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Characterization of activated carbon synthesized from Almond and Groundnut shells

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Abstract

Agricultural waste has been used to synthesize high quality Activated Carbon (AC) with versatile application. This study synthesized AC from agricultural waste (almond and groundnut shells) by chemical activation using phosphoric acid and zinc chloride. Physicochemical properties of the produced AC were carried out using standard analytical methods while, chemical characterization was carried out using Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM) equipped with EDX detector. The results of physicochemical properties of the produced ACs were as follows: ash content (5.01- 7.00) %, bulk density (0.28-0.51) g/mL, porosity (45.0- 52.0) %, moisture content (0.66- 1.11) % and pH (5.04- 6.50). FTIR results indicate that regardless of activating agents, all the activated carbon produced contains oxygenated functional groups; O-H (hydroxyl group), C-O (phenol group) and C=O (carboxylic group). Variation in morphology of the produced AC was observed in terms of the applied precursor and activating agent. The external surfaces are full of cavities and quite irregular as a result of activation. The type of activating agent influences the percentage yield of carbon in the produced AC and the carbon matrix also contains some heteroatoms which govern their surface chemistry. The study recommended that the produced activated carbon can be used as adsorbent in the removal of organic pollutants in wastewater.

1. Introduction

The importance of activated carbon to a growing society cannot be overemphasized. Carbonaceous materials are activated in order to improve their properties and usability. Activated Carbons (AC) are highly porous adsorbent materials with high surface area which have wide applications in domestic, commercial, environmental and industrial settings [1-3]. Several reports have indicated that AC is an effective adsorbent in water treatment, primarily to remove organics and other compounds that affect the taste and odour of municipal wastewater [4-6]. They also function as antidote for treating poisoning and overdose [7].

The production of highly efficient commercial activated carbon does not only attract a huge acquisition cost but the process of its production, regeneration and re-use had also not been fully investigated (especially in developing countries) [8,9]. The difficulties encountered in procuring commercial AC have led researchers to sourcing agricultural byproducts as alternative precursor for the production of cheap and efficient AC. These byproducts are abundant, readily available with low-cost and renewable.

These byproducts represent unused resources and environmentally friendly materials with high carbon content [2,10-12]. AC with high adsorption capacity, considerable mechanical strength, and low ash content have been synthesized from agricultural wastes like bagasse, hard shells of apricot stones, almond, walnut and hazelnut shells [9,11,13-19].

Almond and groundnut shells are typical wastes which are generally discarded and readily available as lignocellulosic materials. Groundnut shell is a carbonaceous fibrous material while, the cell walls of almond shell consist of cellulose, silica, lignin and carbohydrates which have hydroxyl groups in their structures [10,11]. Nigeria produces 100,000 tons of almond fruit (*Terminalia catappa*) [20] and 6,217.300 Tonneth of Groundnut annually (CEICDATA.COM National Bureau of Statistics of the Federal Republic of Nigeria, 2017)

Activated carbon is produce by the exposure of these precursors either by physical or chemical activation methods. By this, the high quality activated carbon produced can compete with the commercially produced ones. Chemical treatment involves carbonization of raw material followed by activating with strong dehydrating and oxidizing agent such as H_3PO_4, $ZnCl_2$, KOH and NaOH under nitrogen atmosphere [6,21]. Physical activation involves carbonization of raw material in the absence of oxygen and in the presence of inert gases followed by activation using oxidizing agent such as steam, air, carbon dioxide or their combination [18]. In many cases, chemical activation is preferred to physical activation because it offers several advantages like decrease activation temperature and production of AC with well-developed porous structure [9,18,22]. Production of cheap AC from agricultural waste will assist agricultural economy serving as an alternate source of income. It will also reduce the cost of waste disposal thereby, making the environment cleaner [4,12]. The objective of this study is to produce porous activated carbon from agricultural waste (Almond and Groundnut Shell) using zinc chloride and phosphoric acid as chemical activating agents. Characterization study was carried out using Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM) equipped with EDX detector.

