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Green Activation of Activated Carbons Prepared from Palm Nut Shells: A Green Approach Based on Extracts of Gabonese Plants

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Abstract

This study presents a green approach for the preparation of activated carbons (ACs) from biomass (palm nut shells) using aqueous extracts of Gabonese plants (*Hallea ciliata* (HC), *Ricinodendron heudelotii* (RH) and *Nauclea latifolia* (NL)) as bioactivation agents. The objective was to evaluate the influence of secondary metabolites (plant biomolecules) present in the aqueous extracts on the pore structure and surface chemistry of resulting bioactivated carbons, with a view to applications in water treatment in rural zones. Phytochemical screening and antioxidant analyses revealed that the extracts are rich in secondary metabolites. The HC extract exhibited the highest contents of polyphenols (2418.49 µg GAE/g), tannins (1175.73 µg GAE/g) and proanthocyanidins (2069.24 µg GAE/g), as well as strong antioxidant activity (AAI = 3.47). Activated carbons obtained from green activation showed improved textural properties compared to the water-impregnated reference material (AC_{-water}). In particular, the specific surface area increased from 367 m^2/g for AC_{-water} to 385 - 447 m^2/g for the activated carbons obtained from green activation, with preferential development of microporosity. The AC_{-HC} sample exhibited the most developed surface properties (S_{BET} = 446.51 m^2/g, micropore volume = 0.144 cm^3/g) and the highest adsorptive capacity (iodine number = 520 mg/g). These results demonstrate that secondary metabolites from aqueous extracts of Gabonese medicinal plants can act as effective green activation agents for the preparation of microporous activated carbons through an environmentally friendly process under the experimental conditions investigated.

Keywords

Biomass, Gabonese Medicinal Plants, Secondary Metabolites, Green Activation, Bioactivated Carbons, Water Depollution

1. Introduction

In Gabon, access to safe drinking water remains a major public health challenge, particularly in rural areas where water treatment and distribution infrastructures are limited. Rural populations rely mainly on surface waters, which are frequently exposed to natural and anthropogenic sources of contamination. These water resources often contain pathogenic agents such as bacteria, viruses, and protozoa, responsible for numerous waterborne diseases, including diarrhea, dysentery and typhoid fever [1] [2]. Furthermore, the country possesses significant agricultural and plant resources whose valorization remains largely underexploited. Agricultural residues and local medicinal plants, although abundant, renewable and easily accessible, represent a considerable potential source of raw materials that can be exploited to develop local and sustainable solutions to combat waterborne diseases and reduce the contamination of surface waters accessible to rural populations. In this context, the use of porous materials such as activated carbon for water purification or filtration could be one solution.

Activated carbon is widely recognized for its excellent adsorption properties against organic and inorganic contaminants present in water [3]-[5]. However, conventional industrial production processes are often costly and difficult to access for rural populations [3] [5] [6]. The preparation of activated carbon generally involves two essential steps: carbonization and activation. Carbonization consists of heating the raw material (or biomass) at temperatures ranging from 400 to 1000˚C in an inert atmosphere (nitrogen or argon) in order to remove volatile components and concentrate the carbonaceous matter [7] [8]. Activation subsequently allows the development of porosity and specific surface area of the material [9] [10]. This activation can be achieved through either physical or chemical methods. Physical activation employs steam or carbon dioxide (CO_2) at high temperatures (700˚C - 1000˚C) [9], whereas chemical activation involves the prior impregnation of the biomass with chemical activating agents such as phosphoric acid (H_3PO_4) [9] [11], potassium hydroxide (KOH) [5] [9] [11] or zinc chloride ($ZnCl_2$) [3] [11] [12].

From a sustainable development perspective, the production of activated carbon from locally available biomasses (nut shells, corn stalks, rice husks, sawdust, etc.) using environmentally friendly processes represents an economically viable alternative. In this context, the development of alternative pathways inspired by the principles of green chemistry has attracted increasing interest. These approaches aim to reduce the environmental impact of synthesis processes, notably through the use of moderate temperatures, natural activating agents and less aggressive operating conditions. Among these strategies, bioactivation (process at the end of which secondary metabolites act and participate in the activation of activated carbons) methods based on plant-derived substances appear particularly promising.

Gabon, rich in exceptional plant biodiversity, represents an invaluable reservoir of medicinal plants traditionally used for their therapeutic properties, making them valuable resources for the treatment of numerous diseases [13] [14]. Several species widely distributed in Gabonese forests have demonstrated significant antibacterial and antifungal activities [13]-[15]. These include *Hallea ciliata* and *Annickia chlorantha* (antiplasmodial, antibacterial, antirheumatic properties, etc.) [16] [17], *Ricinodendron heudelotii* and *Nauclea latifolia* (treatment of dysentery, respiratory and digestive infections, including diarrhea) [13] [18] and *Alchornea cordifolia* (antifungal and anti-inflammatory activities) [19]. The aqueous extracts of these plants, rich in bioactive compounds such as polyphenols, flavonoids, tannins and essential oils, possess chemical functionalities that may act as bioactivating agents during the preparation of activated carbons, thereby promoting the development of porosity and surface area.

The combined use of local biomasses and plant aqueous extracts for the preparation of activated carbons intended for water purification (rivers, lakes, etc.) represents an innovative, sustainable solution adapted to the specific needs of rural zones in Central Africa and particularly in Gabon.

The objective of this study is therefore to develop a green method for preparing activated carbons from palm nut shells using biomolecules derived from aqueous extracts of *Hallea ciliata*, *Ricinodendron heudelotii* and *Nauclea latifolia*. Particular attention is paid to the influence of secondary metabolites derived from aqueous extracts of plants on the pore structure and surface chemistry of the resulting activated carbons, with a view to water depollution applications in rural environments.

