

Formulation and Characterization of Activated Carbon from Rubber Seed Husk Using Physical and Chemical Methods

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Abstract— Rubber seed husk is currently being discarded as agricultural waste, leading to economic loss. The current work assesses the possibility of converting this waste into activated carbon for potential use as an adsorbent. This was achieved using the physical method at 450°C for 1h and the chemical activation method through a one-step activation process with H₃PO₄ as the activating agent and an impregnation ratio of 1:1. The physicochemical properties of the formulated activated carbon were investigated, and functional groups were assessed using Fourier transform infrared spectroscopy (FTIR). The results of the physicochemical properties of the formulated activated carbon prepared using the physical method were: bulk density (0.4784 g/mL), pH (6.4), moisture content (0.730%), ash content (29.302%), volatile matter (21.66%), iodine number (16.624 mg/g), and surface area (525.4 m²/g). Results of the physicochemical properties of activated carbon prepared using chemical methods were: bulk density (0.4764 g/mL), pH (6.2), moisture content (0.2976%), ash content (29.302%), volatile matter (28.788%), iodine number (18.009 mg/g), and surface area (563.8m²/g). Regardless of method employed, FTIR investigations have revealed presence of alcohols, phenols, aldehydes, aromatics, amines, amides, carboxylic acids, esters, alkenes, and alkanes in manufactured activated carbon. The obtained result shows that high-quality activated carbon can be formulated from rubber seed husks using either physical or chemical methods.

Keywords — Activated carbon; Adsorbent; Chemical activation; Physical activation; Pollution, Rubber seed husk; Wastewater.

I. INTRODUCTION

Recently, the entire world has been impacted by several different environmental issues, including air pollution, water pollution etc. caused by human activity. Due to this, there has been an ongoing rise in demand for activated carbon for water treatment and purification purposes. For instance, water pollution, often known as heavy metal pollution of water bodies, is gaining prominence as a global environmental problem. Once ingested, these metals cause variety of health issues, including liver dysfunction, skin diseases, kidney failure in both adults and children, and conditions connected to cancer. Adverse changes in water quality result in about 50 million deaths worldwide each year, with the majority of these deaths occurring in developing countries in Africa and Asia [1]. Therefore, it is now importance for researchers from around the world to reduce concentrations of these heavy metal pollutants in water bodies to safe levels. A variety of approaches, including adsorption, ion exchange, reverse osmosis, electrolysis, filtration, floatation, precipitation, coagulation, and membrane separation, have been proposed and explored to address this issue of water contamination [2]. Due to its ease of use, effectiveness, low cost, and simplicity, the adsorption strategy has shown to be the most effective of these techniques [3]. Water quality can be improved by variety of treatment methods,

one of which uses AC to remove wastewater pollutants. Therefore, a low-cost adsorption system is in high demand, and efforts are being made to find a low-cost alternative adsorbent that is highly effective. Scientific interest in the use of easily available, high-carbon agricultural wastes as adsorbents for water filtration has lately increased [4]. In this study, rubber seed husk, a type of agricultural-waste, is investigated as a potential precursor in the production of high-quality activated carbon. The objectives of this work are to quantitatively convert rubber seed husk into powdered activated carbon (PAC) and to characterize its surface functional groups using an FTIR analyzer. There is no or little research information on producing powdered activated carbon (PAC) from rubber seed husk or hull using both physical and chemical methods.

