lies in the valorization of agricultural biomass for the production of activated carbon, which can be used to treat these pollutants. In this study, two lignocellulosic biomasses from the Ziguinchor region in Senegal were used: saba senegalensis and palm tree husks. The chemical activation of the biomasses was carried out using phosphoric acid (85%), varying several parameters: the raw material/acid ratio, carbonization temperature (500 °C to 800 °C), and the process duration (40 to 60 min). The results showed that the best activated carbons were obtained with an impregnation ratio of 1:2, a carbonization temperature of 800 °C, and a 60-minute carbonization time. Textural and chemical analyses of the activated carbons were conducted, along with adsorption tests on pollutants such as ibuprofen in aqueous solutions. The performance of the activated carbons developed was compared to that of a commercial activated carbon (L27). The activated carbons produced demonstrated good adsorption capacities, making them a promising solution for treating water contaminated with pharmaceutical compounds.

Keywords Activated carbon · Biomass · Adsorption · Isotherm · Kinetics

1 Introduction

For several years now, a great deal of research has been carried out on the study of lignocellulosic materials because their use falls within the field of biomass recovery in the materials or energy sector [1–7]. These materials are of growing interest in the activated carbon industry but also in the field of composite materials, textiles and fibers [8–11]. Many studies deal with the transformation of lignocellulosic materials from agricultural or plant waste (carbon-rich sources) into carbonaceous adsorbent materials, for organic molecule or metal adsorption (for example hexavalent chromium) [12–14]. The wide range of raw materials used for their elaboration and the diversity of the elaboration

processes mean that adsorbent materials with very different textural and chemical properties are obtained [12]. The greatest difficulty is to be able to identify the raw materials with a high potential to become activated carbons and likely to develop interesting textural properties. Some unexploited biomass residues such as millet stalks, rice husks, roasted tree hulls, cashew nut hulls, ditakh hulls, saba senegalensis hulls, etc., are often eliminated by simple combustion (very often without valorizing the energy) in order to solve problems of congestion and pollution (leaching, methanization, etc.). Activated carbons can be produced from any organic plant materials rich in carbon. Thus, agricultural waste, due to its abundance and availability, is a good source of raw material for the production of activated carbons. However, their activability potential depends on their composition in hemicelluloses, cellulose and lignin. One of the major challenges in the commercial production of activated carbons is to identify new inexpensive precursors and to this end much research has been reported on the development of activated carbons from agricultural waste, including corn cobs [15], olive pits and walnut shells [16], sunflower seed shells, sugar cane bagasse [17], almond shells, peach pits, grape seeds, apricot kernels, cherry pits [18], peanut shells, rice

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shells [19] and coconut shells [20, 21]. These feedstocks were considered to be suitable precursors due to their high carbon and low ash content [22].

For example, in Senegal, the green region of Ziguinchor is located in a Sudano-Guinean climatic zone characterized by good rainfall, making the land fertile for growing cereals, but also for growing fruit trees and wild shrubs. This context generates the discharge of large quantities of agricultural residues which can have a negative impact on the environment and whose valorization to produce energy or to obtain materials developed for application in water treatment is a promising way to make the production chains more profitable [7]. Ibuprofen can have harmful effects on the environment and when discharged into wastewater. It can contaminate aquatic and terrestrial ecosystems. Even at low concentrations, it can have toxic effects on various aquatic organisms such as fish, snails, and bacteria. Its persistence in the environment is a major problem, partly due to inadequate drug disposal. Ibuprofen is primarily eliminated via urine and reaches wastewater treatment plants through the sewage collection network. It is important to be able to remove it efficiently in particular on activated carbons.

The activated carbon adsorption process is still widely used in wastewater treatment plants, water treatment plants and for the treatment of industrial effluents. It remains a very effective process for removing many molecules, particularly pharmaceutical compounds [23, 24]. Nevertheless, it is very difficult to optimize these decontamination processes by adsorption on activated carbon without a good knowledge of the material and the interactions that can exist between the adsorbent material and the molecule to be adsorbed [25]. For this purpose, two raw materials were selected with a high tonnage that have never been tested in the context of the development of adsorbent materials: “saba senegalensis” and “ronier shell”, both from the Ziguinchor Region. The predictive model established by Boundzanga [1] was used to determine the activability potential of lignocellulosic materials. These biomasses were activated with phosphoric acid using the chemical activation procedure developed by Pereira [26] to produce activated carbons. Several parameters were studied: the impregnation ratio between the raw material and the activating agent (ratio 1:1 and 1:2), the optimal carbonization temperature (500 °C, 600 °C, 700 °C and 800 °C) as well as the residence time (40 minutes and 60 minutes) at the final temperature in order to optimize the porous properties of the ACs for the adsorption of ibuprofen in water. The developed ACs were characterized by nitrogen isotherm at 77 K, elemental analysis (CHONS) and FTIR analysis in order to study the influence of the chemical activation process parameters on the composition, surface

chemistry and porous properties of the adsorbent materials. The most promising ACs, namely SS-1/2–800-60, SS-1/2–800-40, RS-1/2–800-60, RS-1/2–800-40 were tested for the adsorption of ibuprofen (a pollutant often found in wastewater and worldwide and particularly in wastewater from a hospital in Ziguinchor (Senegal)) and compared to a commercial AC L27 (supplied by Jacobi Carbons, Vierzon, France).

2 Materials and Methods

2.1 Material

Two agricultural residues (coconut shells and Saba Senegalensis) were chosen as precursors for activated carbon because coconut shells and Saba Senegalensis are widely cultivated in Senegal and the consumption of their fruits generates a lot of waste in the form of shells, which cause congestion and air pollution after incineration.

Native to West Africa, Saba senegalensis belongs to the Apocynaceae family, and is a wild plant that is mainly used for food. In Senegal, the fruit is consumed as is or processed into puree, nectar, syrup, preserves, etc. It is a plant with enormous therapeutic virtues, from the fruit to the roots, including the leaves, bark and latex. Despite its high nutritional potential, its numerous therapeutic virtues and its economic potential, Saba senegalensis remains under-exploited [27].

Saba senegalensis hulls were collected in the Ziguinchor Region (southern Senegal), then washed and dried naturally in an environment deprived of sunlight for four weeks (Fig. 1).

Borasse (Borassus) is a genus of palms that includes nine species native to the tropical regions of Sahelian Africa (Ethiopia, Niger, Nigeria, northern Togo, Senegal, etc.), Asia and New Guinea. They are large palms with a smooth, grey stipe that can reach 30 m in height. The fruits are grouped in tight clusters and are ovoid or globular, smooth and yellow, brown or orange. Ronier shells were collected in the Ziguinchor Region (southern Senegal) and then washed and dried naturally in an environment deprived of sunlight for four weeks (Fig. 2).

The commercial GAC (L27), widely used in water treatment, was supplied by Jacobi Carbons (Vierzon, France). It was washed several times with water to remove surface impurities and any residual acidity due to the activation treatment, followed by drying at 80 °C for 24 h.

Ibuprofen (Sigma Aldrich) (Fig. 3) stock solution of 200 mg.L⁻¹ was prepared by dissolving the compound in ultra-purified water.

Fig. 1 *Saba senegalensis* hulls **a)** before drying and **b)** after drying



Fig. 2 *Ronier* shells **a)** before drying and **b)** after drying

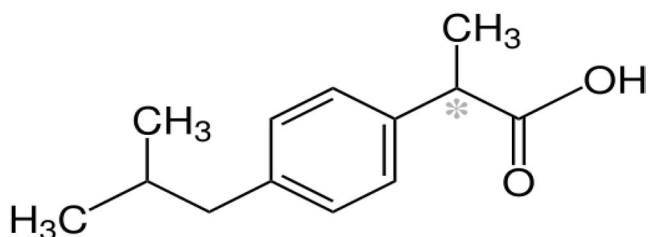


Fig. 3 Chemical structure of ibuprofen

2.2 Adsorption Studies

For the kinetic studies, a weight of 20 mg of AC was added to 50 mL of ibuprofen solution. The initial concentration value of ibuprofen (C_0) was 200 ppm. The samples were kept in a magnetically stirred reactor at room temperature (20 ± 1 °C) throughout the experiment. Stirring conditions were chosen to favor a good material/solution contact and to avoid material attrition. At suitable time intervals (5–15–30–45–60–90–120–180–240–300–360–420–450 min), 50 mL of samples were extracted from the reactor using a 10 mL syringe and were filtered by means of Whatman 0.45 μm filters. The concentration of ibuprofen was determined at $\lambda=222.4$ nm using an Agilent Technologies Cary 60 UV/VIS spectrophotometer.