2. Experimental

2.1. Preparation of Plant Extracts

The leaves of *Hallea ciliata* (HC), *Ricinodendron heudelotii* (RH) and *Nauclea latifolia* (NL) were collected in Mounana, Haut-Ogooué Province, south-eastern Gabon. These leaves were dried at room temperature and protected from light for several days. After drying, the plants were ground using a laboratory blender (Laboratory Blender, Torrington, CT, USA). The resulting powder was used for maceration extractions. 10 g of powder were mixed with 200 mL of distilled water. The mixture was stirred under magnetic agitation for 24 h at room temperature. The aqueous extracts were then obtained by vacuum filtration of the solution. The experiment was repeated three times to confirm the results.

2.2. Phytochemical Screening

Phytochemical screening was carried out on all aqueous extracts to detect the presence of secondary metabolites (alkaloids, flavonoids, saponins, quinones, gallic tannins, polyphenols, reducing sugars…) as described in the works of Ngoua-Meye-Misso *et al.* [20].

2.3. Determination of Secondary Metabolite Contents in Aqueous Extracts

The total polyphenol content of the different extracts was determined using the Folin-Ciocalteu method [21], with gallic acid as the calibration standard. 250 µL of extract was mixed with 125 µL of Folin-Ciocalteu reagent and 1 mL of 7.5% sodium carbonate solution (Na_2CO_3, RLGLET; purity 99.5%). The mixture was incubated at room temperature for 30 min. Polyphenol concentrations were obtained using a UV-Visible spectrophotometer (Thermo Electron Corporation, Biomate 5) at 765 nm. Results were expressed as micrograms of gallic acid equivalents (GAE) per gram of dry plant material.

The total flavonoid content of the extracts was determined by the aluminum chloride ($AlCl_3$) colorimetric method [22] [23], using quercetin as the reference standard. 1 mL of extract was mixed with 1 mL of 2% aluminum chloride solution ($AlCl_3$, Prolabo Scientifique; purity 98.5%). After incubation at room temperature for 10 min, absorbance was measured with a UV-Visible spectrophotometer (Thermo Electron Corporation, Biomate 5) at 415 nm. The results were expressed as micrograms of gallic acid equivalent (GAE) per gram (g) of dry plant material.

Tannin content was determined according to the reference method of the European Community [20]. 250 µL of extract was mixed with 1 mL of ammonia solution (NH_3, Atlanticlabo; purity 20%) at 8 g/L and 250 µL of ferric ammonium citrate solution (28% iron and 3.5% ammonia; Atlanticlabo; purity 20%). Absorbance was measured using a UV-Visible spectrophotometer (Thermo Electron Corporation, Biomate 5) at 525 nm. Results were expressed as micrograms of tannic acid equivalents (TAE) per gram of crude extract.

The proanthocyanidin content was determined following the method described by Prigent [24] and subsequently used by Ngoua-Meye-Misso *et al.* [20]. 170 µL of the extract was mixed with 2.3 mL of an HCl-butanol reagent, prepared from hydrochloric acid (HCl, Emsure; purity 33%) and butanol (Atlanticlabo; purity 98%). The mixture was incubated at room temperature for 1 h, then the absorbance was measured using a UV-Visible spectrophotometer (Thermo Electron Corporation, Biomate 5) at 550 nm. Results were expressed as apple proanthocyanidin equivalents (APE).

2.4. Study of Antioxidant Activity

2.4.1. Measuring the Antiradical Activity of Plants

The antiradical activity was evaluated based on the reduction of DPPH (2,2-diphenyl-1-picrylhydrazyl) and adapted from the method described by Scherer and Godoy [25] to determine the antioxidant activity index (AAI). A 2 mg/mL solution of plant extract was prepared by dissolving 12 mg of dried crude extract in 6 mL of ethanol (RSBC Scientific Business Company; purity 96%). Working solutions ranging from 1 to 1000 µg/mL were then prepared by serial dilutions. For each sample, a 0.1 M DPPH solution (The solvent used is ethanol at 98% for the preparation of DPPH solution and the baseline was established with the solvent; BOC; purity 98%) was added to the extract solution. The mixtures were incubated for 30 min at room temperature in the dark. Absorbance was measured at 517 nm using a UV-Visible spectrophotometer (Thermo Electron Corporation, Biomate 5). Ascorbic acid (Vitamin C) was used as reference and the percentage inhibition (%I) of the DPPH free radical was determined by Equation (1) [26].

$$\%I = \frac{\left(A_{\text{DPPH}} - A_{\text{extract}}\right)\times 100}{A_{\text{DPPH}}} \tag{1}$$

where %I is the percentage of inhibition (%I) of the DPPH radical, A_{DPPH} is the optical density of the DPPH radical and A_{extract} is the optical density of the aqueous extract.

The IC_{50}, defined as the concentration required to scavenge 50% of DPPH radicals, was determined by plotting the percentage inhibition curve as function of extract concentrations. The antioxidant activity index (AAI) of an extract was defined in Equation (2) according to Scherer and Godoy [25]:

$$\text{IAA} = \frac{[\text{DPPH}]_{\text{f}}}{\text{IC}_{50}} \tag{2}$$

where IAA is the index of antioxidant activity, $[\text{DPPH}]_f$ is the final concentration of DPPH in µg/mL and IC_{50} is the concentration required to reduce 50% of the DPPH radical.