II. RELATED WORK

The substance known as activated carbon (AC) is a dark granule or powder that has a high internal surface area, a well-developed pore volume, and a high adsorption capacity [5], [6]. Because of its considerable adsorption capacity, rapid adsorption kinetics, and large surface area, AC possesses exceptional adsorption abilities [7]. Many industrial uses for activated carbon (AC) exist, including gas, wastewater, and water treatment; gold purification;

carbon capture; extracting metals; food processing; medical fields; sewage treatment; air filters in gas masks and respirators; filters in compressed air; chemical and pharmaceutical treatment; and many other applications ascribable to their capacity for adsorptive properties [8]. Generally, raw materials for the production of AC are those with high carbon contents but low inorganic or mineral contents, such as wood, lignite, peat, and coal. Besides, lots of agricultural waste products and byproducts, such as macadamia nutshells, coconut shells, wood, cotton stalks, almond shells, rice husks, and date pits, have successfully been transformed into activated carbon (AC), etc. [9]. Globally, commercial AC use is on the rise. However, there is growing amount of study interest in low-cost agricultural wastes or byproducts due to economic and environmental considerations. Replacements for activated carbon that have relatively high carbon content are available [10], [11]. In this study, a local agricultural waste called rubber seed husk or hull is being explored as a source of activated carbon (AC). Rubber seed hulls are an agricultural by-product of rubber trees (*Hevea brasiliensis*), and according to [12], the economic relevance of rubber plants (*Hevea brasiliensis*) is mostly centered on rubber latex, with little to no consideration given to the potential utility of other rubber plants by-products. Activation is conducted via either physical or chemical method. Physical activation approach involves carbonizing carbon-base materials to remove majority of volatile matter, then activating the char that results using activating agents like carbon (IV) oxide, steam, air, or a combination of these agents [13]. Alternately, chemical activation approach entails soaking or impregnating char materials with H_3PO_4 , $ZnCl_2$, Na_2CO_3 , $NaOH$, KOH , etc. and then exposing them to heat treatment in single step in an inactive environment [14], [15]. The physical activation (PA) method differs from chemical activation method in the sense that it avoids mixing impurities derived from chemical activating agents compared to chemical activation method [16]. Furthermore, AC formulated using chemical activation method usually contains inorganic materials that could create environmental pollution. On that account, the physical activation method is preferred to chemical activation(CA) method in terms of environmental safety concerns, as no chemical is required. However, [17], [18] have stated that chemical activation(CA) technique is preferred to physical activation(PA) considering yield, simplicity, low temperature, and reduced activation time. Besides, [17] have opined that some physicochemical properties of AC affect its appropriateness for specific applications. Therefore, these processes require a proper understanding of the relationship between activated carbon and its properties.

III. METHODOLOGY

Sample preparation

The rubber seed husk was collected from a rubber plantation in Ozoro, Delta State, Nigeria. After washing in distilled water(H_2O) to remove impurities/contaminants.

Thereafter, seed husk samples were dried in an electric oven at $105^\circ C$ temperature for 72 h. After mechanical crushing, the resultant particles were filtered to a particle size of 100 μm . Figure1 shows the husk of dried rubber seeds.



Figure 1: Dry Rubber Seed Husk

Experimental Procedure: Activation of Rubber Seeds Husk

(A). Physical Activation

200g of seed husk sample was measured, then burned in electric muffle-furnace at $450^\circ C$ temperature for 1hr, and transferred into a desiccator for cooling. The cooled sample was pulverized and sieved using a 100 μm sieving apparatus. Sample was further washed in sufficient water(H_2O) and subsequently dried using electric oven. The steam from a pressure cooker was then made to pass through the burnt sample for 30 minutes .

(B). Chemical activation

This was done using a single-step chemical activation (CA) process. Using a 1:1 ratio, 100 g of material was impregnated with H_3PO_4 . Thereafter, the mixture was vortexed for 20 minutes. The mixture was dried in oven set at temperature of $105^\circ C$ for 48 hours to ensure that every last trace of moisture from sample material had been eliminated. It was then chilled for 45 minutes at room temperature. In absent of gases such as carbon dioxide (CO_2), water (H_2O), oxygen (O_2), and nitrogen (N_2), the dried sample material was carbonized in muffle furnace for 1 hour at $45^\circ C$ at a heating rate of $5^\circ C$ per minute. The calcined samples were ground in a mortar once they had cooled to ambient temperature.