For the equilibrium experiments (adsorption isotherms), a weight of 20 mg of AC was added to 50 mL of ibuprofen solution in glass reactors. The initial concentration of

ibuprofen ranged from 20 to 200 mg.L^{-1} . For the isotherms, an equilibrium time of 450 min, based on kinetic results, was chosen for each experiment. The amount of ibuprofen adsorbed at t time q_t (mg.g^{-1}) and at equilibrium q_e (mg.g^{-1}) was calculated from the following equations:

$$q_t = \frac{(C_0 - C_t)}{W} \cdot V \text{ and } q_e = \frac{(C_0 - C_e)}{W} \cdot V \quad (1)$$

where C_0 , C_t and C_e (mg.L^{-1}) represent ibuprofen concentration in the initial solution, at time t , and at equilibrium time respectively, V is the solution volume (L) and W the weight of the adsorbent (g).

The ibuprofen AC removal efficiency (Y in %) was calculated by using the equation below:

$$Y = \frac{C_0 - C_t}{C_0} \cdot 100 \quad (2)$$

2.3 Preparation of Activated Carbons

The activated carbons used in this work were developed from two biomasses: *Saba Senegalensis* shells (SS) and *Ronier* shells (RS). The dry hulls were ground using a RETSCH PM100 planetary ball mill to obtain biomass strands on a millimeter scale. A mass of 1 g of lignocellulosic raw materials was mixed with 1 g of phosphoric

acid H_3PO_4 (85%, Sigma Aldrich) to obtain an impregnation ratio of 1:1. After adding a volume of distilled water to the mixture, the whole was kept under stirring for 30 min at a temperature of 80 °C. The resulting product was then dried under vacuum at a temperature of 110 °C for 15 h before undergoing heat treatment. The carbonization of the product obtained after drying was carried out by using Tube Furnace 21,100 from Barnstead Thermolyne. The product obtained after carbonization was then washed with a solution of hydrochloric acid HCl (37%) and stirred for 20 min. It was then rinsed with distilled water until a neutral pH is obtained. The neutral product obtained was dried in an oven at 105 °C for 15 h before being recovered and the final mass obtained was weighed.

The heat treatment was carried out with different final carbonization temperatures of 500 °C, 600 °C, 700 °C and 800 °C and two different plateau values (40 min and 60 min). Thus, four SS activated carbons with an impregnation ratio of 1:1 were prepared from the protocol described above. The same protocol as above was repeated for the preparation of four other SS activated carbons with an impregnation ratio of 1:2 (1 g of crushed SS shells and 2 g of H_3PO_4 phosphoric acid) with the same step time and the same temperature ranges. With the same impregnation ratios (1:1 and 1:2) and temperature ranges (500 °C to 800 °C), eight other SS activated carbons were prepared with a 60-minute step following the same protocol. The activated carbons prepared from Ronier hulls were prepared following the same experimental protocol as that used for the preparation of *Saba senegalensis* ACs. The activated carbons were identified by a code corresponding to the letters of the raw material (SS for *Saba Senegalensis* shells and RS for Ronier shells), the ratio used in the activation procedure (1:1 or 1:2), the temperature of the heat treatment (in °C), and the time of the plateau at the final temperature (in minutes). For example, RS-1:1-800-40 is a sample resulting from the activation of the Ronier shells with phosphoric acid at a ratio 1:2 at 800 °C during 40 min.

2.4 Characterization of Adsorbents

The porosity of the adsorbent material was characterized through a conventional nitrogen adsorption isotherm at −196 °C (77 K) using a Micromeritics ASAP 2020. The sample was previously degassed at 250 °C for 48 h under a residual vacuum of less than 10^{-4} Pa. The adsorption isotherms were carried out with relative pressures ranging from $5 \cdot 10^{-6}$ to 0.99.

Both the specific microporous volume W_0 ($\text{cm}^3 \cdot \text{g}^{-1}$) and the mean pore size L_0 (nm) were estimated from the linear part of the Dubinin–Radushkevich (D–R) plots [28, 29]. Sing α_s plots were also used to determine the specific external surface S_{ext} ($\text{m}^2 \cdot \text{g}^{-1}$) [29], assuming that for slit-shaped

micropores the specific microporous surface S_{micro} ($\text{m}^2 \cdot \text{g}^{-1}$) could be estimated using the specific microporous volume and the mean pore size [28]. The micropore size distribution was obtained with the DFT method (Micromeritics Software).

Thermogravimetric experiments were done by a thermobalance (STA 449 F5 Jupiter, NETZSCH).

A mass of approximately 0.030 g of sample was placed in the thermobalance under a $160 \text{ mL} \cdot \text{min}^{-1}$ argon flow. Systematically, a blank experiment was carried out, under the same conditions, in order to determine the correction required for the base-line. The procedure consisted of a temperature increase at a $20 \text{ }^\circ\text{C} \cdot \text{min}^{-1}$ heating ramp from ambient temperature to 800 °C (to study a larger temperature domain), followed by a one-hour interval at this final temperature and a cooling to room temperature at a cooling rate of $10 \text{ }^\circ\text{C} \cdot \text{min}^{-1}$.

TEM analyses were conducted using a transmission electron microscope, a Philips CM20 Lab6, 200 kV. A dispersion of the powder, ground in ethanol, with 3 min of ultrasonic treatment, was deposited on standard holey carbon-covered copper TEM grids.

The lignocellulosic composition (cellulose, hemicelluloses, lignin, ash and extractable compounds) of the precursors was analyzed following a procedure described in detail elsewhere [1, 30]. In brief, the precursors were soxhlet extracted with ethanol for 1 h followed by water for 1 h. For these extracted samples, Klason and acid soluble lignins were determined by using a 72% sulfuric acid solution. Holocellulose was quantified with sodium chlorite treatment and α cellulose content was determined by extraction with 17.5% aqueous sodium hydroxide of the holocellulose powder. Cellulose content was evaluated by the sum of α cellulose content and ashes and hemicelluloses amount was obtained by the difference between holocellulose and cellulose. The ashes were obtained by weight after the incineration of samples at 600 °C for 4 h. All the analyses were done in duplicate.

The temperature rate of $15 \text{ }^\circ\text{C} \cdot \text{min}^{-1}$ was first set to reach 105 °C. At 105 °C, a 3 min level was maintained to determine the humidity of the weighed samples (SS and RS). After the humidity determination step, a second speed of $20 \text{ }^\circ\text{C} \cdot \text{min}^{-1}$ was then programmed to reach 950 °C for the samples (SS and RS). A cooling process then began with the same speed until reaching 450 °C. This part of the heating program determines the volatiles of each sample. The sample obtained after the volatile matter determination step was reintroduced into the oven for heating up to 800 °C carried out in air with a rising speed of $15 \text{ }^\circ\text{C} \cdot \text{min}^{-1}$ and followed by isothermal maintenance at 800 °C for 2 min to determine the ash content. The method for assessing fixed carbon content was to first determine the moisture content,

then the volatile matter content and finally the ash content. By subtracting the percentage of humidity, volatile matter and ash, the fixed carbon content was estimated.

3 Results and Discussion

3.1 Biochemical Analyses of the Raw Materials

3.1.1 Biomass Quantification: H-C-L

The results of the biochemical quantification in terms of hemicelluloses, cellulose and lignin are shown in Fig. 4. One can see that the cellulose fraction of RS is higher than the composition obtained with SS, while the lignin fraction in SS is higher than that obtained for RS. The hemicellulose composition of the two materials is almost the same. These results also clearly established that cellulose and lignin are the major compounds for the two lignocellulosic precursors studied. The predictive calculation proposed by Boundzanga et al. [1] makes it possible, from the biochemical composition of a lignocellulosic precursor, to estimate the contribution of hemicellulose, cellulose and lignin to the yield and porosity of the corresponding activated carbon. The compound responsible for the formation of micropores

in the final activated carbon is lignin, whereas hemicelluloses and cellulose are responsible for the formation of mesopores [1]. These results predict a good adsorption power of the activated carbons of the two lignocellulosic precursors.