2.4.2. Evaluation of Total Antioxidant Activity

The phosphomolybdenum test is defined as a test for antioxidant capacity and is used to confirm the results of the antioxidant activity of plant extracts. It involves a qualitative and quantitative evaluation of the antioxidant activity index [27]. The assessment of antioxidant activity is based on the reduction of Mo(VI) to Mo(V) and the subsequent formation of a green phosphate/Mo(V) complex under acidic conditions [28]. 300 µL of crude extract was dissolved in methanol, followed by the addition of 3 mL of reagent solution consisting of 0.6 mol/L sulfuric acid, 28 mmol/L sodium phosphate and 4 mmol/L ammonium molybdate. The mixture was incubated at 70˚C for 90 min and then allowed to cool to room temperature. The absorbance was measured at 695 nm using a Thermo Electron Corporation Biomate 5 spectrophotometer. Total antioxidant activity was expressed as ascorbic acid equivalents.

2.5. Preparation of Activated Carbons

Palm nut shells collected in the city of Franceville, in the Haut-Ogooué region of south-eastern Gabon, were used as the raw material for the preparation of activated carbons (ACs) using aqueous extracts of *Hallea ciliata, Ricinodendron heudelotii* and *Nauclea latifolia* as green activating agents. The palm nut shells were cleaned, ground, washed with distilled water and then oven dried at 110˚C for 24

h. Subsequently, 80 g of the pretreated shells were impregnated with the aqueous extracts (1:1 volume/mass ratio) for 24 h and dried again in an oven at 110˚C for 24 h. After drying, the impregnated shells were carbonized under air at 400˚C for 1 h 30 min with a heating rate of 5˚C/min. Prepared activated carbons (ACs) were cooled to room temperature, washed with distilled water, dried at 110˚C for 48 h and sieved to obtain powder with a particle size (x) of 0.04 mm < x (sieves: TAM-ISAR, standard: AFNOR NF X11-501). The activated carbons prepared were designated AC-$_{\text{water}}$, AC-$_{\text{HC}}$, AC-$_{\text{HR}}$ and AC-$_{\text{NL}}$, corresponding respectively to activated carbon impregnated with distilled water (reference) and with aqueous extracts of *Hallea ciliata, Ricinodendron heudelotii* and *Nauclea latifolia*.

2.6. Characterization of Activated Carbons

2.6.1. Determination of Methylene Blue and Iodine Numbers

The methylene blue number (I_{BM}) was determined using the method of the European Centre of Chemical Industry Federations (Cefic, 1986) [29] and the iodine number (I_{I_2}) was determined according to ASTM D4607-94 method [30]. These two (2) methods have been revisited and described in recent works of Mougnala *et al.* [5]. Therefore, the I_{BM} is given by the relation in Equation (3):

$$I_{BM} = \frac{(C_0 - C_r)V}{m_{CA}} \tag{3}$$

where I_{BM} is the methylene blue number (mg/g), C_0 is the initial concentration of BM (mol/L), C_r is the residual concentration of methylene blue (mol/L), V is the volume of BM solution (L) and m_{CA} is the mass of activated carbon (g).

The iodine number is given by Equation (4):

$$I_{\mathrm{I}_2}\left(\mathrm{mg/g}\right) = \frac{A - \left(DF \times B \times V_{\text{thiosulfate}}\right)}{m_{CA}} \tag{4}$$

where I_{I_2} is the iodine number (mg/g), A is the concentration of the solution I_2 (mol/L) × molar mass of I_2 (126.93 g/mol), B is the concentration of $\mathrm{Na_2S_2O_3}$ (mol/L) × molar mass of I_2 (126.93 g/mol), $\mathrm{DF} = \frac{V_{\mathrm{I}_2} + V_{\mathrm{HCl}}}{V_{\text{filtrat}}}$ (dilution factor) and m_{CA} is the mass of powdered activated carbon charcoal used (g).

The residual concentration is determined (C_r) by the following relation (5):

$$C_r\left(mg/L\right) = \frac{C_{\mathrm{Na_2S_2O_3}} \times V_{eq}}{V_{\text{filtrat}}} \tag{5}$$

where C_r is the residual concentration of the solution I_2 (N), $C_{\mathrm{Na_2S_2O_3}}$ is the concentration of $\mathrm{Na_2S_2O_3}$ (N), V_{eq} is the volume of $\mathrm{Na_2S_2O_3}$ to the equivalence (mL) and V_{filtrat} is the titrated diode volume (mL).

The curve's path $I_{\mathrm{I}_2} = f\left(C_{\text{residuelle}}\right)$ allows us to determine the iodine index, the value of which Q_{I_2} (mg/g) corresponds to the residual concentration 0.04 N.

2.6.2. Theoretical Studies of Specific Surface Areas (S_{BET}) and Pore Volumes

The theoretical study of specific surface areas and pore volumes of prepared activated carbons (ACs) was carried out with the aim of estimating the specific surface areas and of modeling of the microporous using the iodine and methylene blue number values. The method used was developed by Nunes *et al.* [31]. It has been revisited and described in recent works of Mougnala *et al.* [5]. The theoretical calculation of these physical parameters is defined by the equations below.

$$S\left(\mathrm{m}^2/\mathrm{g}\right)=2.28\times10^2-1.01\times10^{-1}\times I_{BM}+3\times10^{-1}\times I_{\mathrm{I}_2}+1.05\times10^{-4}\times I_{BM}^2 +2\times10^{-4}\times I_{\mathrm{I}_2}^2+9.38\times10^{-4}\times I_{BM}\times I_{\mathrm{I}_2} \tag{6}$$

where, S is the calculated specific surface area (m^2/g), I_{BM} and I_{I_2} are respectively the methylene blue and the iodine number (mg/g).