2. Material and Methods

2.1. Sample collection and preparation

Almond and groundnut shells were selected for this research based on availability and accessibility. Almond fruits were first collected from Itamerin Comprehensive High School, Ago-Iwoye, Ogun State, Nigeria, while groundnuts fruits were bought from a local market in Ibadan, Oyo state. After mechanical crushing to remove the inner seeds, the shells were washed with warm water, rinsed with distilled water and sun dried for 5 days in order to reduce moisture content. The dried shells were then crushed into smaller sizes with mortar and pestle and prepared ready for carbonization in the furnace.

2.2. Carbonization and activation

Carbonization was done using vecstar furnace set at 500 ^oC for 2 hrs. The carbonized product was allowed to cool and prepared ready for activation with H_3PO_4 and $ZnCl_2$.Chemical activation was carried out using the method of Udeozor and Evbuomwan, [23] with slight modification. 50 g of each of the carbonized product was mixed with 150 mL Phosphoric acid for H_3PO_4 activation and 150 mL of 0.5 M Zinc Chloride for $ZnCl_2$ activation. Each was thoroughly mixed until it formed a paste which was then transferred into a crucible. The crucibles containing the samples were placed in the furnace and heated at a temperature of 500 ^oC for 2 hr. After cooling, each sample was washed with 500 mL of 1.2 M HCl followed by 500 mL distilled water until neutrality to remove excess activating agent before filtration with whatman filter paper. The residue was oven-dried at 110 ^oC for 1 hr, and then kept in an air tight container for further analysis and labeled as GPAC (Groundnut Phosphoric acid Activated Carbon), GZAC (Groundnut Zinc chloride Activated Carbon), APAC (Almond Phosphoric acid Activated Carbon) and AZAC (Almond Zinc chloride Activated Carbon).

2.3. Physical Characterization of the produced Activated Carbon

Characterization of the various properties of the produced activated carbons was carried out using standard methods. The pH, ash content, moisture content and porosity were determined using the method of Gumus and Okpeku, [17]. Bulk density was determined using the method of Adiotomre, [24]. Ash content was calculated using the equation:

$$\text{Ash \%} = \frac{\text{Ash Weight}}{\text{Oven dry weight}} \text{X } 100. \quad (1)$$

The percentage moisture content was determined using this equation:

$$X_o \text{ \%} = \frac{W1-W2}{W1} X100 \quad)$$

Where, X_o % = moisture content on wet basis, W_1= initial weight of sample in grams, W_2 = final weight of sample (in grams) after drying. The bulk density was calculated using this equation:

$$\text{Bulk density} = \frac{\text{Mass of sample}}{\text{Bulk volume}} \quad (3)$$

2.4. Chemical characterization

2.4.1. Fourier Transform Infra-Red (FT-IR) Analysis

Functional groups and the chemical bonds present in each sample were determined using Fourier Transform Infrared Spectroscopy (FTIR). The FTIR spectra of the materials were acquired in the range 380–4000 cm^{-1} at a resolution of 4 cm^{-1} using Spectrum 100 infrared spectrometer equipped with universal diamond crystal attenuated total reflection(ATR) accessory (Perkin Elmer, USA).

2.4.2. Scanning Electron Microscopy (SEM)

Morphological studies of the samples were carried out using a Zeiss Ultra Plus field emission gun scanning electron microscope (FEGSEM) equipped with EDX detector (Germany). Materials were deposited on carbon tape stuck to aluminum stubs and were coated with gold with the aid of sputter coater to minimize charging. It was set at a voltage of 1.2 kV (10 mA) and a vacuum 20 Pa for 10 min. Elemental composition of the samples was also observed using scanning electron microscopy equipped with energy dispersive spectrometer.

3. Results and discussion

3.1. Result of physico-chemical properties

The results of the physico-chemical properties of the activated carbon produced from almond and groundnut shells are presented in Table 1. pH of the produced AC follows this trend: GZAC<APAC<GPAC<AZAC. GZAC is the most acidic while others are slightly acidic. Generally, adsorbents with pH of 6-8 are acceptable in most applications and can be used for commercial purpose [25]. pH of APAC, GPAC and AZAC falls within this range. The pH results recorded in this study is similar to that of Kamaraj and Umamaheswari, [12] which affirms that maximum adsorption takes place at slightly acidic and neutral medium. Activated carbon produced from this work can hereby be used to treat water samples with high alkalinity.