3.1.2 CHONS of Raw Materials

The CHONS analyses carried out on the raw materials of SS and RS made it possible to obtain the percentages of Carbon, Hydrogen, Oxygen and Nitrogen of the samples which are given in Table 1.

CHONS analyses show a main percentage of carbon of about 48.8% and 43.8% for SS and RS respectively. Almost the same composition of hydrogen and oxygen was found for the two materials. Overall, the CHONS chemical compositions of the two raw materials were close and were representative of lignocellulosic materials.

3.1.3 Thermogravimetric Analyses of Raw Materials

Thermogravimetric analyses were carried out on the two raw lignocellulosic precursors SS and RS and results are depicted in Fig. 5. Moisture and highly volatile compounds were eliminated up to a temperature close to 150 °C. Strong weight losses were observed when the temperature

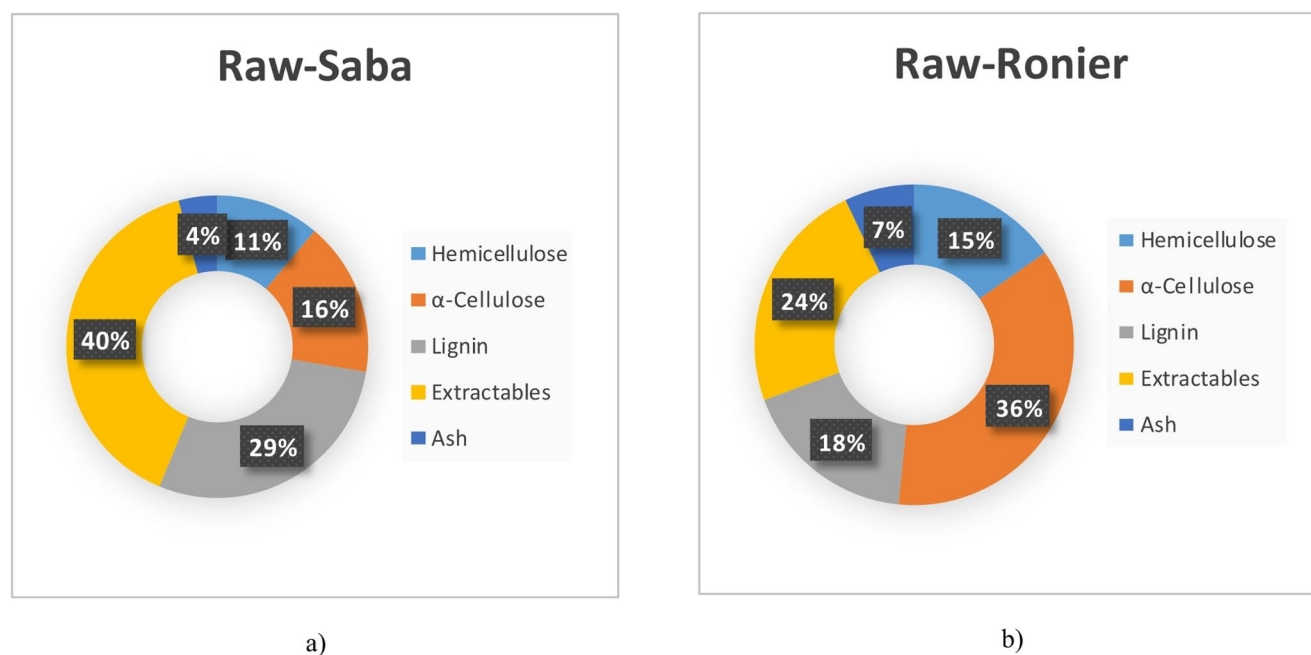


Fig. 4 Biochemical composition (in wt.%) in hemicelluloses (H.), cellulose (C.), and lignin (L.) of the raw lignocellulosic precursors : **a)** Saba senegalensis and **b)** ronier shell

Table 1 CHONS analysis results

Sample	%C	%H	%O	%N	%S	Water+Residues
RAW-SABA	48.8	5.7	34.4	0.3	0.0	10.8
RAW-RONIER	43.8	5.9	35.8	2.3	0.0	12.2

Fig. 5 a) Raw-saba and b) raw-rônier TGA-DTG experimental curves

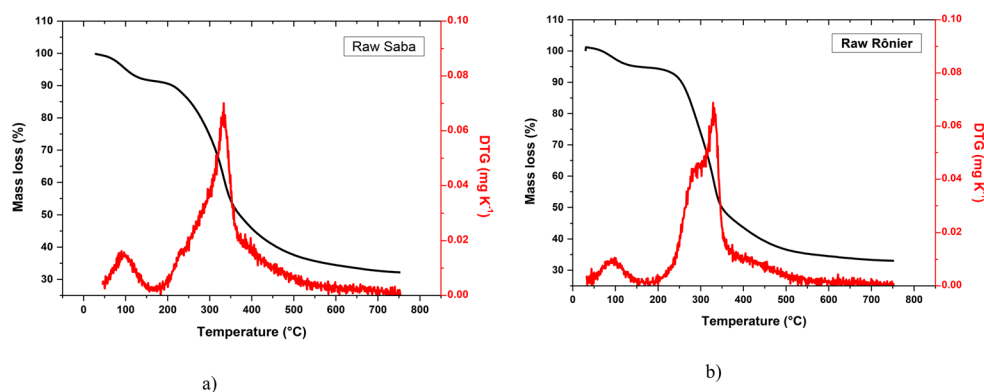


Table 2 Proximate analysis (wt%)

Proximate analysis (wt%)	SS	RS
Moisture	9.6	5.8
Volatile matter	50.1	55.0
Fixed carbon	36.2	32.1
Ash content	4.1	7.1

increased from 200 °C to 400 °C as volatile matter and tar are eliminated due to carbonization or active pyrolysis [31]. Previous studies have reported that the second stage can be divided into hemicelluloses decomposition between 180 °C and 270 °C, and cellulose decomposition between 270 °C and 400 °C [21, 31, 32]. The third stage (400–550 °C) corresponds to the decomposition of structurally stable components of the biomass [31]. Above 550 °C, less weight loss was observed as the decomposition of lignin takes place above this temperature [31, 32].

The volatile matter contents of the raw biomasses SS and RS were respectively 50.1% and 55.0% but those of ash for SS and RS had a respective value of 4.1% and 7.1% (Table 2). These values show that these two biomasses have high volatile matter contents but low ash contents, making them good raw materials for the preparation of activated carbon [33].

3.2 The Activated Carbons Produced

3.2.1 Loss of Mass and Yield for the AC SS-1:1–40 and 60 and SS-1:2–40 and 60

A study of mass loss and yield was carried out on the processed activated carbons. Figure 6 shows a variation in mass loss and yield as a function of the final activation temperature. The mass loss of the activated carbons prepared from SS hulls with ratios of 1:2 and 1:1 and a final plateau of 40 min varied respectively between 53.8% and 86.8% and between 38.0% and 71.8% with yield varying respectively between 46.2% and 13.2% and between 62.0% and 28.2%. For a final plateau of 60 min, the results of the mass loss of the AC SS hulls with ratios of 1:2 and 1:1 varied

respectively between 46.0% and 82.5% and between 38.3% and 77.4%. Their yield respective returns for the same ratios varied between 54.0% and 17.5% and between 61.7% and 22.6%, respectively. The higher the quantity of activating agent in the ratio, the more the mass loss increased and the yield decreased. The time from the plateau to the final activation temperature has little impact on mass loss and efficiency in the case of SS.