The theoretical modeling of the microporous volume of prepared activated carbons was described in Equation (7):

$$V_m=5.60\times10^{-2}-1.00\times10^{-3}\times I_{BM}+1.55\times10^{-4}\times I_{\mathrm{I}_2}+7.00\times10^{-6}\times I_{BM}^2 +1.00\times10^{-7}\times I_{\mathrm{I}_2}^2+1.18\times10^{-7}\times I_{BM}\times I_{\mathrm{I}_2} \tag{7}$$

where, V_m is the microporous volume used (cm^3/g), I_{BM} and I_{I_2} are respectively the methylene blue and the iodine number (mg/g).

The theoretical study of the total pore volume was also described in Equation (8) (correspond à la concentration résiduelle):

$$V_t\left(\mathrm{cm}^3/\mathrm{g}\right)=1.37\times10^{-1}-1.90\times10^{-3}\times I_{BM}+1.00\times10^{-4}\times I_{I_2} \tag{8}$$

where, V_t is the total volume of the porous material calculated (cm^3/g), I_{BM} and I_{I_2} are respectively the methylene blue and the iodine number (mg/g).

2.6.3. Surface Functional Groups and of pH at Point of Zero Charge ($\mathrm{pH}_{\mathrm{pzc}}$)

The quantification of surface functional groups was carried out according to the Boehm titration method [32] and the pH at point of zero charge ($\mathrm{pH}_{\mathrm{pzc}}$) was based on the acid-base titration method described by Amola *et al.* [33]. These methods of quantification of surface functional groups and of determination of $\mathrm{pH}_{\mathrm{pzc}}$ have been revisited and described in works of Mve Mfoumou *et al.* [3] [5] [34]-[36].

3. Results and Discussion

3.1. Characterizations of Aqueous Extracts

The properties of extracts were assessed through phytochemical screening, determination of secondary metabolite contents and evaluation of antioxidant activity indices (AAIs) for each plant. Phytochemical screening was conducted to identify the major chemical groups present in the aqueous extracts of the studied plants. The screening results (**Table 1**) indicate that all investigated plants contain gallic tannins, alkaloids, flavonoids, polyphenols, quinones, saponins and simple sugars, with higher abundances observed in *Hallea ciliata*. These results are consistent with those reported by Raphaël *et al.* [37] in their phytochemical and antioxidant activity study of *Mitragyna ciliata* (*Hallea ciliata*), a medicinal plant from Gabon and are also in agreement with the work of Ohoueu *et al.* [38] on the phytochemical characterization and toxicity assessment of the hydroethanolic stem bark extract of *Nauclea latifolia*.

Table 1. Results of phytochemical screening of aqueous extracts of *Hallea ciliata* (HC), *Nauclea latifolia* (NL) and *Ricinodendron heudelotii* (RH).

Chemical groups	HC	NL	RH
Polyphenols	+++	+++	+++
Gallic tannins	+++	++	+++
Alkaloids	+++	++	++
Flavonoids	+	+	++
Quinones	+++	−	++
Saponins	+++	+	−
Simple sugars	+++	+	+++

+++ = very abundant; ++ = abundant; + = not abundant and − = not detected. HC = *Hallea ciliata*; NL = *Nauclea latifolia*; RH = *Ricinodendron heudelotii*.

Table 2 summarizes the results obtained from the quantification of secondary metabolite contents (organic compounds) in aqueous extracts. These results corroborate the phytochemical screening data and indicate that the aqueous extract of *Hallea ciliata* (HC) exhibits higher contents of polyphenols (2418.49 µg GAE/g d.m), flavonoids (190.14 µg QE/g d.m), total tannins (1175.73 µg TAE/g d.m) and total proanthocyanidins (2069.24 µg PPE/g d.m) compared to the aqueous extracts of RH and NL (**Table 2**). Furthermore, the aqueous extract of HC shows a markedly higher phenolic content (2418.49 µg GAE/g d.m) than those of RH (997.03 µg GAE/g d.m) and NL (872.29 µg GAE/g d.m). Comparable results were reported by Raphaël *et al.* [37] in their phytochemical and antioxidant activity study of *Mitragyna ciliata* (*Hallea ciliata*), where the aqueous extract was found to contain a substantial amount of polyphenols. However, in their study, the hydroethanolic extract exhibited the highest polyphenol content at 3.557 mg GAE/g dry matter.

Table 2. Results of organic compounds (secondary metabolites) content of aqueous extracts of *Hallea ciliata* (HC), *Nauclea latifolia* (NL) and *Ricinodendron heudelotii* (RH).

Organic compounds	Contents		
	HC	RH	NL
Polyphenols (µg EAG/g d.m)	2418.49 ± 80.21	997.03 ± 51.34	872.29 ± 70.27
Tannins (µg EAT/g of d.m)	1175.73 ± 65.16	405.39 ± 73.37	177.74 ± 68.99
Flavonoids (µg EQ/g d.m)	190.14 ± 0.59	169.1 ± 6.70	134.11 ± 10.09
Proanthocyanidins (µg EPP/g d.m)	2069.24 ± 0.87	1657.48 ± 21.57	1368.41 ± 14.97

d.m = dry matter; HC = *Hallea ciliata*; NL = *Nauclea latifolia*; RH = *Ricinodendron heudelotii*.