Determination of some physical/chemical properties of rubber seed husk AC

Moisture Content

Sample of rubber seed husk-activated carbon weighing 2 g was placed in a petri dish and baked in an electric oven for

1 hour at 100 °C. Prior to being weighed, the hot sample was allowed to cool in a desiccator. This procedure was followed up until a consistent weight was established. Then, Equation 1 was used to calculate the moisture content.

$$\% \text{ moisture content} = \frac{W_1 - W_2}{W_3} \times \frac{100}{1} \quad (1)$$

Where:

W_1 =Weight of petri-dish +sample weight before drying;

W_2 =Weight of petri-dish +sample weight after drying;

W_3 =Weight of sample

Ash content

2g of rubber seeds husk AC was measured and put into a platinum crucible, then heated for 3hrs at 500°C in electric muffle furnace. The heated samples were allowed to cool in a desiccators before being weighed. Ash content(%) was determined by Equation 2.

$$\text{Ash content (\%)} = \frac{W_1 - W_2}{W_3} \times \frac{100}{1} \quad (2)$$

Where;

W_1 = the crucible's empty weight;

W_2 = the crucible's empty weight + sample weight before burning;

W_3 = the crucible's empty weight + ash weight

pH

The sample pH was measured using a H/99/300 Hanna pH meter. Before rinsing with a little amount of sample, the electrodes were first rinsed with distilled water and then dried. A small beaker was filled with enough of the sample to submerge the electrode tip to a depth of about 2 cm. pH meter was turned on to measure sample's pH.

Bulk density

Following filling to the 10 mL mark, rubber seeds husk AC was weighed and added to graduated measuring cylinder with a 10 mL capacity. The cylinder bottom was then repeatedly tapped until the sample level remained unchanged. Bulk-density was calculated using Equation 3.

$$\text{Bulk density g/mL} = \frac{\text{Weight of Sample (g)}}{\text{Volume of Sample (mL)}} \quad (3)$$

Iodine Value

The iodine value was calculated using method of [19]. A 250-mL beaker was filled with 1g of AC and 25mL of a 0.023 M iodine (I) solution. The liquid was vortexed for ten minutes before being filtered through a funnel filled with clean, ashless glass wool. A 0.1095 M thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$) solution was titrated with 20mL of clear filtrate until a stable pale yellow hue was produced. The prepared starch indicator was added, and titration started slowly until a colorless solution was seen. The entire procedure was repeated twice, and then the approach was used without a sample (blank titration). The iodine value was determined using equation 4.

$$\text{Iodine value} = \frac{(V_2 - V_1) \times 12.69 \times N}{\text{Weight of Sample (g)}} \quad (4)$$

Where:

N =Normality of thiosulphate($\text{Na}_2\text{S}_2\text{O}_3$) solution;

V_1 =Volume of thiosulphate($\text{Na}_2\text{S}_2\text{O}_3$) (mL) used for titration;

V_2 =Volume of thiosulphate($\text{Na}_2\text{S}_2\text{O}_3$) (mL) used for blank titration;

W =Mass of sample used(g).

Surface area(A)

Using the sea's method, surface area(A) of the synthesized powdered AC was calculated. The pH was raised to 3 by combining 1.5g of AC with 100 mL of diluted hydrochloric acid. After that, the liquid was thoroughly mixed before 30 g of sodium chloride (NaCl) was added. The final suspension included 150 ml of deionized water (H_2O). To increase sample pH from 4 to 9, sodium hydroxide 0.1N was titrated into the produced solution. It was noted how much sodium hydroxide was used, or volume (V). After that, surface area was calculated using Equation 5.

$$A = 32V - 25 \quad (5)$$

Where:

A =surface area of formulated activated carbon(AC)

V =volume of sodium hydroxide required to increase pH of sample from 4 to 9.