3.2.2 Mass Loss and Yield for the AC RS-1:1–60 and RS-1:2–60

Figure 7 shows that the mass loss of RS activated carbons produced with a process duration of 60 min and ratios of 1:1 and 1:2 respectively varied between 38.0% and 71.2% and between 41.4% and 69.8%. Their yield varied respectively between 62.0% and 28.8% and between 58.6% and 30.2%.

The same trend was observed for Ronier shells. Overall, for the same activation operating conditions, the mass losses were lower and the yields were similar to those of SS. The greater the concentration of the phosphoric acid activating agent, the greater the mass losses.

3.2.3 Porous Properties of ACs Produced

The porosity results of the ACs selected after a heat treatment at different temperatures, two ratios (1:1 and 1:2), a carbonization time of 60 and 40 min compared to the commercial AC L27 are reported in Table 3.

Table 3 shows that activated carbons obtained from RS with phosphoric acid (1:1 ratio with a 60-minute step at different temperatures) have different porous properties. However, between 500 °C and 700 °C, the microporous volume decreases from 0.35 cm³.g⁻¹ to 0.21 cm³.g⁻¹ (40% decrease), the microporous surface decreases from 526 m².g⁻¹ to 331 m².g⁻¹ (37% decrease), the external surface area increases from 129 m².g⁻¹ to 170 m².g⁻¹ (24% increase) and the total surface area decreases from 655 m².g⁻¹ to 514 m².g⁻¹ (22% decrease). ACs produced at 800 °C have a better developed porosity with a microporous volume of 0.31

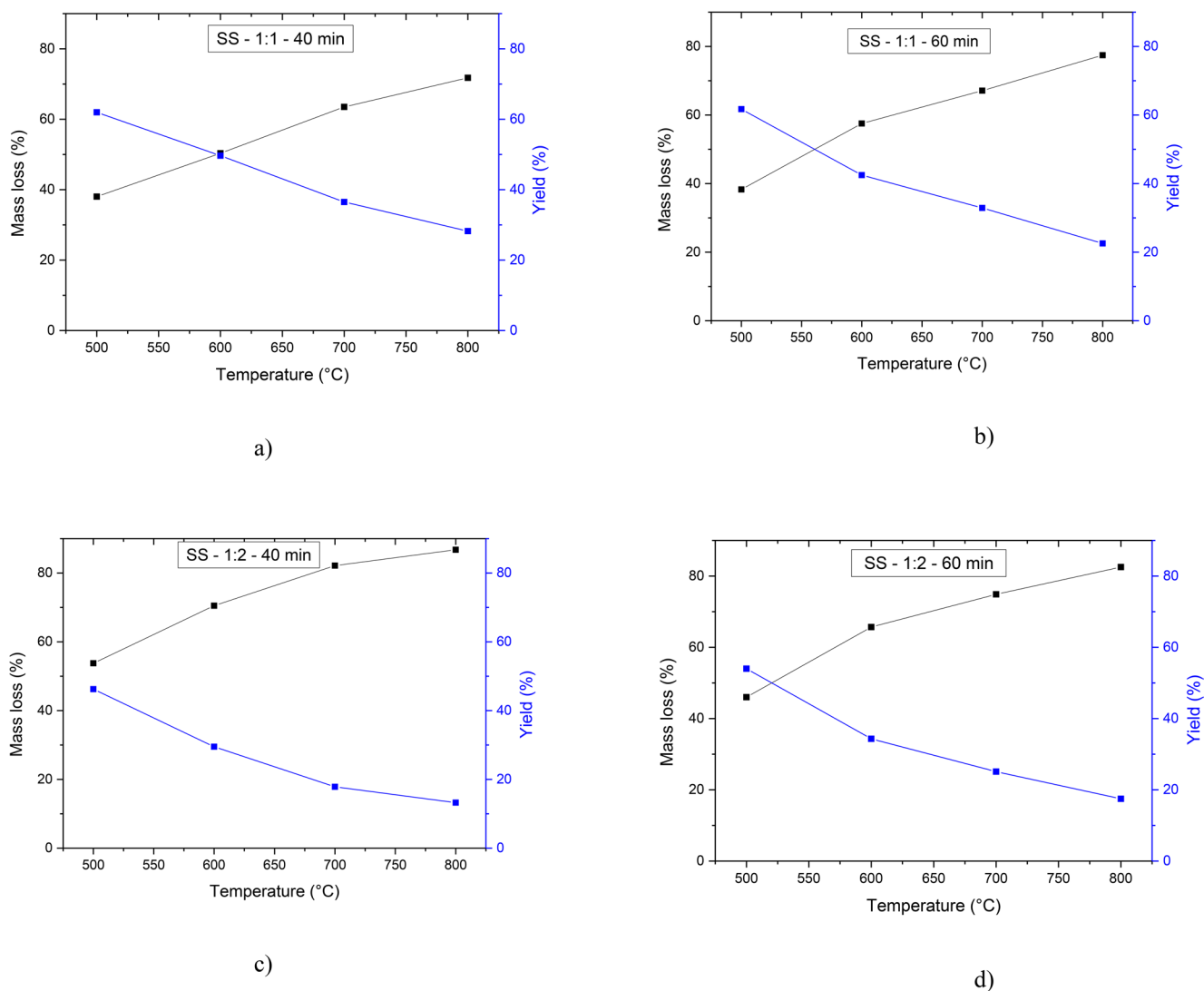
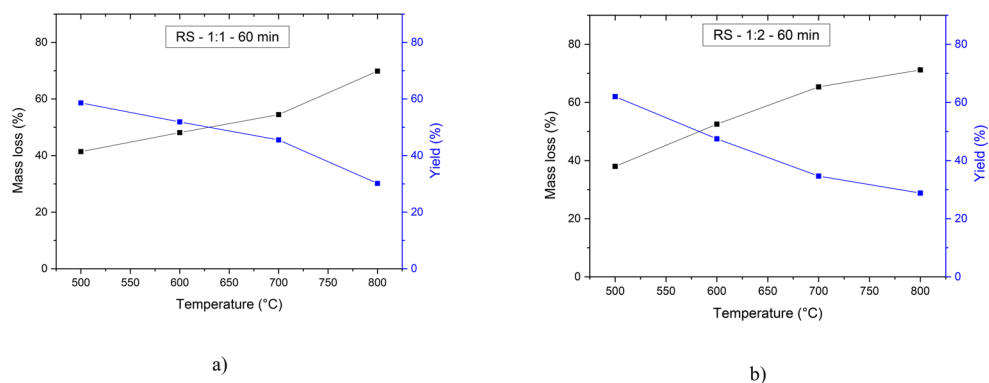


Fig. 6 Mass loss and yield as a function of the final temperature for two different plateaux **a)** SS-1:1-40, **b)** SS-1:1-60, **c)** the SS-1:2-40 and **d)** SS-1:2-60

Fig. 7 Mass and yield as a function of the final temperature **a)** RS-1:1-60 and **b)** RS-1:2-60



$\text{cm}^3 \cdot \text{g}^{-1}$, a microporous surface area of $517 \text{ m}^2 \cdot \text{g}^{-1}$ and a total surface area of $687 \text{ m}^2 \cdot \text{g}^{-1}$. These results show that with this Raw material and under these operating conditions, as the temperature increases, the microporous volume

decreases as well as the average micropore width. These ACs are mostly microporous with a microporosity percentage between 80.3% and 58.2% depending on the average micropore width. This type of activated carbon, with this

Table 3 Textural characteristics of activated carbons from RS raw materials

	W_0 (cm ³ /g)	L_{01} (nm)	L_{02} (nm)	S_{BET} (m ² /g)	S_{ext} (m ² /g)	S_{micro} (m ² /g)	S_{tot} (m ² /g)	% Micro	% Meso
RS 1:1 plateau 60 min									
RS-1:1-500-60	0.35	1.33	-	956	129	526	655	80.3	19.7
RS-1:1-600-60	0.27	1.70	-	751	228	318	546	58.2	41.8
RS-1:1-700-60	0.21	1.27	-	612	183	331	514	64.4	35.6
RS-1:1-800-60	0.31	1.20	-	875	170	517	687	75.2	24.8
RS 1:2 plateau 60 min									
RS-1:2-500-60	0.44	2.06	-	1313	533	427	960	44.5	55.5
RS-1:2-600-60	0.41	1.85	-	1224	527	443	970	45.7	54.3
RS-1:2-700-60	0.36	1.39	-	1045	417	518	935	55.4	44.6
RS-1:2-800-60	0.36	1.41	-	1041	370	511	881	58.0	42.0
RS 1:2 plateau 40 minutes									
RS-1:2-800-40	0.46	1.53	-	1311	437	601	1038	57.9	42.1
L27	0.57	1.85	-	1575	444	616	1060	58.1	41.9