Based on phytochemical screening results, the aqueous extracts of studied plants (*Hallea ciliata*, *Nauclea latifolia* and *Ricinodendron heudelotii*) contain diverse classes of organic compounds (secondary metabolites). Consequently, various chemical reactions may occur between the functional groups of these chemical species and those present on the surface of palm nut shells during the impregnation and activation stages of activated carbon production. Indeed, the bioactivation approach developed in this work relies on the use of plant aqueous extracts as chemically active systems capable of simultaneously modulating surface chemistry and pore structure development in activated carbons derived from palm nut shells. Unlike conventional chemical activating agents (KOH, H_2SO_4, $ZnCl_2$, …), plant aqueous extracts cannot be regarded as single reagents but rather as complex reaction media, whose effects arise from the combined chemical actions of multiple phytochemical families (organic compounds/secondary metabolites) acting as bioactivating agents.

Table 3 summarizes the results obtained from the determination of the antioxidant activities of aqueous extracts of *Hallea ciliata* (HC), *Ricinodendron heudelotii* (RH), and *Nauclea latifolia* (NL). The results indicate that all extracts exhibit lower antioxidant activity than vitamin C, used as the reference standard. The antioxidant activity index (AAI) of the HC extract (AAI = 3.47) is higher than those of the aqueous extracts of RH and NL (AAI = 1.90 and 1.72 respectively), with corresponding IC_{50} values of 14.39, 26.30 and 28.97 µg/mL for HC, RH and NL respectively. These values remain lower than those obtained for vitamin C (AAI = 7.90 and IC_{50} = 6.42 µg/mL). Moreover, the results of phosphomolybdenum test are consistent with those obtained from the DPPH radical scavenging test (**Table 3**). Specifically, the AAI of the HC extract (3.52 mg VtCE/g) is higher than those of RH and NL, which exhibit values of 1.83 and 1.67 mg VtCE/g respectively. According to the classification proposed by Scherer and Godoy [25], plant extracts exhibit low antioxidant activity when AAI < 0.5, moderate activity when 0.5 < AAI < 1, strong activity when 1 < AAI < 2 and very strong antioxidant activity when AAI > 2 [20] [25]. Accordingly, all aqueous extracts investigated in this study display strong to very strong antioxidant activity, with the HC extract showing the highest activity (**Table 3**).

Table 3. Results of the antioxidant activity of aqueous extracts of plants *Hallea ciliata* (HC), *Nauclea latifolia* (NL), *Ricinodendron heudelotii* (RH) and vitamin C used as a reference standard.

Aqueous extracts	Antioxidant activity (DPPH)		Antioxidant capacities
	IC_{50} (µg/mL)	AAI	Dry extract VtCE (mg)/g
Vitamin C (reference)	6.42 ± 0.45	7.90 ± 0.678	-
HC	14.39 ± 0.109	3.47 ± 0.012	3.52 ± 0.0429
HR	26.30 ± 0.241	1.90 ± 0.041	1.83 ± 0.066
NL	28.97 ± 0.514	1.72 ± 0.043	1.67 ± 0.0445

Based on characterization results of aqueous extracts of plants from *Hallea ciliata* (HC), *Ricinodendron heudelotii* (RH) and *Nauclea latifolia* (NL), phytochemical analyses revealed the predominant presence of phenolic compounds, tannins, flavonoids and organic acids, particularly in the extract of *Hallea ciliata* (HC). These chemical species are expected to play a key role in the earliest stages of thermal treatment. Organic acids and phenolic functional groups, which exhibit weakly acidic behavior [39], can promote dehydration and condensation reactions within the lignocellulosic matrix [39], leading to a more structured carbonization process and limiting the formation of volatile tar products [40].

Palm nut shells are lignocellulosic biomasses mainly composed of cellulose, hemicellulose and lignin (**Figure 1**) [41]. During the impregnation step, the organic compounds or bioactivating agents (phenolic compounds, tannins, flavonoids and organic acids) present in the plant extracts penetrate the lignocellulosic matrix of the palm nut shells and interact with the hydroxyl groups of cellulose and lignin through hydrogen bonding and weak intermolecular interactions, which can be schematically expressed as follows:

Biomass-OH + biomolecules (chemical compounds) → Biomass-O/(chemical elements)/H-O-R.

These interactions may promote a homogeneous distribution of activating species throughout the palm nut shells (**Figure 1**). Furthermore, under inert atmosphere (or in air) and at temperatures ranging from 400˚C to 800˚C, the organic compounds contained in the plant extracts undergo thermal decomposition, producing gaseous species and carbonaceous residues according to two main reaction pathways [40]:

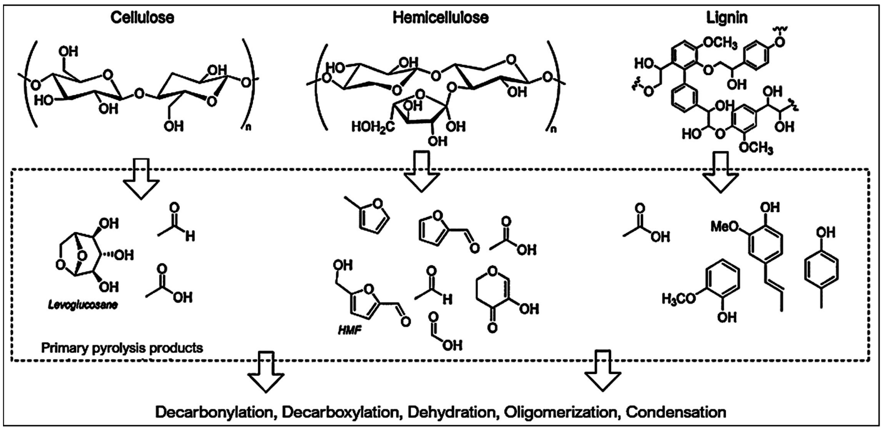

Figure 1. Main primary products of the degradation of the three components of lignocellulosic biomass during pyrolysis [40].

