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**Use of activated carbon from vegetable residual biomass with SiO2 Nanoparticles for the removal of organic compounds in wastewater from the hydrocarbon sector**

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Colombia has significant potential for the cultivation of cocoa, coffee, and oil palm. However, activities also generate substantial amounts of vegetable residual biomass. For this reason, various industrial sectors have shown interest in utilizing vegetable residual biomass to promote environmental sustainability. In this research, activated carbons were prepared from vegetable residual biomass combined with SiO2 nanoparticles for the removal of representative organic compounds in founds in produced water from the hydrocarbons industry. The organic compounds selected as model species for the development of this research were methylene blue (MB), phenol (Ph), and sodium dodecylbenzenesulfonate (SDBS). The activated carbons from residual vegetable biomass of cocoa, coffee, and oil palm kernel were prepared in a tubular furnace using the thermochemical process with a phosphoric acid-to-biomass impregnation ratio of 1:1, under reaction conditions of 430 °C for 30 minutes. The impregnation treatment of the activated carbons was performed using the coprecipitation method at the laboratory scale, employing ethanol solutions with 5,400 mg/L of SiO2 nanoparticles. From the Scanning Electron Microscopy / Energy Dispersive Spectroscopy (SEM-EDS) analysis, it was determined that the new adsorbent materials contain channels that facilitate the impregnation of SiO2 nanoparticles.

Textural tests showed that the prepared carbons exhibited surface areas in the range of 600 to 1200 m²/g. The removals of methylene blue (MB), phenol (Ph), and sodium dodecyl benzene sulfonate (SDBS) were 93.85%, 97.09% and 98.15% by weight, respectively. However, the addition of SiO2 nanoparticles increased both the surface area and the percentage of removal of the organic compounds. The adsorption results were fitted to adsorption isotherms, and according to the correlation coefficient R², the isotherms were classified in ascending order with the Freundlich>Langmuir>Temkin models. The methodology proposed in this study demonstrates that the activated carbon/SiO2 nanoparticles are suitable for removing organic compounds present in wastewater.

* 1. Introduction

Wastewater from the oil and gas industry is utilized for various reasons: underground injection, discharge, farming, and distribution to third parties (Marathe et al., 2021). According to the ANH's audited production reports for 2023, Colombia produced an average of 777.000 barrels per day (BPD), with a water/oil ratio of 14 barrels of water for each barrel of crude oil, estimating more than 10.878.000 BPD or 1.729.463 m3 per day, of which Ecopetrol produced 495.967.000 m3 per day according to its integrated management report (Agencia Nacional de Hidrocarburos, 2024; Ecopetrol, 2024). The water quality is influenced by the area, rock type, and other liquids involved in the recovery process and is impacted by the presence of suspended particles, sediments, heavy metals, phenols, hydrocarbons, and dissolved oils, among others (Belmont et al., 2017).

Colombia has a lot of heavy and extra heavy crude oil, so it is common to implement enhanced recovery methods, among the various possibilities is the use of surfactants. This is a possible compound to be eliminated during the treatment of production waters (Delgadillo et al., 2020). Surface treatment for produced water involves three major phases. In the primary phase, the goal is the removal of oil droplets and suspended solid particles, these contaminants are removed using large tanks by density difference or gravity action, in the secondary phase gas flotation processes are used, in which gas bubbles help to separate the crude oil from the treated fluid. The tertiary phase includes advanced oxidation and membrane technologies, which can be expensive depending on the required purity of the fluid. An eco-friendly and cost-effective option is the use of adsorbent materials, which are chosen for their accessibility and appealing cost/benefit ratio (Villegas et al., 2017).

Activated carbon is as an adsorbent material, that removes organic compounds, heavy metals, and free chlorine in water treatment (Castro-Suarez et al., 2024; Gul-Zaman et al., 2021). By using residual biomass and its characteristics, activated carbons can be produced through chemical and/or physical activation processes, and based on the process conditions, it is possible to achieve well-formed pore structures, a large surface with structured macro, meso, and microporous textural characteristics with active functional groups. Similarly, nanoparticles are used as a better approach in the water treatment process because of their bigger surface area, shorter distance between particles, and more reaction sites compared to traditional methods, which are often restricted by the number of active sites or inability to selectively remove contaminants (Kumar et al., 2023). Metal oxide nanoparticles demonstrate potential as adsorbents for the sequestration of organic and inorganic impurities due to attributes such as high removal efficiency, diverse magnetic properties, and economic viability. However, their high surface energy leads to inherent instability and a propensity for agglomeration due to Van der Waals forces, which can compromise adsorption performance (Tiwari et al., 2023). To mitigate these limitations and facilitate practical application, these nanomaterials are often structurally modified through surface functionalization with surfactants or by incorporating them into porous support matrices.