Table 4 Textural characteristics of activated carbons from SS Raw materials

	W_0 (cm ³ /g)	L_{01} (nm)	L_{02} (nm)	S_{BET} (m ² /g)	S_{ext} (m ² /g)	S_{micro} (m ² /g)	S_{tot} (m ² /g)	Micro (%)	Meso (%)
SS 1:1 plateau 40 min									
SS-1:1-500-40	0.51	1.43	2.17	1410	111	713	824	86.5	13.5
SS-1:1-600-40	0.41	1.16	2.07	1161	26	707	733	96.4	3.6
SS-1:1-700-40	0.39	1.27	-	1076	76	614	690	89.0	11.0
SS-1:1-800-40	0.42	1.23	2.14	1192	92	683	775	88.1	11.9
SS 1:1 plateau 60 min									
SS-1:1-500-60	0.51	1.45	-	1398	136	703	839	83.8	16.2
SS-1:1-600-60	0.40	1.27	-	1109	114	630	744	84.7	15.3
SS-1:1-700-60	0.41	1.19	-	1166	89	689	778	88.6	11.4
SS-1:1-800-60	0.49	1.24	-	1377	68	790	858	92.1	7.9

kind of porosity, could be used in particular in gas phase adsorption.

On the other hand, activated carbons obtained from RS with phosphoric acid (1:2 ratio, with a 60-minute stop at the final temperature) have better porous properties than those obtained with a 1:1 ratio, due to the higher amount of phosphoric acid with a higher external surface value. Between 500 °C and 800 °C, the microporous volume decreases from 0.44 cm³.g⁻¹ to 0.36 cm³.g⁻¹ (18% decrease), the microporous surface area increases from 427 m².g⁻¹ to 511 m².g⁻¹ (16% increase), the external surface area decreases from 533 m².g⁻¹ to 370 m².g⁻¹ (31% decrease) and the total surface area decreases from 960 m².g⁻¹ to 881 m².g⁻¹ (8% decrease). These results show that with this raw material and under these operating conditions, as the temperature increases, all porous property values decrease except for the microporous surface area which increases. The ACs in this series have microporous and mesoporous domains with a microporosity percentage between 58.0% and 44.5%. This kind of activated carbon could be used in liquid phase adsorption. Depending on the parameters studied, it is interesting to note that with a single raw material, it is possible to obtain different ranges of AC for different applications.

The raw SS was studied by varying the plateau at the final activation temperature (40 min and 60 min), the ratio of raw material to activating agent (1:1 and 1:2) and the final activation temperature (between 500 °C and 800 °C).

Table 4 presents the textural characteristics of activated carbons from SS raw materials as a function of these parameters. The textural properties of the commercial activated carbon, L27, are also included.

The 40-minute plateau, 1:1 ratio series shows that as the activation temperature increases (between 500 °C and 800 °C), the overall values of the porous properties decrease. These activated carbons are highly microporous with a predominant microporous domain between 86.5% and 96.4%. Increasing the step to 60 min does not significantly change the values of the porous properties obtained previously.

Increasing the amount of phosphoric acid, with a ratio of 1:2, significantly increases the external surface area, resulting in an increase in the mesoporous domain. There is also an increase in the average micropore width. In this SS series with a 1:2 ratio, a 60-minute step increases the porous properties, with microporous volumes between 0.54 cm³.g⁻¹ and 0.69 cm³.g⁻¹.

The ACs obtained from the raw material SS have more developed porous properties than the ACs obtained from the raw material RS. It is interesting to observe that ACs obtained from SS have porous properties very similar to those of the commercial activated carbon L27 (Jacobi Carbons) which is widely used in liquid phase adsorption and is very efficient for the adsorption of many organic molecules [17, 34, 35].

The graphical representation in Fig. 8, also called the “ L_0/W_0 diagram” [29] which represents the evolution of the average micropore width (L_0) as a function of the microporous specific volume (W_0) makes it possible to compare the materials obtained with the two raw materials. In addition, this diagram is a very important tool, as it enables activated carbons produced by different conventional methods (chemical activation and physical activation) to be compared, and

new elaboration protocols to be defined for particular applications. In this representation, which concerns only microporosity, three areas were defined:

- The beginning of Domain I corresponding to microporous materials with narrow pore sizes (in particular carbonizations and molecular sieves),
- Domain II accounts for the majority of common commercial activated carbons,
- Domain III is characteristic of samples with poorly developed heterogeneous porosity.

Figure 8a shows that the ACs obtained from the raw material SS are close to domain 2. The SS 1:1 series with a 40 or 60-minute plateau are very close and are in the middle of domain 2. On the other hand, the SS series 1:2 with a

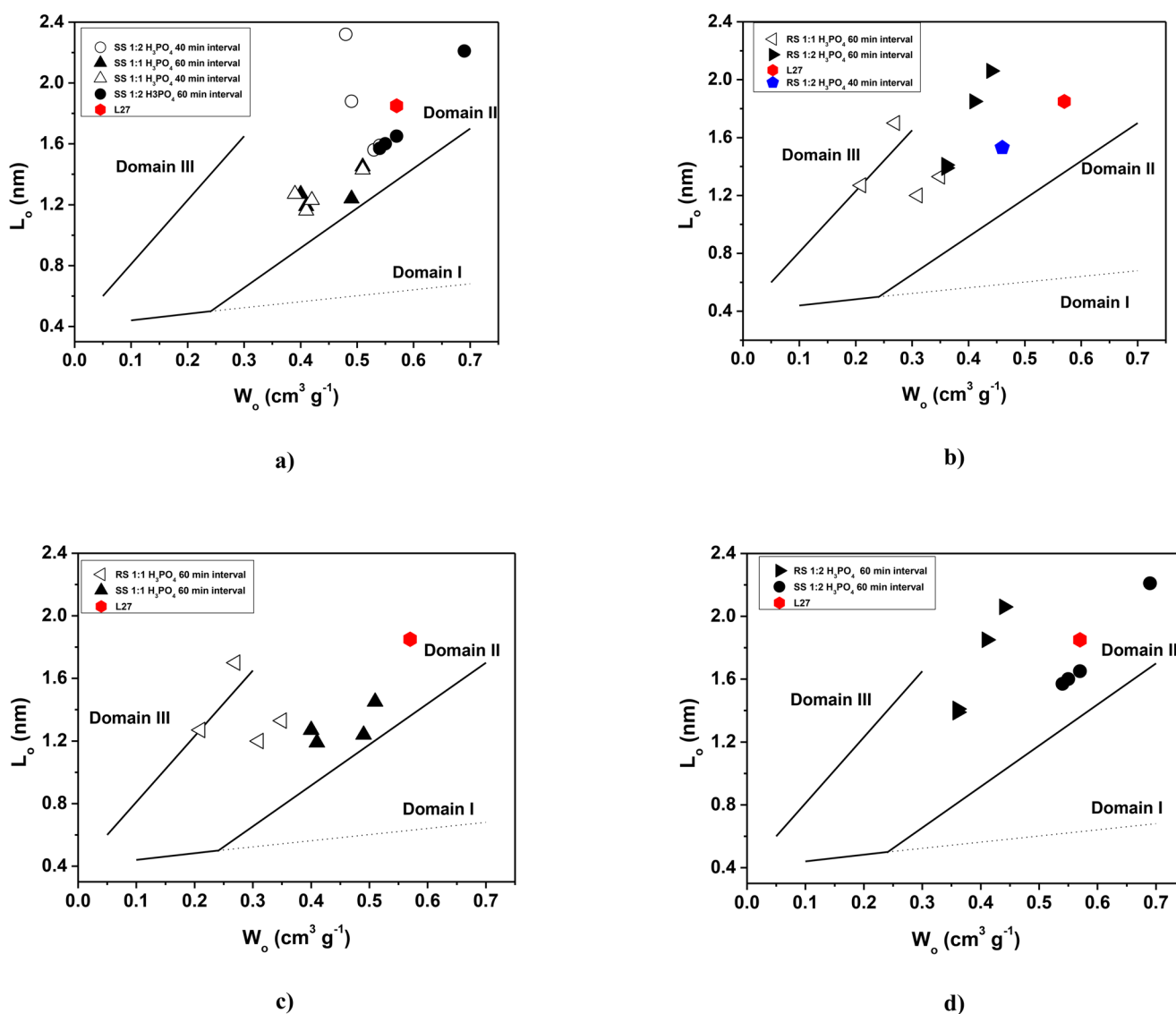
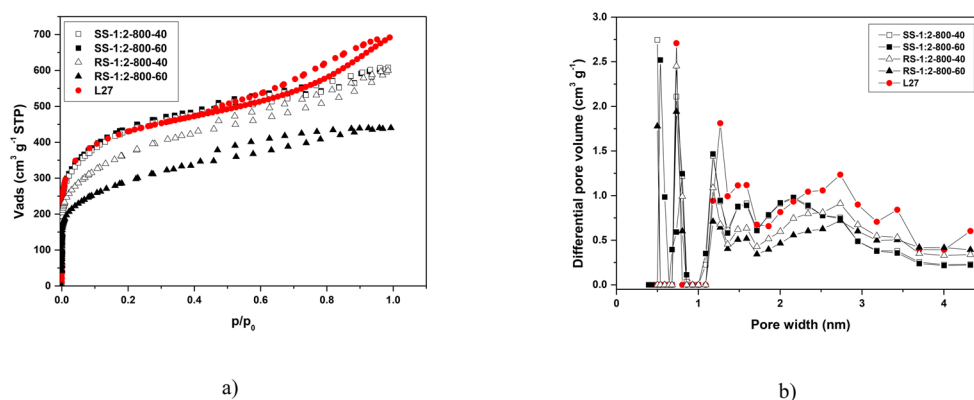