Decomposition of organic acids:

$$R-COOH \xrightarrow{\Delta} CO_2 + CO + H_2O$$

Thermal degradation of sugars and polyphenols:

$$C_6H_{12}O_6 \xrightarrow{\Delta} C(s) + CO + CO_2 + H_2O$$

The released gases may generate internal stresses responsible for the initial formation of micropores [9]. In addition, the gases produced during pyrolysis can react with the solid carbon derived from palm nut shells, leading to reactions [42], such as:

- Mild gasification (Boudouard reaction):

$$\mathrm{C(s)+CO_2 \rightarrow 2CO}$$

- Vapor-carbon:

$$\mathrm{C(s)+H_2O \rightarrow CO+H_2}$$

These reactions can induce the widening of micropores and/or the formation of mesopores, thereby increasing the specific surface area and pore volume of the activated carbon [9]. Simultaneously, the biomass (palm nut shells) undergoes overall carbonization involving dehydration and depolymerization reactions, which enhance structural rigidity while generating volatile tar products [43]. This transformation can be summarized by the following equation:

$$\text{Biomass} \longrightarrow \text{Carbon} + \text{Tars} + \text{Gas}$$

Carbon residues from plant extracts, mainly aromatic compounds, may act as a secondary carbon source:

$$\text{Aromatic compounds} \rightarrow \mathrm{C(s)} + \text{Volatile products}$$

This dual carbon contribution is expected to improve the structural stability of the resulting activated carbon. Furthermore, oxygen-containing residues from the plant extracts may promote the introduction of surface functional groups on the activated carbon [44]:

$$\mathrm{C(s)+O_2/H_2O \rightarrow C{-}OH, C{=}O, COOH}$$

These surface functional groups (**Figure 1**), including carboxylic, phenolic and lactonic groups, enhance the hydrophilicity of prepared activated carbons and strengthen their affinity for the adsorption of organic pollutants and metal ions [40] [45].

The study of surface chemistry of prepared activated carbons could help to support these hypotheses.

3.2. Characterization of Activated Carbons

The physical and chemical properties of activated carbons prepared by water impregnation ($\mathrm{AC\text{-}_{water}}$), used as the reference material and by impregnation with plant aqueous extracts ($\mathrm{AC\text{-}_{HC}}$, $\mathrm{AC\text{-}_{RH}}$, and $\mathrm{AC\text{-}_{NL}}$) are summarized in **Table 4**. The results show that the use of plant aqueous extracts significantly modifies the porous texture and adsorption potential of green activated materials. The reference material ($\mathrm{AC\text{-}_{water}}$) exhibits a specific surface area ($S_{\mathrm{BET\ cal}}$) of 367.40 m^2/g and a micropore volume ($V_{m\ \mathrm{cal}}$) of 0.104 cm^3/g, indicating a moderately developed porous structure. In all cases, impregnation with aqueous extracts leads to an increase in specific surface area and microporosity, highlighting the activating effect of organic compounds (bioactivators) derived from plant extracts (**Table 4**).

Table 4. Physical, adsorptive properties and surface chemistry of prepared activated carbons.

Adsorbents	$S_{\text{BET cal}}$ (m^2/g)	$V_{m\ \text{cal}}$ (cm^3/g)	$V_{t\ \text{cal}}$ (cm^3/g)	I_{BM} (mg/g)	I_{I_2} (mg/g)	pH$_{\text{pzc}}$	Basic functions (mmol/g)	Acid functions (mmol/g)			
								Total	**Carboxylic**	**Lactone**	**Phenolic**
AC-$_{\text{water}}$	367.40	0.104	0.216	22.93	360	7.10	0.90	0.85	0.60	0.20	0.05
AC-$_{\text{HC}}$	446.51	0.144	0.230	21.69	520	6.80	1.05	1.15	1.10	0.03	0.03
AC-$_{\text{RH}}$	404.68	0.126	0.217	19.02	440	6.10	1.10	1.55	0.70	0.35	0.50
AC-$_{\text{NL}}$	384.69	0.118	0.209	17.01	400	6.50	1.05	1.90	1.25	0.03	0.63

AC-$_{\text{water}}$: Activated carbon impregnated with water (reference). AC-$_{\text{HC}}$: Activated carbon impregnated with aqueous extract of *Hallea ciliata*. AC-$_{\text{RH}}$: Activated carbon impregnated with aqueous extract of *Ricinodendron heudelotii*. AC-$_{\text{NL}}$: Activated carbon impregnated with aqueous extract of *Nauclea latifolia*. I_{BM}: Methylene blue number (mg/g). I_{I_2} : Iodine number (mg/g). pH$_{\text{pzc}}$: pH at the point of zero charge. $S_{\text{BET cal}}$: Specific surface area (m^2/g) calculated by the method of Nunes *et al.* [31]. $V_{m\ \text{cal}}$: Microporous volume (cm^3/g) calculated by the method of Nunes *et al.* [31]. $V_{t\ \text{cal}}$: Total pore volume (cm^3/g) calculated by the method of Nunes *et al.* [31].

