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Preparation and Characterization of Activated Carbon from Groundnut and Egg Shells as Viable Precursors for Adsorption

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ABSTRACT: This study was carried out to prepare groundnut shell (GS) and eggshell (ES) into activated carbon (AC) and characterize the AC using Association of Official Analytical Chemists (AOAC) and American Standard for Testing and Materials (ASTM) methods. The AC produced was characterized for: pH, moisture content, volatile matter, ash content, fixed carbon, bulk density and surface area. Surface functional groups and surface morphology were also determined using Fourier Transformed Infrared (FT-IR) and Scanning Electron Microscope (SEM) respectively. The ranges of the following results were achieved for the biomasses: Groundnut shell Activated Carbon (GSAC) and Eggshell Activated Carbon (ESAC) respectively: pH (6.80±0.101–7.80±0.011); moisture content (14.10±0.101−12.90±.110%); volatile matter (9.20±0.112−9.90±0.012%); ash content (8.98±0.111−5.80±0.111%); fixed carbon (67.70±0.010−71.40±110%); bulk density (370.00±0.000−380.00−0.000 g/L); surface area (880.00±0.100−800.00±0.000 m^2/g). The agro-wastes have high carbon contents and low inorganic which make them viable adsorbents. FT-IR analysis revealed the presence of oxygen surface complexes such as carbonyls and OH groups on the surface of the ACs in addition to good pore structures from SEM studies revealed that the agro-wastes could be good precursors for ACs production. The overall results showed that the AC produced from the agro-wastes can be optimally used as good and effective adsorbents, thereby ensuring cheaper, readily available and affordable ACs for the treatment of effluent, waste water and used oils.

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Activated carbons are black solid substances resembling granular or powdered charcoal. They are economically produced by the activation and pyrolysis of renewable, readily available and cheaper carbonaceous precursors which are mainly industrial and agricultural by-products such as bagasse (Boonpoke *et al*., 2011), rice husk (Boonpoke *et al*., 2011; Ajala and Ali, 2020), coconut shell (Gawande and Kaware, 2017), sawdust (Alzaydien, 2016; Subramani and Revathi, 2015), empty palm fruit bunch (Hidayat and Sutrisno, 2017), Physic nut waste (Elelu *et al*., 2019), pruning mulberry shoot (Wang *et al*.,2010), bamboo stem (Ijaola *et al*., 2013), chickpea (Ozsin *et al*., 2019), acorn shell (Saka, 2012). The process produces a porous materials with a large surface area (500-1500 m^2/g) (Wang *et al*., 2010) and a high affinity for organic compounds, chlorine, heavy metals, objectionable tastes and odour in effluent or colour substances from gas or liquid streams (Ajala and Ali, 2020). This is possible as a result of their highly developed pore structure and large internal specific surface area (Mansour *et al*., 2020; Wan et al., 2010; Hidayat and Sutrisno, 2017). However, the performance properties of activated charcoal depend largely on raw material source (Sivakumar *et al*., 2012). Adsorption of pollutants on activated charcoal has become acceptable as a result of its versatility, environmental compatibility, relative abundance and low- cost starting materials, usually, waste products, adsorption of a broad range of pollutants, fast adsorption kinetics, and ease of production (Mansor et al., 2020; Amirza *et al*., 2017; Reza *et al*., 2020). Activated charcoals had been used successfully in modern waste treatment plants for water filtration and detoxification treatment of impure waters (Ajala and Ali, 2020; Abraham *et al*., 2018; Jacob *et al*., 2017; Olagunju *et al*., 2015; Ijaola *et al*., 2013; Sivakumar *et al*., 2012), effluent and waste treatment (Marichelvan and Azhagurajam, 2018; Tak *et al*., 2015; Yusufu *et al*., 2012), adsorption of pesticide (Gokhale, 2020), dye adsorption (Wu *et al*., 2020; Mansour *et al*., 2020; Ani *et al*., 2020), heavy metal sorption from aqueous media (Ijaola *et al*., 2013; Mopoung *et al*., 2015; Elelu *et al*.,2019; Özsin *et al*., 2019; Ani *et al*., 2020), and prevention against novel Corona virus (SARS-CoV-2) (Reza *et al*., 2020). Commercial activated charcoals