Fig. 8 Graphical representation, also called the “ L_0/W_0 diagram” for the activated carbons SS, RS and L27

Fig. 9 a) Adsorption and desorption isotherms of N_2 to 77 K and b) distribution of micropore sizes (DFT) of activated carbons made from SS and RS (ratio 1:2) with phosphoric acid



60-minute step is very interesting and is at the top of domain 2 (specific microporous volume developed with large micropores). The SS 1:2 series with a 40-minute step has a different, more vertical, tendency than the one with the 60-minute step between domains 2 and 3. All of these ACs can find applications in liquid phase adsorption because they are close to the commercial AC L27. RS ACs activated with a 1:1 and 1:2 ratio are less well-developed than SS ACs. They are located between domains 3 and 2 (Fig. 8b) and are further away from AC L27. On the other hand, the 1:2 RS AC at 800 °C, with a 40-minute plateau, seems to show interesting porous properties. Figure 8c and 8d show that SS ACs are closer to domain 2 than RS ACs. Nevertheless, these figures show that the two raw materials are readily activable and offer very interesting ACs for liquid phase adsorption with different porous properties.

3.2.4 Isotherms of N_2

Figure 9a shows the adsorption and desorption isotherms of nitrogen at 77 K and the micropore size distributions (according to DFT theory from Micromeritics Software) for activated carbons produced from two types of precursors, namely SS and RS with a 1:2 ratio. The three curves show hysteresis shapes at $P/P_0=0.45$, indicating the presence of mesopores. The shapes of the isotherm curves of AC SS-1.2–800 °C-60 min and SS-1.2–800 °C-40 min are similar. Figure 9b shows that the size distributions of the

micropores present two microporous domains for the three samples: a first domain between 0.5 and 1 nm and a second wider domain between 1 and 2 nm, a value at the limit of the mesoporous domain.

3.2.5 Predictive Calculation

The predictive calculation set up and detailed by Boundzanga et al. [1] was applied to heat treatment with a chemical activating agent in order to estimate the burn-off rates of each lignocellulosic precursor and to estimate the respective contribution of each compound (cellulose, hemicelluloses and lignin) to the carbon mass of the final material. The predictive calculation used here for chemical activation uses all the equations detailed in [1]. The results obtained from this predictive calculation are shown in Table 5. They concern chemical activation with phosphoric acid for RS and SS, at 500 °C and 800 °C, for a ratio of 1:1 and 1:2 for a plateau time at the final temperature of 60 min.

For AC made from RS, lignin-derived carbon ranges from 22.6% to 28.3%, cellulose-derived carbon from 49.5% to 58.3% and hemicelluloses-derived carbon from 16.7% to 22.3%. Whatever the activation parameters, cellulose is the majority compound contributing to carbon production in the final AC RS. For AC made from SS, lignin-derived carbon ranges from 47.3% to 54.3%, cellulose-derived carbon from 26.2% to 33.2% and hemicelluloses-derived carbon from 15.9% to 20.7%. In this case, lignin is the predominant

Table 5 Contribution of each fraction in the activated carbons composition (SS and RS) estimated by the predictive calculation

	Production H. (H _C %)	Production C. (C _C %)	Production L. (L _C %)	calculated yield (%)	experimental yield (%)	Deviation from the average (%)
RS						
RS-1.1–500.1-60'	22.3	49.5	28.3	63.1	58.6	2.2
RS-1.2–500.2-60'	21.7	55.6	22.6	66.9	62.0	2.5
RS-1.2–800.2-60'	16.7	58.3	24.7	23.1	29.0	3.0
SS-1.1–500.1-60'	19.5	26.2	54.3	61.9	62.0	0.1
SS-1.2–500.2-60'	20.7	32.1	47.3	68.6	46.0	11.3
SS-1.2–800.2-60'	15.9	33.2	50.1	25.9	17.5	4.2

compound contributing to carbon production in the final AC SS. For the two raw materials studied, as the ratio increases, there is an increase in the carbon content of cellulose and a decrease in the carbon content of lignin. Boundzanga et al. [1] showed that, overall, lignin was responsible for microporosity and hemicelluloses and cellulose were responsible for mesoporosity. This is confirmed in this work by combining the results of Fig. 9; Tables 3, 4 and 5.

The ACs prepared from SS, which contain more carbon from lignin, have a higher proportion of micropores than the ACs prepared from RS, which contain more cellulose and which have a higher proportion of mesopores than ACs from SS.

3.3 Kinetics of Ibuprofen Adsorption on ACs

The ACs with the best porous properties were selected and tested for their ibuprofen adsorption capacities. They were compared to the L27. The evolution of the adsorption capacity as a function of time at 20 °C and pH=6 for each activated carbon (L27, SS-1:2–800 °C-40 min and 60 min, RS-1:2–800 °C-40 min and 60 min) for the ibuprofen molecule are shown in Fig. 10. For each activated carbon, the adsorption equilibrium time differed: 90 min for RS-1:2–800 °C-40 min, 120 min for SS-1:2–800 °C-60 min, 240 min for SS-1:2–800 °C-40 min and RS-1:2–800 °C-60 min and 300 min for L27. The rapid fixation is due to the great affinity of activated carbon for the retention of ibuprofen. Similar results were also reported by Gbamele [36]. As shown in Fig. 10, the adsorption kinetics are correctly described by the pseudo-second-order kinetic model in its linear form, with correlation coefficients all greater than 0.99 (Table 6). Activated carbons produced from RS or SS all have a higher adsorption capacity than commercial activated carbon L27 (117 mg.g⁻¹). The two RS ACs have the same adsorption capacity (129 mg.g⁻¹) and the two ACs made from