The activated carbon AC-$_{\text{HC}}$, impregnated with the aqueous extract of *Hallea ciliata* (HC), exhibits the highest specific surface area (446.51 m^2/g), corresponding to an increase of approximately 22% compared to the reference AC-$_{\text{water}}$. This enhancement is accompanied by a marked increase in micropore volume (0.144 cm^3/g), indicating preferential formation of micropores during activation. AC-$_{\text{RH}}$ and AC-$_{\text{NL}}$ carbons also show higher specific surface areas (404.68 and 384.69 m^2/g respectively) than the reference material. In contrast, the total pore volume remains relatively stable (**Table 4**), ranging from 0.209 to 0.230 cm^3/g. This suggests that the action of aqueous extracts does not induce a massive increase in overall porosity, but rather a redistribution of pore size toward the microporous domain.

This observation is corroborated by the evolution of the iodine number [30]. Indeed, the iodine number increases significantly after impregnation with aqueous extracts, rising from 360 mg/g for AC-$_{\text{water}}$ to 520 mg/g for AC-$_{\text{HC}}$. The AC-$_{\text{RH}}$ and AC-$_{\text{NL}}$ carbons also display higher iodine numbers (440 and 400 mg/g respectively) than the reference material. This confirms the enrichment in microporosity, which is particularly favourable for the adsorption of small molecular weight pollutants [30]. Conversely, the methylene blue number slightly decreases after impregnation, from 22.93 mg/g for AC-$_{\text{water}}$ to values ranging between 17.01 and 21.69 mg/g for bioactivated carbons. This decrease suggests a relative reduction in the mesoporous fraction accessible to large molecules [29], as a direct consequence of the increased dominance of microporosity. According to the literature, typical iodine number for activated carbons ranges between 500 and 1200 mg/g [46]. In the present study, only AC-$_{\text{HC}}$ meets this characteristic (**Table 4**). The specific surface areas and pore volumes obtained in this work are comparable to those reported by Ubago-Pérez *et al.* [47] for granular and monolithic activated carbons prepared from olive stones using KOH as chemical activating agent, as well as to the results of Mougnala *et al.* [5] concerning the removal, kinetics, and thermodynamics of Fe(II) adsorption onto activated carbons in aqueous media.

Overall, AC-$_{\text{HC}}$ clearly stands out due to its highest specific surface area, micropore volume and iodine number. The high surface area and microporosity of AC-$_{\text{HC}}$ make it a particularly suitable material for the adsorption of organic and inorganic pollutants in aqueous media. These results further indicate that impregnation with plant aqueous extracts, such as *Hallea ciliata* (HC), which is rich in organic compounds (**Table 2**) and/or exhibits strong antioxidant activity (**Table 3**), promotes the development of a microporous structure during the preparation of ACs in our experimental conditions.

Similarly, the pH at point of zero charge (pH_{pzc}) and the surface chemistry of the activated carbons obtained after impregnation with plant aqueous extracts (AC-$_{\text{HC}}$, AC-$_{\text{RH}}$ and AC-$_{\text{NL}}$) are significantly modified compared to the reference carbon AC-$_{\text{water}}$ (**Table 4**). AC-$_{\text{water}}$ exhibits a pH_{pzc} of 7.10, which is associated with relatively low total acidity (0.85 mmol/g) and a moderate predominance of carboxylic functions (0.60 mmol/g), while phenolic groups remain very limited (0.05 mmol/g), as shown in **Table 4**. This surface composition is characteristic of a slightly basic surface and is consistent with the measured pH_{pzc} value (7.10). In contrast, a systematic decrease in pH_{pzc} is observed for activated carbons prepared using plant extracts (**Figure 2**). The pH_{pzc} values decrease to 6.80 for AC-$_{\text{HC}}$, 6.50 for AC-$_{\text{NL}}$ and 6.10 for AC-$_{\text{RH}}$ (**Figure 2**). This trend is directly correlated with a marked increase in acidic surface functionalities (**Table 4**). Total acidity increases to 1.15 mmol/g for AC-$_{\text{HC}}$, 1.90 mmol/g for AC-$_{\text{NL}}$ and 1.55 mmol/g for AC-$_{\text{RH}}$, confirming the enrichment of the carbon surface with acidic sites following impregnation. Although a slight increase in basic functionalities is also observed (from 0.90 mmol/g for AC-$_{\text{water}}$ to approximately 1.05 - 1.10 mmol/g for bioactivated carbons), this effect remains secondary compared to the more pronounced increase in acidic surface groups.

A detailed analysis of the nature of acidic surface functionalities reveals differentiated behaviours among the prepared activated carbons. The AC-$_{\text{HC}}$ adsorbent is dominated by a high content of carboxylic groups (1.10 mmol/g), while exhibiting very low amounts of lactonic (0.03 mmol/g) and phenolic (0.03 mmol/g) functions, which accounts for the moderate decrease in pH_{pzc}. In contrast, AC-$_{\text{NL}}$ combines a high concentration of carboxylic groups (1.25 mmol/g) with a significant proportion of phenolic functionalities (0.63 mmol/g), leading to the highest total acidity and a lower pH_{pzc} value (**Table 4**). The AC-$_{\text{RH}}$ adsorbent is characterized by a more balanced distribution of carboxylic (0.70 mmol/g), lactonic (0.35 mmol/g) and phenolic (0.50 mmol/g) groups, a configuration associated with the lowest pH_{pzc} observed (6.10). These pH_{pzc} values are similar to those reported in studies by Mve Mfoumou *et al.* [3] [35] [36] on activated carbons prepared from palm nut shells via chemical activation using $ZnCl_2$ as an acidic activating agent and are comparable to results of Langama *et al.* [48] on the preparation and characterization of activated carbons from asparagus palm shells using KOH as a basic activator.