The produced activated carbon exhibited low bulk density with GPAC having the lowest and APAC the highest. The bulk density of the samples follows this trend: GPAC<AZAC<GZAC<APAC, the bulk density of APAC is higher than some of the reported results in literature (Table 1). Bulk density is an important property used in characterizing good AC. It gives a measure of the amount of AC required as

adsorbent per volume unit and determines its adsorptive strength and retention level [26,27]. The higher the density the better the filterability of activated carbons and the better the quality of AC [25,28].

The result of moisture content follow this trend; GZAC<AZAC<GPAC≈APAC, moisture content of AC produced by activating with phosphoric acid is higher than zinc chloride. Generally, the recommended activated carbon storage moisture content is less than 3 % [29].The moisture content of all the produced AC are lower than the recommended level, theone recorded for commercial AC (CAC) (Table 1) by Al-Balushi et al. [30] and those reported in literature by Aji et al. [15],Gumus and Okpeku, [17] and Ratan et al. [31]. High moisture content encourages the growth of microorganisms (fungi) within the macropores and micropores of the activated carbon, blocking the pores and thereby reducing the adsorptive capacity of the carbon and the shelf-life [32]. Also, excessive moisture can dilute activated carbon and affect the actual weight required during the adsorption experiment [33]. Hence, moisture content of activated carbon should be maintained as low as possible.

Porosity describes the number of pores present in a sample. A pore is a class of void connected to the external surface which allows the passage of fluids into or through a material. The porosity of all the produced AC is similar with AZAC having the highest. Porosity enhances adsorption capacity of the adsorbent as highly porous carbon can adsorb large amount of organic compounds [31,34].

Table 1: Physicochemical properties of the produced activated carbon.

Properties	GPAC	GZAC	APAC	AZAC	CAC	DSAC	GPAC (Aji)
pH	6.07±0.03	6.50±0.05	6.09±0.04	5.04±0.02	9.10	6.70	6.80
Bulk density (g/mL)	0.28±0.02	0.33±0.01	0.51±0.02	0.30±0.00	0.35	0.36	0.42
MoistureContent (%)	1.10±0.05	0.66±0.07	1.11±0.07	0.68±0.08	4.95	7.99	15.00
Porosity (%)	45.0±2.5	45.0±1.5	48.0±3.4	52.0±3.5			
Ash content (%)	6.00±0.88	7.00±0.92	5.01±1.02	6.00±0.96	5.00	6.00	6.00

CAC – Commercial AC and DSAC – Date AC from Al-Balushi et al., [30] GPAC (Aji) – Groundnut Phosphoric acid AC from Aji et al. [15]

Ash content is the residue that remains when carbonaceous portion of a material is burned off and it is a measure of inorganic impurities in the carbon [35,36]. The ash content of all the produced AC falls within the range of most ash content of agricultural waste according to ASTM standard (≤8). GZAC has the highest while APAC the lowest. Ash content of phosphoric acid activated AC is less than that of zinc chloride activated AC. Phosphoric acid activated AC are considered most active since ash in activated carbon is not desirable and is considered an impurity [37]. Ash content recorded in this study compared favourably with those reported by Aji *et al*. [15], but varies greatly with that of Nabais *et al*. [38] and Ratan, [31] which gave a lower value of 1.1 and 1.0 respectively.

3.2. Chemical characterization

3.2.1. Functional group characterization using FTIR

The surface chemistry of activated carbon determines its applicability and adsorptive behavior [39]. Fourier Transform Infrared Spectroscopy (FTIR) was used to determine the functional groups present on the produced AC. Figure1 shows the FTIR results for Groundnut and almond shell using Phosphoric acid (A &C) and Zinc Chloride (B & D) as activating agents. The peaks appearing in the FTIR spectrum were assigned to various functional groups according to their respective wave numbers. Functional groups of activated carbon determine their chemical reactivity and influence their interaction with adsorbates [19].