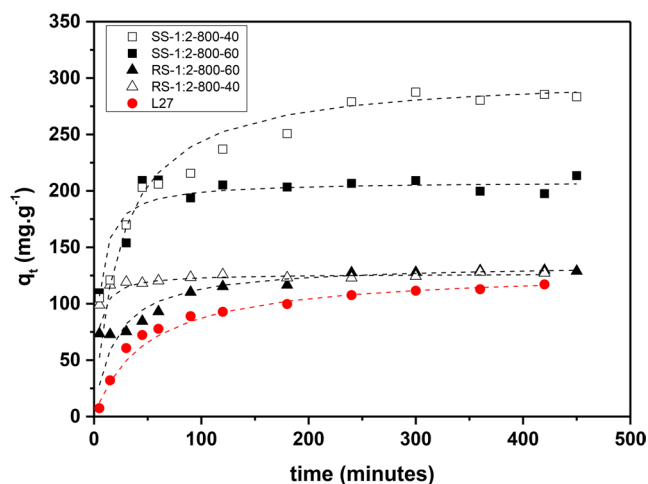


Fig. 10 Kinetics of ibuprofen adsorption onto five activated carbons

Table 6 Pseudo-second order kinetic model for ibuprofen adsorption at 20 °C

L27	$q_{e \text{ exp}}$ (mg g ⁻¹)	$q_{e \text{ theo}}$ (mg g ⁻¹)	k_2 (g g ⁻¹ min ⁻¹)	R^2
	117	127	0.00020	0.999
RS-1.2–800 °C-40 min	129	130	0.00170	0.999
SS-1.2–800 °C-40 min	285	303	0.00013	0.998
RS-1.2–800 °C-60 min	129	135	0.00038	0.998
SS-1.2–800 °C-60 min	200	208	0.00101	0.997

SS have the best adsorption capacities (200 mg.g⁻¹ and 285 mg.g⁻¹, respectively, for SS-1:2–800 °C-40 min and SS-1:2–800 °C-60 min). This difference is closely related to the porous properties of the materials: SS ACs have a larger microporous surface area than RS ACs and L27; in addition, they have a large external surface area.

3.4 Equilibrium of Ibuprofen Adsorption on Activated Carbons

An equilibrium time of 360 min was chosen for the study of adsorption isotherms for the 5 activated carbons. The adsorption isotherms, shown in Fig. 11, are correctly described by the Langmuir model, with correlation coefficients all greater than 0.987 (Table 7).

Table 7 shows that the maximum single-layer adsorption capacity (q_{mL}) was obtained with AC SS-1.2–800 °C-40 min (294 mg.g⁻¹), followed by SS-1.2–800 °C-60 min (227 mg.g⁻¹), RS-1.2–800 °C-40 min (185 mg.g⁻¹), RS-1.2–800 °C-60 min (167 mg.g⁻¹) and L27 (147 mg.g⁻¹). The q_{mL} values give access to the occupied area of the activated carbon. This contact surface was calculated using the Chemskech software, considering that the adsorption of the ibuprofen molecules takes place in a horizontal position.

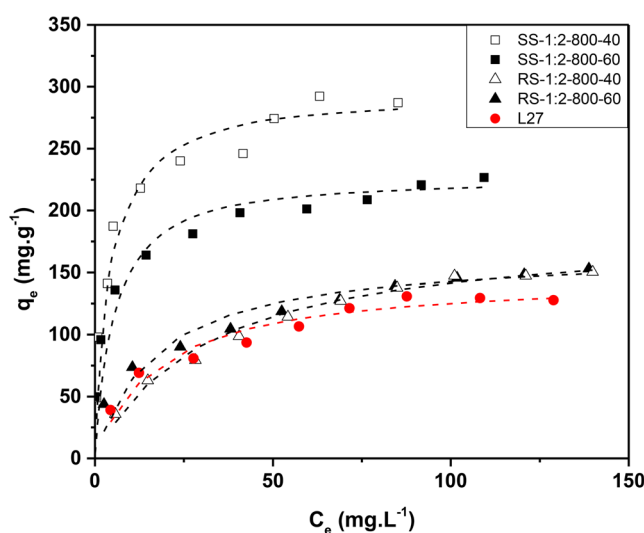


Fig. 11 Equilibrium adsorption isotherms of ibuprofen onto five activated carbons at 20 °C

Table 7 Langmuir isotherm constants for ibuprofen at 20 °C

L27	$q_{m \text{ exp}}$ (mg g ⁻¹)	$q_{mL \text{ theo}}$ (mg g ⁻¹)	b (L mg ⁻¹)	R^2	$S_{\text{micro}^{\circ}\text{occ}^{\circ}}$ (m ² .g ⁻¹)	$S_{\text{micro}^{\circ}\text{occ}^{\circ}}$ (%)
	130	147	0.057	0.987	201	33
RS-1.2–800 °C-40 min	152	185	0.325	0.992	256	43
SS-1.2–800 °C-40 min	290	294	0.272	0.995	403	59
RS-1.2–800 °C-60 min	150	167	0.060	0.989	229	45
SS-1.2–800 °C-60 min	219	227	0.234	0.995	312	45

This implies that there is an overlap of the aromatic ring of the molecules on the aromatic rings of the activated carbon. In this configuration, the surface area of the molecule was determined: $S_{\text{ibuprofen}} = 4.70.10^{-19} \text{ m}^2$.

According to the following equation, the occupied surface can be calculated:

$$S_{\text{occ.}} = q_{mL} \times N_A \times S_{\text{ibuprofen}} \quad (3)$$

with q_{mL} converted to mol.g⁻¹ and N_A Avogadro's number in mol⁻¹.

The surface area occupied by ibuprofen is significant on the AC SS-1.2–800 °C-40 min, with 59% of the microporous surface area occupied compared to the L27 where only 33% of the surface area is occupied. This may be due to the fact that L27 contains many oxygenated surface groups that no doubt slow down and limit access to the micropores that are the site of adsorption despite a well-developed porosity. The other three ACs have an occupied surface area of 43% and 45%. The ideal activated carbon for ibuprofen adsorption should possess the textural properties of SS-1.2–800 °C-40 min without too many oxygenated surface functional groups.

The two activated carbons derived from Saba Senegaleensis, namely SS-1.2–800.2-40 min and SS-1.2–800.2-60 min, exhibit superior adsorption capacities compared to the activated carbons obtained from Ronier Shell (RS-1.2–800.2-40 min and RS-1.2–800.2-60 min). Transmission Electron Microscopy (TEM) analysis of the RS-1.2–800.2-40 min material (Fig. 12a) reveals a dense and poorly aerated morphology, while the activated carbon SS-1.2–800.2-40 min (Fig. 12b) shows a structure composed of organized lamellar fibers, characterized by a dilated morphology, lower density, and relatively well-aerated open porosity (red circle).

Increasing the activation time to 60 min promotes the opening of the porosity of RS-1.2–800.2-60 min, which then resembles that observed for SS-1.2–800.2-40 min. However, this extension of treatment also leads to a significant alteration of the structure (green circles, Figs. 12c). This degradation is particularly pronounced for the activated carbon SS-1.2–800.2-60 min (green circle, Fig. 12d), whose porosity is denser and less open compared to that of SS-1.2–800.2-40 min.

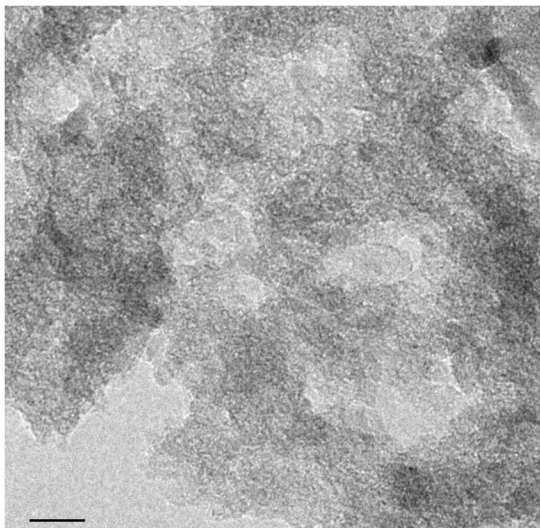
Indeed, the increase in thermal treatment time from 40 to 60 min causes structural deformation, regardless of the starting material used (Fig. 12e and f, orange circles). This explains why the two activated carbons, SS-1.2–800.2-40 min and SS-1.2–800.2-60 min, exhibit very similar or even identical porous characteristics (Table 5). Despite this similarity, the activated carbon SS-1.2–800.2-40 min stands out due to its highest ibuprofen adsorption capacity and a significant occupied surface area.