This research shows the adsorption behavior of activated carbon from residual vegetable biomass from cocoa (cocoa shell), coffee and palm oil, as well as the subsequent impregnation with silica nanoparticles (SiO2) for the removal of contaminants detected in wastewater for the biomass that showed the best development of porosity measured through the surface area. The analysis was performed by Fourier transform infrared spectroscopy (FTIR-ATR), scanning electron microscopy (SEM), specific surface area (BET) and UV-Vis spectroscopy, where the removals of methylene blue (MB), phenol (Ph) and sodium dodecylbenzenesulfonate (SDBS) were 93.85%, 97.09% and 98.15% by weight, respectively. The methodology proposed in this study demonstrates that activated carbon/SiO2 nanoparticles are suitable for take away organic compounds present in wastewater.

* 1. Materials and Methods
     1. Carbon Activation

Plant biomasses were selected as precursors from the Santander region in Colombia. The residual biomasses were initially ground to obtain particles between 100 and 120 mesh. The crushed material was sun-dried for 3 days to reduce moisture, followed by oven drying at 105 °C for 8 h, repeated until a constant weight was achieved. The activation process was performed using chemical activation with 85% H3PO4 at an impregnation ratio of 1.5. For this, 300 g of dry biomass was mixed with H3PO4 under constant stirring at 100 °C for 1 h. The activated biomass was subjected to a thermochemical process in a nitrogen (N2) atmosphere at a flow rate of 20 mL/min at 480 °C for 30 min. The resulting activated carbon was washed until the pH of the washing water reached 6–7 and finally dried in an oven at 100–105 °C for 24 h (León et al., 2023).

* + 1. Characterization of Activated Carbon/SiO2

The characterization was performed in two stages. The first stage involved FTIR-ATR spectroscopy to analyze the surface chemistry of the three adsorbent materials and nitrogen (N2) adsorption/desorption analysis to determine textural properties such as surface area. The material with the highest surface area was selected for SiO₂ nanoparticle impregnation. FTIR spectra were acquired using a Thermo Scientific© Nicolet Summit X FTIR in the mid-infrared (MIR) range, coupled with an attenuated total reflectance (ATR) cell featuring a diamond crystal at a fixed 45° incidence angle. Spectra for control and impregnated carbons were normalized using OMNIC® software. The Brunauer, Emmett and Teller (BET) method based on nitrogen (N₂) adsorption/desorption isotherms at 77 K was applied to determine specific surface area and pore volume. Activated carbon samples impregnated with SiO₂ nanoparticles were weighed in 9 mm borosilicate glass cells using a Vac Prep 061 MICROMERITICS® device.

After selecting the activated carbon with the highest surface area, finally, characterization was performed by scanning electron microscopy (SEM). Secondary electron (SE) and backscattered electron (BSED) imaging, along with energy-dispersive spectroscopy (EDS), were conducted using a FEI QUANTA FEG 650 environmental SEM under the following conditions: magnification (200X–3000X), working distance (9.5 mm), high voltage (15.00 kV), and detectors (BSED and Everhart-Thornley ETD). EDS analysis was performed using an EDAX APOLO X detector with 126.1 eV resolution (Mn Kα) (León et al., 2023).

* + 1. Impregnation of Activated Carbon with SiO2 NPs

Activated carbon/SiO2 was synthesized via the coprecipitation method. Suspensions at 5400 mg/L SiO2-NPs were dispersed in ethanol and stabilized using ultrasonic treatment. After stabilization, 4 g of activated carbon was added to the stabilized fluid and sonicated for 1 h. The resulting impregnated carbon was refluxed for 3 h, then washed, filtered, and dried at 110 °C for 3 h.

* + 1. Adsorption of Persistent Organic Pollutants (POPs)

The adsorption capacity of the AC/SiO₂ composite was assessed through equilibrium isotherms using the batch adsorption process. Experiments were performed with methylene blue (MB), phenol (Ph), and sodium dodecylbenzene sulfonate (SDBS) in 25 mL centrifuge tubes. The effect of initial pollutant concentrations (5–350 mg/L) was analyzed while maintaining a constant contact time. Each tube contained 5 mL of dilution solution at 25 ± 1 °C, followed by adding a predetermined amount of AC/SiO₂. The samples were agitated in an isothermal shaker at 150 rpm for 18 hours. Using Lambert-Beer's law, a linear correlation between absorbance and concentration was established through calibration curves to determine the final pollutant concentrations. The adsorption capacity (*qe*) of the activated carbon was then calculated using Equation (1), where *qe* (mg/g) is the adsorption capacity, the Co (mg/L) and Ce (mg/L) is the pollutant concentration at the initial and at equilibrium time, respectively, W (g) is the mass of dried adsorbent and V (L) is the volume of the aqueous phase (Wu et al., 2024).