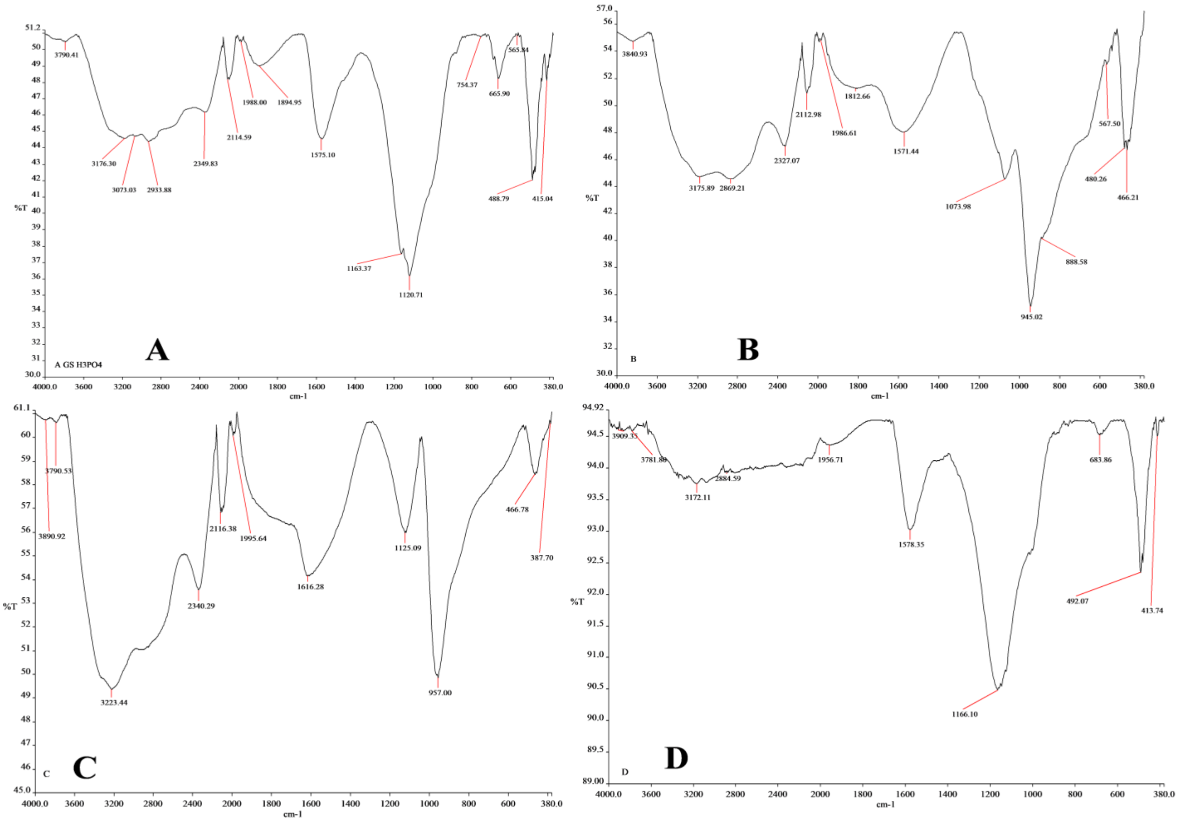

Figure 1: FTIR result of the produced activated carbon from Groundnut and almond shell using Phosphoric acid (A &C) and Zinc Chloride (B & D) as activating agent.