4 Conclusions

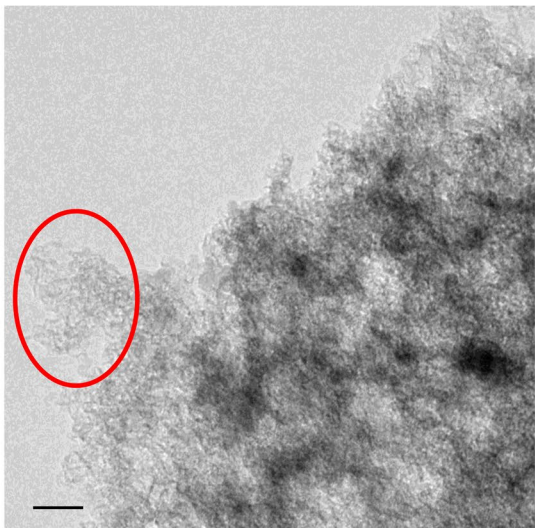
In light of the results of the biochemical analyses reported here, raw SS and RS biomasses are good precursors for the production of activated carbon.

The SS and RS shells were used for the production of activated carbon by chemical activation with H₃PO₄. The influence of final carbonization temperatures (500 °C to 800 °C) and impregnation ratios (1:2 and 1:1) on activated carbon yield, specific surface area and pore development were studied. The yields of activated carbons decrease with the increase in the mass quantity of the activating agent, the impregnation ratio and the carbonization temperature. The carbonization temperature and the impregnation ratio have a significant effect on the pore characteristics of the activated carbons produced. It was observed that the specific surface area, the BET surface area with a maximum value obtained at 500 °C and the pore volume of SS and RS activated carbons increase with the increase in the mass of the activating agent of the impregnation ratio. An increase in carbonization temperature from 500 °C to 800 °C can induce shrinkage of the carbon structure, leading to a reduction in pore surface area. The SS and RS activated carbons obtained with an impregnation ratio 1:2 both have microporous and mesoporous structures suitable for adsorption in the liquid phase, whereas those with an impregnation ratio 1:1 are very microporous and therefore suitable for adsorption in the gas phase. Phosphoric acid acts as a modifying

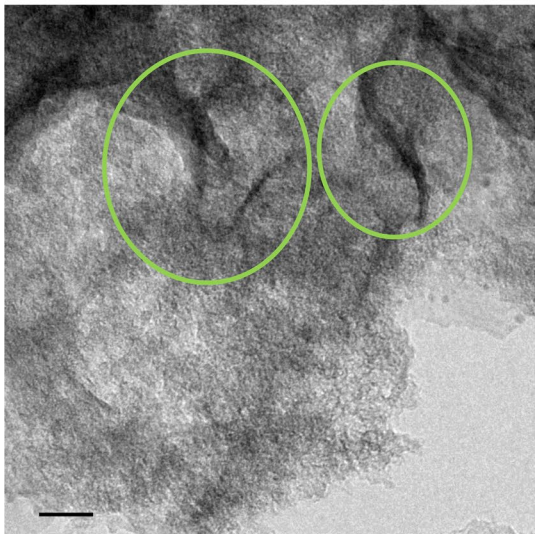
Fig. 12 TEM Images of activated carbons : **a**) RS-1.2-800°C-40min (66k), **b**) SS-1.2-800°C-40min (66k), **c**) RS-1.2-800°C-60min (66k), **d**) SS-1.2-800°C-60min (66k), **e**) RS-1.2-800°C-60min (5k) and **f**) SS-1.2-800°C-60min (5k)



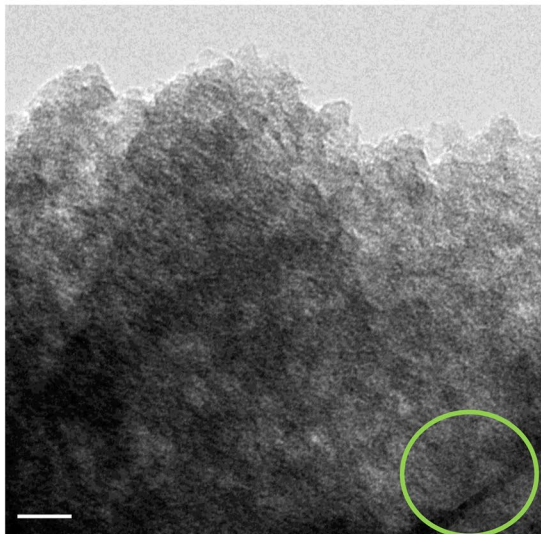
a) RS-1.2-800°C-40min (66k)



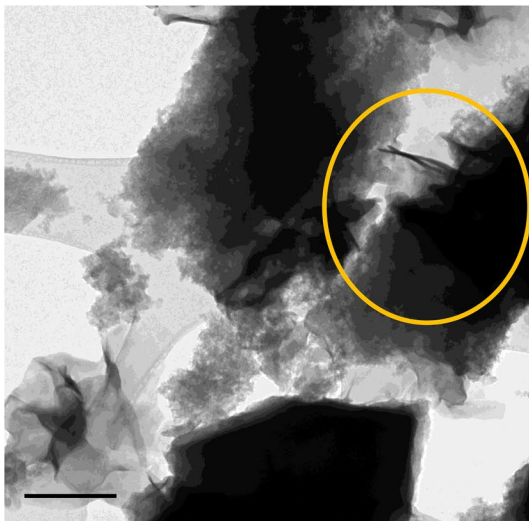
b) SS-1.2-800°C-40min (66k)



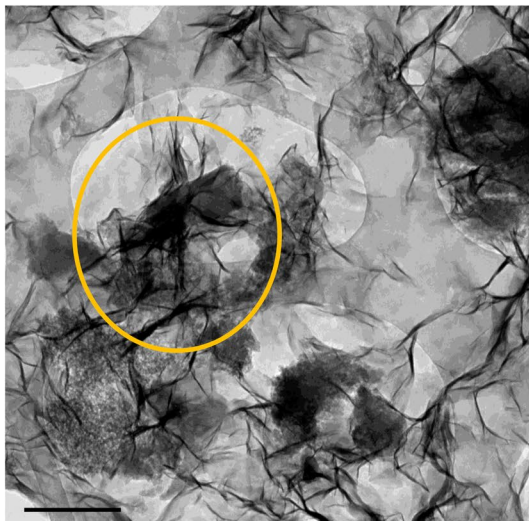
c) RS-1.2-800°C-60min (66k)



d) SS-1.2-800°C-60min (66k)



e) RS-1.2-800°C-60min (5k)



f) SS-1.2-800°C-60min (5k)

agent, increasing porosity and giving rise to surface groups of an acidic nature. Consequently, four activated carbons obtained with a ratio of 1:2 were selected as adsorbents for the adsorption of ibuprofen in synthetic wastewater in comparison with a commercial activated carbon L27. Among the suggested adsorbents, SS and RS activated carbons with an impregnation ratio 1:2 showed more promising results than commercial activated carbon L27 in the removal of ibuprofen from synthetic wastewater under the conditions studied. The adsorption was found to follow the pseudo-second-order kinetic model and the Langmuir isotherm model indicating the chemisorption process and adsorption on a monolayer, respectively. Other studies could be carried out to find the possible application of other SS and RS activated carbons developed for the elimination of other organic pollutants.

Author Contributions Lamine Diouf: the conception and design, analysis and interpretation of the data; the drafting of the paper; Saïdou Diop: analysis and interpretation of the data; the drafting of the paper, revising it critically for intellectual content; Benoît Cagnon: analysis and interpretation of the data; the drafting of the paper, revising it critically for intellectual content.

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Declarations

Ethical Approval Not applicable.

Informed Consent Not applicable.

Conflict of Interest There is no financial or non-financial interests that are directly or indirectly related to the work submitted for publication.

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