Volatile matter

To determine volatile matter, 1.0g of sample activated carbon(AC) was heated at temperature of 500°C for ten minutes. Using Equation 6, volatile matter was calculated as follows.

$$\% \text{ Volatile} = \frac{\text{Weight of the Volatile component (g)}}{\text{Oven dry weight (g)}} \times \frac{100}{1} \quad (6)$$

Determining the functional groups of rubber seeds husk activated carbon

Using a Fourier-transform-infrared (FTIR) analyzer, surface functional groups of the rubber seed husk AC were identified. Potassium bromide was combined with 0.5g of AC. The mixtures was grounded to fine powder and then placed into a mold to generate fine plates. For analysis, the plates were put into spectrophotometer.

IV. RESULTS & DISCUSSION

Table 1 shows results of the physico-chemical properties of rubber seed husk AC.

Table 1: Physico-chemical Properties of Rubber Seeds Husk Activated-Carbon(AC)

Properties	Physical Activation	Chemical Activation
Bulk Density(g/mL)	0.4784	0.4764
pH	6.4	6.2
Moisture Contents(%)	0.730	0.2976
Ash Contents(%)	29.302	29.302
Volatile Matter(%)	21.66	28.788
Iodine Number(mg/g)	16.624	18.009
Surface Area (m ² /g)	525.4	563.8

For physical activation(PA) and chemical activation(CA) procedures, the bulk densities were 0.4784 g/mL and 0.4764 g/mL, respectively. Physical activation had a larger bulk density, with a 0.002 g/mL difference. The bulk densities that were measured in this investigation were within the 0.45-0.55 g/mL range established by [20]. Additionally, the bulk-density finding agrees with published values for bean husk at 450°C at 0.41 g/cm³, coconut shell at 0.43 g/cm³, and plantain peel at 0.45 g/cm³ [21], [22].

The pH results show 6.4 and 6.2 respectively for physical activation(PA) and chemical activation(CA) method. Physical activation (PA) had a pH that was 0.2 higher than chemical activation (CA). pH is generally affected by method and reagent used for activation process. Likewise, pH also affects activated carbon(AC) adsorption rate. According to reports, basic species adsorb poorly at low pH compared to acidic species, however the opposite is actually true [19], [23]. The result shows that formulated activated carbons(AC's) is slightly acidic. The results are also lower than pH of 6.70 reported for beans husk [21].

The moisture contents of formulated activated carbon(AC) were 0.730 % and 0.2976 %, respectively for physical activated and chemical activated carbons of rubber seeds husk. The moisture contents result are far below 4.5 % minimum standards [20]. Generally, activated carbons products are sold on a moisture-free basis. For many purposes, moisture content does not affect activated carbon(AC) adsorptive power, but they dilute the carbon [19]. Therefore, activated carbon's (AC) moisture content should be as low as possible [24].

Ash content for both physical(AC) and chemical(AC) was 29.302%. Activated carbon's ash-content is used to evaluate its quality. The residual minerals in AC after carbonization and activation process are referred to as ash content [25]. In this present study, ash content was above a maximum of 10 % specified in [20]. But the findings of this study agreed with the 29.47% value for steam-activated eggshells published by [23]. High ash level is also undesirable, as it will reduce the material's overall activity and mechanical strength, according to [26]. Adopting an adsorption technique with low ash concentrations is advised to enhance the potential for adsorption.

As seen in Table1, volatile matter in AC produced was 21.66% for physical activation and 28.788% for chemical activation(CA). Due to activating agent used, there were more volatile materials during chemical activation. Though value obtained for physical activation agrees with 25 % maximum for powder-activated carbon specified by [20]. In contrast to studies done by [27], who reported 57% volatile matter utilizing coconut shells, the values obtained in this investigation were lower, [28] also obtained 78.83 ± 5.58 and 92.03 ± 3.74 respectively for banana's empty-fruit bunch) and Delonix regia fruit-pods. But [29] have a reported volatile matter of 23.33% for carbonized plantain phosphoric(H₃PO₄) acid AC. These

figures are consistent with the findings of this investigation. The substances that have not evaporated during carbonization but have done so at 950°C are generally considered to be volatile matter [30].