Peaks observed at 3709, 3804 cm^{-1} 3790 cm^{-1} 3781 cm^{-1}in GPAC, GZAC AZAC and APAC corresponds to O-H stretching vibrations from alcohol, water molecule or carboxylic acid. This is found in all the produced AC and it is a common peak usually found in most AC produced from agricultural waste [10,15]. Some similar peaks were found in AC produced by activating with either phosphoric acid or zinc chloride. The peaks found in the spectrum of GPAC at 3176 cm^{-1} and 3073 cm^{-1}, GZAC at 3175 cm^{-1}, and AZAC 3172 cm^{-1} corresponds to C-H stretching vibrations for an alkene. The peaks at 2933 cm^{-1} (GPAC), 2869 cm^{-1} (GZAC), correspond to C-H stretching vibrations for an alkane [11]. The peak at 3223 cm^{-1} corresponds to a C-H stretching vibration for an alkyne which were found on almond produced AC (3223.44 (APAC) and 3172.11 (AZAC)). Also, similar peaks at 2349 cm^{-1}(GZAC), 2327 cm^{-1}(GPAC), 2340.29 cm^{-1} (APAC)corresponds to a C=O stretching vibration for a carbonyl group.
The peak observed on GPAC at 2114 cm^{-1}, GZAC at 2112 cm^{-1}and APAC at 2116.38 corresponds to a C≡C stretching vibration but, this was not observed in AZAC. The peaks at 1575 cm^{-1}, 1571cm^{-1},1578 cm^{-1} 1616 cm^{-1}observed in the spectra of GPAC, APAC,GZAC and AZAC respectively corresponds to C=C aromatic ring stretching vibrations [9] and peaks at 1120 cm^{-1},1073 cm^{-1}, 1125 cm^{-1} and 1166 cm^{-1} for GPAC, GZAC, AZAC and APAC corresponds to Si-O or C-O stretching in alcohol, ether or hydroxyl groups [18,40, 41].
The peak at 888.58 cm $^{-1}$ corresponds to O-O stretching [10] as found in GZAC. The region 450-750 cm^{-1} showed six bands (cm^{-1}) 666.90, 565.84, 488.79, 480.26, 567.50 and 466.21 for both zinc chloride and phosphoric acid activated carbon ground nut shell and three bands in the 466.78, 492.07 and 683.86 cm^{-1} for both almond shell zinc chloride and phosphoric acid are associated with the in plane and out-of-plane aromatic ring deformation vibrations [18,38,41,42]. The peaks observed at 1120.71 cm^{-1} and 1125.09 cm^{-1} for AC activated using phosphoric acid.

(GPAC and APAC) could be assigned to the phosphorous species i.e. hydrogen-bonded P=O, O-C stretching vibrations in P-O-C of aromatics [40] and P=OOH [34], which suggests the incorporation of this element in the activated carbon structure as observed in figure 1. This is not found in zinc chloride activated carbons.

FTIR results indicate that regardless of activating agent, all the produced activated carbon contains oxygenated functional groups; O-H (hydroxyl group), C-O (phenol group) and C=O (carboxylic group).This is similar to the findings of researchers in the literature [18,40,41,42].Though, more oxygen containing sharp peaks were observed in AC produced by activating with phosphoric acid compared to that of zinc chloride.

3.2.2. Morphological analysis using SEM

The surface morphology of the produced activated carbons was examined using a scanning electron microscope. Figure 2 shows the SEM micrograph of groundnut shell activated with Zinc Chloride(GZAC)(1) and phosphoric acid (GPAC)(2) at 10 µm (A) and 200 nm (B) while figure 3 shows the SEM of almond shell activated with phosphoric acid (APAC) (1) and Zinc Chloride(AZAC) (2) at 10 µm (A) and 200 nm (B).