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|  | (1) |

When an adsorbent interacts with a surrounding fluid containing a specific species, adsorption occurs until equilibrium is reached after a certain contact time, adsorption isotherms describe the relationship curve depicting the solute concentration in two phases when adsorption reaches equilibrium at their interface under a specific temperature (Xing et al., 2022). The experimental data were fitted to Langmuir, Freundlich, and Temkin isotherm models, with their linearized forms presented below:

* *Langmuir Isotherm:* It is denoted by Equation (2), where *Ce* (mg/L) and *qe* (mg/g) are the pollutant concentration and the amount of adsorbed molecules on the surface of the adsorbent at equilibrium time, respectively. Also, *qmax* (mg/g) indicates the maximum adsorption capacity, and *KL* (L/mg) is the Langmuir constant (Kecili & Hussain, 2018).

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|  | (2) |

* *Freundlich Isotherm:* It is described by Equation (3), where *Ce* (mg/L) and *qe* (mg/g) are the pollutant concentration and the amount of adsorbed molecules on the surface of the adsorbent at equilibrium time, respectively. In addition, *KF* (L/g) is adsorption capacity, and 1/*n* is adsorption intensity; it also indicates the energy's relative distribution and the adsorbate sites' heterogeneity (Kecili & Hussain, 2018).

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| --- | --- |
|  | (3) |

* *Temkin Isotherm:* It is represented by Equation (4), where *qe* (mg/g) is the solid-phase concentration, *Ce* (mg/L) is the equilibrium liquid-phase concentration, *R* (J/mol·K) is the gas constant, *T* (K) is the temperature, and *b*T (J/mol) and *K*T (L/g) are adjustable parameters (Chu, 2021)*.*

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|  | (4) |

* 1. Results and Discussion
     1. Characterization of Activated Carbon

Figure 1 shows FTIR-ATR spectra of activated carbons from cocoa, coffee and oil palm kernel, where it could be observed that the band associated with the C=O bond is intense and appears around 1650 cm-1, this band is associated with the presence of carboxylic acids, additionally it is highlighted that around 1567 cm-1 a band of high intensity was observed which is related to the C=C bonds of the benzene ring, according to what is reported in the literature this type of functional groups support the adsorption process by providing more active sites (Gutierréz, 2019). Additionally, in the characteristic region of the fingerprint, the carbons activated with phosphoric acids show high intensity in the bands associated to the region between 1300 and 900 cm-1 related to phosphate groups, presumably coming from the acid used in its activation, likewise it is highlighted that the band around 1100 cm-1 is typical of the asymmetric stretching of the C-O bond.



Figure 1: FTIR spectra of activated carbons from cocoa, coffee and oil palm kernel.

The BET model is based on the Van Der Waals forces of attraction as the only forces responsible for the adsorption process, focusing explicitly on the kinetics involved, under the premise that for each adsorbed layer there is a dynamic equilibrium. Table 1 shows the results obtained for the different biomasses, in all cases, the observed isotherms corresponded to Type II, which as mentioned by the IUPAC are bilayer isotherms, the activated carbon from cocoa stands out for being the one with the largest surface area developed with 971 m2/g, pore volume of 0.6888 cm3/g and pores with an average size of 3.96 nm, showing that it has widely developed microporosity. For this reason, activated carbon from cocoa biomass was selected for impregnation with nanoparticles.

Table 1: BET characterization of activated carbons.

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| --- | --- | --- | --- |
| Activated carbon type | ABET, m2/g | Pore volume, cm3/g | Pore size, nm |
| Cocoa | 971 | 0.6888 | 3.96 |
| Coffee | 695 | 0.4995 | 4.04 |
| Oil palm | 823 | 0.3680 | 5.29 |

* + 1. Effect of nanoparticles on the activated carbon

Table 2 shows an increase in the surface area when using silica nanoparticles, increasing by 23.89%, obtaining as final area 1203 m2/g, pore volume of 0.6888 cm3/g and pores with an average size of 4.50 nm.