Activated carbon's iodine value determines its porosity and adsorbent power. Iodine number of 16.624 mg/g and 18.009 mg/g were obtained respectively for physical AC and chemical AC of rubber seed husk. Iodine number of chemical activation was higher than physically AC in this report. Chemically, AC with higher iodine number will have larger surface area for adsorption than AC with lower iodine value [31]. The higher iodine number of carbons can be linked to presence of large micropore structure and large quantity of carbon having a sizeable surface area due to the enlargement of their pore structure [32]. The results of this study were also better than those reported by [23] for carbonized chicken eggshells (0.276) and steam-activated eggshells (0.322), respectively.

The surface area results for physical activation were 525.4m²/g and chemical activation(CA) was 563.8m²/g. Chemical activation generated AC with larger surface area compared to physical activation(PA). [33] claims that chemically activated carbon's great porosity is a result of the chemicals' dehydration and organic matter destruction during chemical activation, which led to the carbon's enormous surface area. Nevertheless, this result has further confirmed the report of [34], [35], that chemical activation generates high yield and large surface area(A). In comparison, [36] reported surface area(A) of 975 m²/g reported for groundnut shells. [37] also reported surface area of 392 m²/g and 1589 m²/g respectively for KOH(potassium hydroxide) and H₃PO₄(phosphoric acid) treated rubber seed pericarp-based activated carbon, [38] reported 871.22 m²/g for Nipa palm nut. Adsorbents with large surfaces have high adsorption capacities.

Determining surface functional -groups of rubber seed husk AC

The findings of the FTIR spectra of the rubber seed husk AC obtained using physical activation (PA) method and chemical activation method(CA), respectively, are shown in Figures 2 and 3.

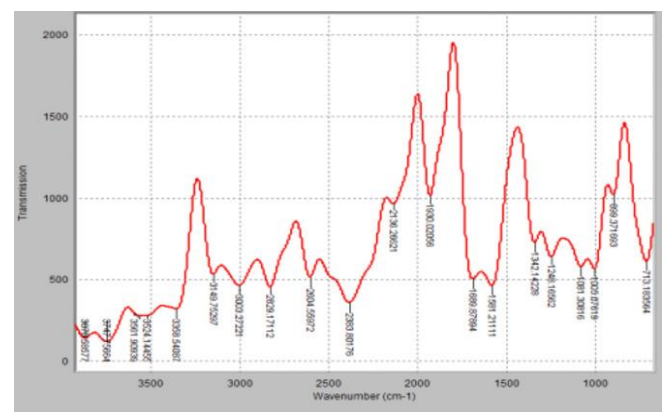


Figure 2: FTIR spectra of rubber seeds husk AC using physical method

Table 2: FT-IR spectroscopy analysis for physical activated carbon

Mid-IR spectrum region	Wavelength (cm ⁻¹)	Appearance	Compound Class	Functional Group
Fingerprint region (600-1500 cm ⁻¹)	713.1836	Strong	C=C bending , C-H	Alkynes
	899.3717	Strong	C-H bending	1,2,4-trisubstituted
	1005.076	Strong	C-F stretching	Aliphatic fluoro compounds
	1081.308	Strong	C-O stretching	Primary alcohol
	1248.166	Medium	C-N stretching	Aliphatic amines
Double bond region (1500-2000 cm ⁻¹)	1342.142	Strong	C-N stretching	Aromatic amines, phenol
	1581.211	Medium	N-H bending	Amines
	1689.879	Strong	C=O	Primary amines
Triple bond region (2000-2500 cm ⁻¹)	1930.021	Weak	C-H bending	Aromatic compound
	2136.266	Weak	C≡C stretching N=C=S stretching	Alkene Isothiocyanate
Single bond region (2500-4000cm ⁻¹)	2383.802	Strong	O=C=O stretching	Carbon dioxide
	2604.559	Strong, broad	O-H stretching	Carboxylic acids
	2829.171	Strong, broad	O-H stretching	Carboxylic acids, Alcohol
	3003.272	Strong, broad	O-H stretching	Carboxylic acids, Alcohol
	3149.753	Strong, broad	O-H stretching	Carboxylic acids, Alcohol
	3358.541	Strong, broad	O-H stretching	Alcohol
	3524.145	Strong, broad	O-H stretching	Alcohol
	3561.909	Medium, sharp	O-H stretching	Alcohol
	3747.757	Medium, sharp	O-H stretching	Alcohol
	3870.586	Medium, sharp	O-H stretching	Alcohol