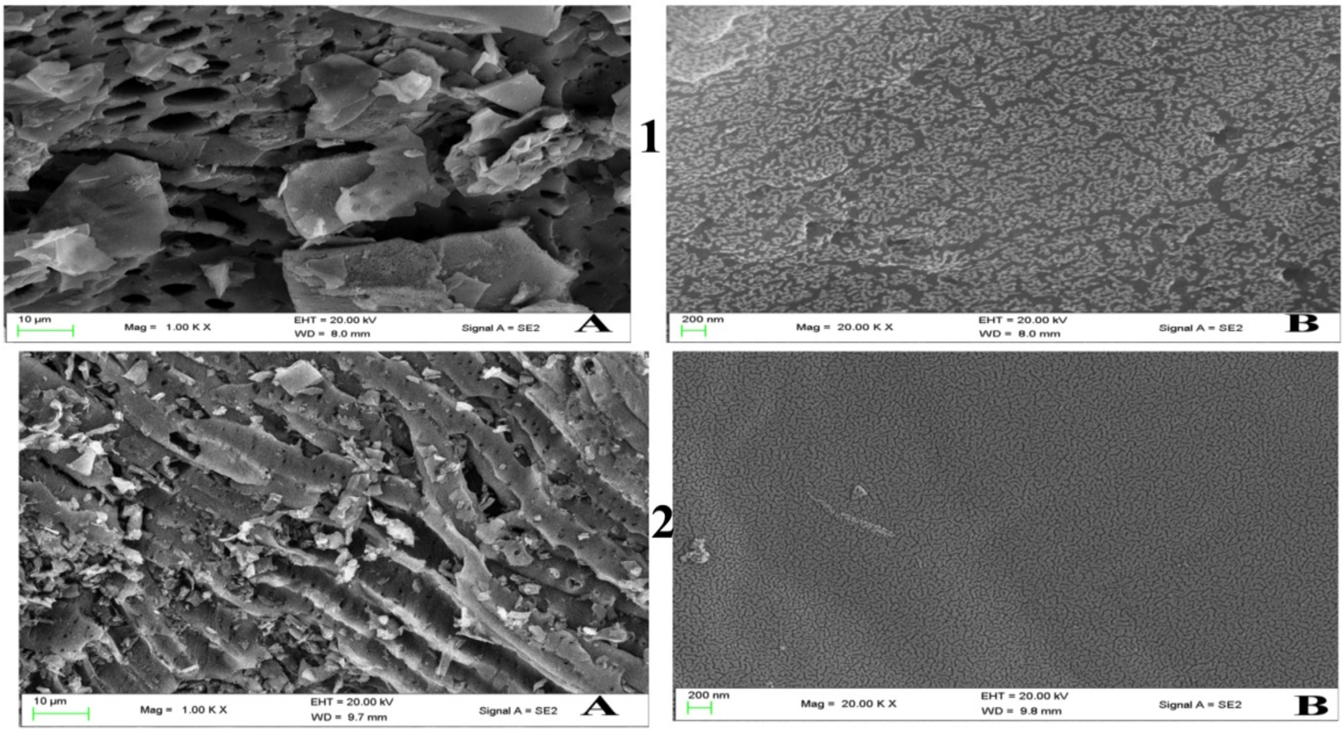

Figure 2: Scanning electron micrographs of groundnut shell activated with Zinc Chloride GZAC (1) and H_3PO_4GPAC (2) at 10 µm (A) and 200 nm (B).

Variation in morphology of the produced AC was observed in terms of precursor used and activating agent. The SEM result of almond AC differs distinctly from that of groundnut. Also, there is slight difference in the morphology of GZAC and GPAC, for 10 µm, the SEM result of GZAC shows that the surface is irregular with rough and heterogeneous cavities due to activation as there are numerous cracks and pits distributed over the external surface. SEM result of GPAC shows rough surface with broken sheet-like morphology. For 200 nm, the surface of GZAC has many irregular micro cracks while GPAC surface appears homogenous and smooth with many micro pores as well. According to the report of Aji et al. [15], the SEM micrographs of carbonized groundnut shell before chemical activation does not have porous surface, but the micrographs of the produced ACs are full of micropores which are due to chemical activation. The SEM result of APAC at 10 µm has some smooth flaky structure with tiny pores having big groove on one side while that of AZAC has rough irregular broken sheet-like morphology, many tiny pores with heterogeneous surface. Observing the samples at 200 nm, numerous micro pores are seen on the surface of the activated carbon with little cracks [44].