Table 2: Effect of nanoparticles in BET characterization of activated carbon.

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| --- | --- | --- | --- |
| Activated carbon type | ABET, m2/g | Pore volume, cm3/g | Pore size, nm |
| Cocoa AC | 971 | 0.6888 | 3.96 |
| Cocoa AC + 5400 mg/L SiO2 | 1203 | 0.8605 | 4.50 |

The structure of activated carbon without nanoparticles and treated with SiO2 nanoparticles at 5400 mg/L was analyzed using scanning electron microscopy (SEM), as shown in Figure 2. According to Figure 2(a), the activated carbon granules display structured surfaces with uneven sizes and disordered channels, which might hinder the adsorption of organic compounds (Ma et al., 2022). However, this irregular shape encourages the attachment of SiO2 nanoparticles, as shown in Figure 2(b), where the appearance of white clusters scattered across the surface of the activated carbon confirms the adhesion of the nanoparticles, contributing to an increased surface area and potentially improving the adsorption efficiency for organic compounds (Buchori et al., 2022).

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| **(a)** | **(b)** |

Figure 2: SEM micrograph from cocoa activated carbon: (a) without NPs and (b) with 5400 mg/L SiO2-NPs.

* + 1. Adsorption of Persistent Organic Pollutants (POPs)

Table 3 shows the adsorption results for each compound, comparing the isothermal models and the removal capacity with and without nanoparticles in parellel. This study shows that the Freundlich isotherm best describes the adsorption process, and the addition of SiO2 further improves adsorption efficiency, especially for SDBS, suggesting potential benefits in modifying activated carbon with silica to enhance adsorption capabilities (León et al., 2023). Finally, Table 4 presents the Freundlich isotherm parameters for the adsorption of POPs, indicating that the presence of OH, C=O, and –CO functional groups, along with the SiO2 content in the activated carbons, greatly affects the adsorption rates of the organic compounds examined. These results highlight the potential of Cocoa AC as an efficient and eco-friendly adsorbent for POPs removal from wastewater.

Table 3: Adsorption of Persistent Organic Pollutants (POPs).

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| --- | --- | --- | --- | --- | --- |
| Persistent Organic Pollutants | Activated carbon type | R2 correlation | | | Adsorption percent, % |
| Langmuir | Freundlich | Temkin |
| Ph | Cocoa AC | 0.9830 | 0.9970 | 0.9770 | 95.12 |
| Cocoa AC + SiO2 | 0.9870 | 0.9920 | 0.9810 | 97.09 |
| MB | Cocoa AC | 0.8996 | 0.9956 | 0.8547 | 92.55 |
| Cocoa AC + SiO2 | 0.9175 | 0.9692 | 0.8907 | 93.85 |
| SDBS | Cocoa AC | 0.9962 | 0.9964 | 0.9858 | 96.76 |
| Cocoa AC + SiO2 | 0.9797 | 0.9875 | 0.9704 | 98.15 |

**Note:** The SIO2 concentration was 5400 mg/L, Phenol (Ph), Methylene Blue (MB), Sodium Dodecylbenzene sulfonate (SDBS).

Table 4: Parameters of Freundlich isotherms in the adsorption

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| --- | --- | --- | --- | --- | --- | --- |
| Activated carbon type | Ph | | MB | | SDBS | |
| *KF* | *n* | *KF* | *n* | *KF* | *n* |
| Cocoa AC | 1.825 | 1.663 | 2.221 | 2.716 | 2.400 | 1.800 |
| Cocoa AC + 5,400 mg/L SiO2 | 2.274 | 1.217 | 1.974 | 3.167 | 1.376 | 1.190 |

Overall, the synthesized activated carbons showed high efficiency in removing organic species in aqueous media. The results are relevant for future projects such as constructing a pilot unit, applying activated carbons under dynamic conditions, and their possible regeneration in water treatment.

* 1. Conclusions

Residual biomass from cocoa, coffee, and oil palm can be repurposed to synthesize activated carbons, offering a sustainable solution to waste accumulation and disposal costs. The resulting activated carbons exhibited high specific surface areas between 695 and 971 m2/g, with cocoa biomass achieving the highest value. Impregnation with SiO₂ nanoparticles increased the surface area by 19%, enhancing pollutant removal efficiency. Adsorption studies for phenol (Ph), methylene blue (MB), and sodium dodecylbenzene sulfonate (SDBS) followed Langmuir, Freundlich, and Temkin isotherms, with the Freundlich model providing the best fit.

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