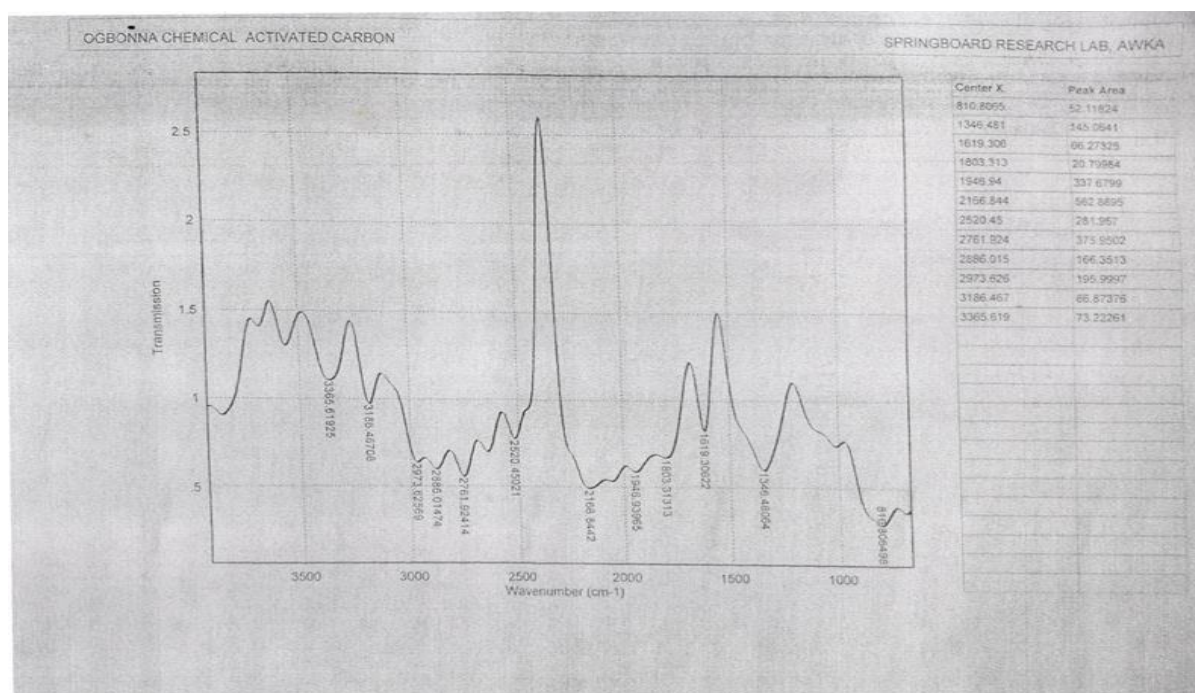


Figure 3: FTIR spectra of rubber seed husk activated carbon prepared using chemical method

Table 3: FTIR spectroscopy analysis for chemical activated carbon

Mid-IR spectrum region	Wavelength (cm ⁻¹)	Appearance	Compound Class	Functional Group
Fingerprint region (600-1500 cm ⁻¹)	810.806	Strong	C-Cl stretching	Halo- compound (Alkyl halide)
	1346.481	Symmetry	O-H bending N-O stretching	Alcohol Nitro compounds
Double bond region	1619.306	Medium	C=C bending	Cyclic alkene,