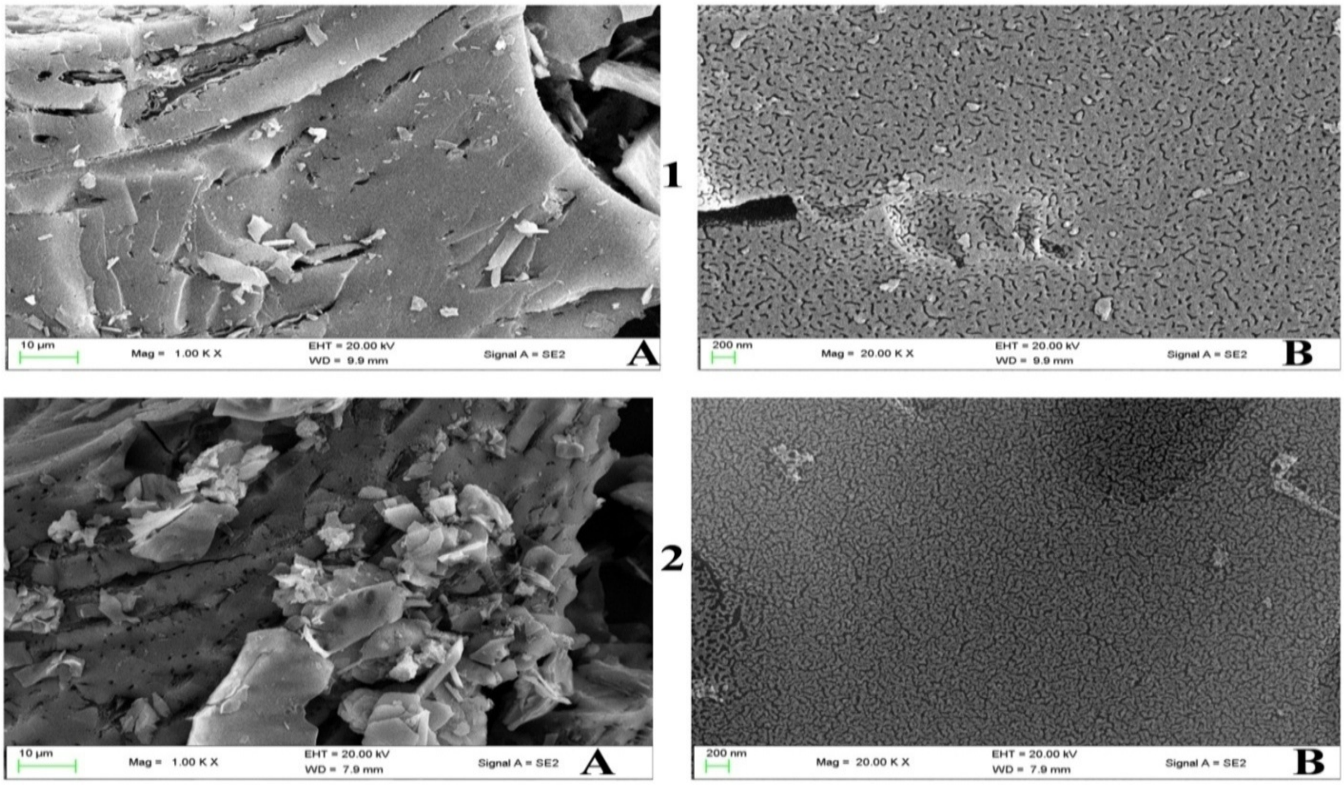

Figure 3: Scanning electron micrographs of almond shell activated with H_3PO_4 APAC (1) and Zinc Chloride AZAC (2) at 10 µm (A) and 200 nm (B).

The openings and holes on it also indicate that there is an increased surface area which can aid adsorption. Apart from having numerous micro pores with irregular big crevice, APAC and AZAC also contain some irregular crevices that are more pronounced in AZAC at 200 nm, indicating relatively high porosity and surface area [14].

3.2.3. Energy Dispersive X-ray Analysis of the produced ACs

Energy dispersive X-ray (EDX) analysis of the produced ACs was performed to estimate the percentage composition of various elements present; the result is presented in figure 4. In this study, the type of activating agent somehow determines the elemental composition of the produced AC. As expected of carbonaceous material, there is a high carbon and oxygen content in all the AC produced [31]. The type of activating agent affects the percentage yield of carbon in the produced AC. The percentage of carbon in phosphoric acid activated carbon is higher than that of zinc chloride activated carbon. The percentage of C, O, and P are similar in GZAC and AZAC. Common elements present in both include: C, O, Al, Si, P, Cl, K, Ca, Fe, and Zn. The percentage of C in APAC and GPAC are similar, the percentage of C in almond activated carbon is more than that of groundnut AC. The carbon matrix also contains some other atoms called heteroatoms like oxygen, halogen, and phosphorus.