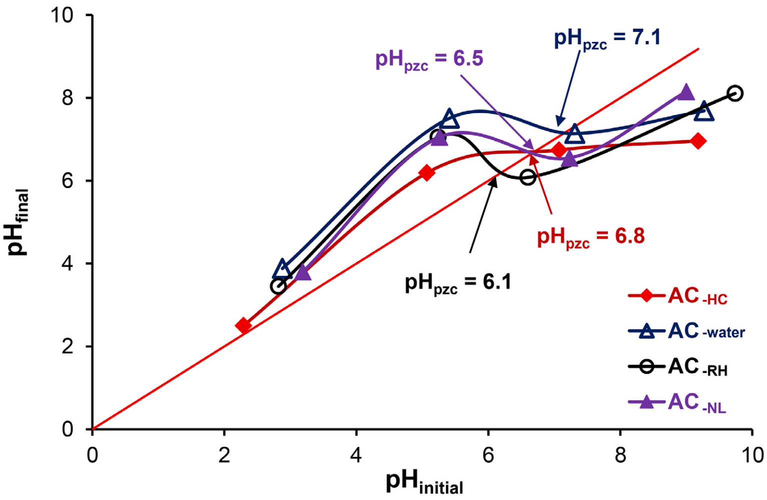

Figure 2. Determination of pH at point of zero charge (pHpzc) of prepared activated carbons.

The observed differences in surface chemistry among the prepared ACs can be attributed to the composition of aqueous extracts used for impregnation. The HC extract is particularly rich in polyphenols (2418.49 µg GAE/g d.m), tannins (1175.73 µg TAE/g d.m) and proanthocyanidins (2069.24 µg PPE/g d.m), yet it predominantly leads to the formation of carboxylic functionalities on the carbon surface, suggesting either preferential formation pathways or limited accessibility of phenolic groups after carbonization. In contrast, the RH and NL extracts, although exhibiting lower overall polyphenol contents (997.03 and 872.29 µg GAE/g d.m respectively), promote the formation of phenolic and lactonic surface groups on activated carbons, as evidenced by their higher measured contents (Table 4). These results demonstrate that the decrease in pH$_{pzc}$ does not solely depend on the overall richness of extracts in phenolic compounds, but rather on their ability to generate stable and accessible acidic functionalities on the material surface.

Consequently, impregnation with plant aqueous extracts for the preparation of AC appears as a mild and selective approach for tuning the surface chemistry of porous materials, allowing fine adjustment of the surface charge of activated carbons. Since the pH$_{pzc}$ determines the net surface charge of an adsorbent [33], such control is critical for optimizing the adsorption of cationic pollutants and polar organic compounds. This result reinforces the potential of functionalized carbons, after impregnation with plant aqueous extracts, for water depollution applications, particularly in rural environments (rivers, wells, etc.), where sustainable and low-cost treatment solutions are required.

4. Conclusions

The objective of this study was to develop a method for preparing activated carbons (ACs) from palm nut shells through green activation using secondary metabolites (plant biomolecules) derived from aqueous extracts of Gabonese plants

(*Hallea ciliata* (HC), *Ricinodendron heudelotii* (RH), and *Nauclea latifolia* (NL)), with the aim of proposing a simple, environmentally friendly and accessible method for producing ACs suitable for rural communities. More specifically, this work sought to evaluate the effect of plant-derived secondary metabolites on the porous structure and surface chemistry of prepared bioactivated adsorbents.

The characterizations of aqueous extracts of HC, RH and NL revealed high contents of secondary metabolites or bioactivators (organic compounds), associated with strong antioxidant activities. Among investigated extracts, *Hallea ciliata* exhibited the highest antioxidant activity (AAI = 3.47) and the highest contents of polyphenols (2418.49 µg GAE/g d.m), tannins (1175.73 µg TAE/g d.m) and proanthocyanidins (2069.24 µg PPE/g d.m) compared to aqueous extracts of NL and RH. These results indicate a strong potential for these organic compounds to interact with the carbonaceous matrix of the biomass during impregnation and carbonization processes.

The characterization of ACs obtained after impregnation with aqueous extracts of HC, RH and NL (AC-$_{\text{HC}}$, AC-$_{\text{RH}}$ and AC-$_{\text{NL}}$) showed a clear improvement in textural properties compared to the reference material impregnated with water (AC-$_{\text{water}}$). The specific surface areas increased from 367 m^2/g for AC-$_{\text{water}}$ to values ranging between 385 and 447 m^2/g for bioactivated carbons, with a preferential development of the microporosity. Compared to other prepared activated carbons, AC-$_{\text{HC}}$ exhibited the highest specific surface area (446.51 m^2/g), iodine number (520 mg/g) and micropore volume (0.144 cm^3/g), exceeding those of AC-$_{\text{RH}}$, AC-$_{\text{NL}}$ and the reference AC-$_{\text{water}}$. These properties make AC-$_{\text{HC}}$ particularly suitable for the adsorption of organic and inorganic pollutants in aqueous media.

Overall, the results demonstrate that biomolecules (secondary metabolites) derived from plant aqueous extracts can act as effective green activating agents, capable of simultaneously influencing the porous structure and surface chemistry of activated carbons. This green approach represents a credible alternative to conventional chemical activation methods and offers strong prospects for the preparation of efficient adsorbent materials within a sustainable and environmentally respectful, specifically targeted toward water depollution (rivers, wells) in rural zones.

However, since adsorption is a surface phenomenon, optimization of the preparation method is necessary to obtain larger specific surfaces and more efficient activated carbons.

Declaration

The authors do not work for, advise, own shares in, or receive funds from any organization that might benefit from this article and have declared no affiliation other than their research organizations.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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