(1500-2000 cm^{-1})			N-H bending	1 st amines
	1803.313	Strong	C=O stretching	Acid halide
	1946.940	Weak	C-H bending	Aromatic compounds
Triple bond region (2000-2500 cm^{-1})	2166.844	Strong	S-C \equiv N stretching	Thiocyanate(isothiocyanate)
Single bond region (2500-4000 cm^{-1})	2520.450	Strong, broad	O-H stretching	Carboxylic acids, Alcohol
	2761.924	Strong, broad	O-H stretching	Carboxylic acids, Alcohol
	2886.015	Strong, broad	O-H stretching	Carboxylic acids, Alcohol
	2973.626	Strong, broad	O-H stretching	Carboxylic acids, Alcohol
	3186.467	Strong, broad	O-H stretching	Carboxylic acids, Alcohol
	3365.619	Medium	O-H, N-H stretching	Alcohol, aliphatic primary amine

As shown in Figures 2 and 3, the FTIR spectra for the physically and chemically AC's of rubber seed husk in this study exhibit twenty (20) and twelve (12) peaks, respectively, that indicate various functional groups. Table 1 and Table 2 provide summaries of the wavelengths, appearance, compound class, and functional groups of FTIR spectra shown in Figures 1 and Figure 2. According to FTIR data, there were peaks at 713.1836, 899.3717, 1005.076, 1081.308, 1248.166, 1342.142, 1581.211, 1689.879, 1930.021, 2136.266, 2383.802, 2604.56, 2829.171, 3003.272, 3149.753, 3358.541, 3524.145, 3561.909, 3747.757 and 3870.586 cm^{-1} for physical AC. For chemically AC, FTIR spectra established peak values at 810.8065, 1346.481, 1619.306, 1803.313, 1946.94, 2166.844, 2520.45, 2761.924, 2886.015, 2973.626, 3186.467 and 3365.619 cm^{-1} . The mid-IR spectrum, which includes single-bond (2500-4000 cm^{-1}), triple-bond (2000-2500 cm^{-1}), double-bond (1500-2000 cm^{-1}), and fingerprint region (600-1500 cm^{-1}) is commonly utilized for FTIR sample analysis, according to [39]. In Tables 1 and 2, the different regions are listed. Both samples of synthesized AC's have several surface functional groups, including O-H, N-H, C-H, C=C, C \equiv C, N=C=S, S-C \equiv N, C-F, C-Cl and other compound classes, according to FTIR analysis. As shown in Tables 1 and 2, carboxylic acids and alcohols are the most frequent substances in the synthetic activated carbon. As a result of these functional groups, carbon has a better ability to absorb various sorts of compounds [40]. The surface chemistry is additionally influenced by the precursor's chemical composition; these functional groups can contribute to the acid-base balance and are hydrophilic [41]. The findings of this study concurred with those of [42] regarding date stone activated carbon; [36] on groundnut shells where the presence of various functional groups was reported. Rich active surface functional groups of AC's play a crucial role in the chemistry of carbon surfaces and the adsorption of heavy metals [43], [44]. Heavy metals are typically absorbed by physical and chemical interactions between adsorbents and their functional groups, and these interactions frequently have a significant effect [45], [46].

V. CONCLUSIONS

This work has demonstrated that activated carbon may be produced from rubber seed husks using both chemical and physical methods. Furthermore, it is possible to claim that rubber seed husk is a good substitute for other sources of AC precursors, demonstrating how some potential solid

waste of plant origin can be converted into beneficial products that can be applied in a variety of ways. The ability to absorb pollutants will also be improved by chemical AC because it has a large surface area (A) than physical AC. The presence of these functional groups like O-H, C-H, C-C, C-N, C=C, C-I, C \equiv N and C \equiv C may also have an impact on how well the produced AC performs on its surface.

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