These heteroatoms are bonded to the edges of the carbon layers; govern the surface chemistry of the activated carbon and influence the adsorbent– adsorbate interactions [34]. These are found in all the produced AC. Presence of other elements in the produced ACs especially ACs produced by activating with zinc chloride may be due to the biological makeup of the plant or impurities from chemical used in the activation process.

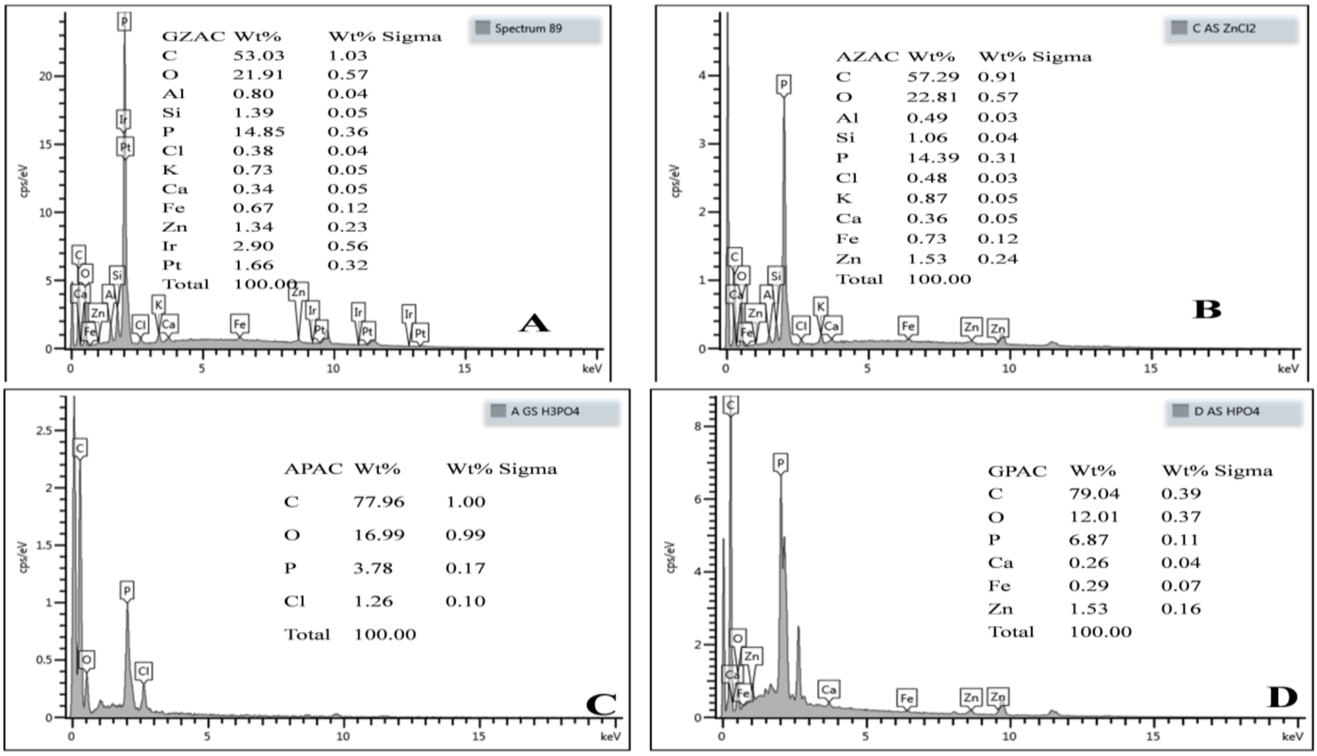

Figure 4: EDX spectra of AC produced by activating with Zinc Chloride; for groundnut (GZAC) (A), for Almond AZAC (B) and AC produced by activating with Phosphoric acid; for Almond (APAC) (C), for groundnut GPAC (D)

Conclusion

Activated carbons with good properties were successfully produced from readily available agricultural wastes like groundnut and almond shells using phosphoric acid and zinc chloride as activating agents. The ACs produced possesses good properties that can compete favourably with commercial AC. The phosphoric acid activated carbon possessed better characteristics than zinc chloride activated carbon. Based on all of these properties, the study recommended that the produced activated carbon can be used as adsorbent in the removal of organic pollutants in wastewater.

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