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are expensive due to the use of non-renewable and relatively high cost starting materials such as coal which are not suitable with respect to pollution control measure (Olagunju *et al*., 2015). In most developing countries, the demand for activated carbon is met by importation at high price, whereas there are massive agricultural and industrial wastes which can be used for its production to meet the local demands and for possible exportation. Recently, researchers had produced activated charcoals from renewable and cheaper precursors, which were mainly agricultural and industrial by-products. Despite extensive scientific researches on its production, there has been dearth of information with respect to its production from groundnut shell, hence, this study. This study was therefore aimed at producing and characterizing an activated charcoal from groundnut shell using $ZnCl_2$ as activating agent. Groundnut is an important subsistence food crops throughout the tropics. The shells have been utilized in a variety of application, such as source of activated carbon (Shukia and Pai, 2005), as fuel when pelletized and made as smokeless briquette (Harrel *et al*, 2010) as a soil conditioner, filler in fertilizer and feeds, or is processed as substitutes for cork and hardboard, or composting with the aid of lignin composting bacteria (Nautiyal, 2002). Eggshell is a by-product of baking industry and confectionaries. Chicken's eggshell typically consists of three parts (ceramic materials present in the outer cuticle, a spongy (calcareous) layer and inner adsorption ability of eggshell and its active carbon for removal of impurities from waste oils (Onawumi *et al*., 2017; Didar, 2017).

The objective of this work is to prepare and characterize cheap, readily available and eco-friendly ACs derived from waste biomasses.

MATERIALS AND METHODS

Procurement and preparation of samples: Procurement of agro-waste: The Egg shells were collected from two different eateries: Mr Biggs Igbonna Area and Osun Mall, Fakunle Area, Osogbo, Osun State, while the groundnut shells were collected from the farm settlement at Idi-Osan, Iragbiji, Boripe LGA, Osun State, Nigeria.

Preparation: The samples were washed thoroughly with tap water in the laboratory and rinsed severally with distilled water to remove stones, debris and dirt. The samples were sun dried for 24 hours and oven-dried at 105ºC for 5 hours and allowed to cool in desiccators. The dried samples were pulverized to a desired particle size.

Modification of agro-wastes: The method described by Bello *et al*. (2017) was slightly modified by increasing the molarity of Phosphoric acid from 0.3 M to 0.5 M. Dried and pulverized sample were carbonized using muffle furnace, a carefully weighed 14.0 ± 0.01 g of raw sample were put into a beaker containing 250 cm^3 of 0.5 M phosphoric acid (H_3PO_4). The content of the beaker were thoroughly mixed and heated on a hot plate until a thick paste was formed. The pastes of each sample were transferred into a crucible which was placed in a furnace and heated at 500ºC for 1 hour. Thereafter, the samples were allowed to cool and then washed with distilled water to a pH of 6.8 ± 0.10, oven dried at 105ºC for 5 hours and the adsorbents were stored in an air-tight container for further analysis and usage Bello *et al*.(2017)

Characterization of agro-wastes

pH Value: 3g of each sample was weighed and soaked into 30ml of boiling deionised water for 24hrs. The pH readings was observed with a digital pH meter, Jenway 3520 (ASTM: D 3838). The pH of carbon is important to the adsorption of pollutant in solution.

Determination of moisture content: Three crucible were cleaned with ethanol, dried, labelled A, B & C and pre-weighed using an analytical weighing balance. 2g of each of the biomasses was weighed in each Petri dish. The sample was dried in the vacuum oven at a temperature of 50^0C for 3 hours, cooled in desiccators and weighed. The drying and weighing was repeated twice until constant weight was achieved. The moisture content was achieved following the method of AOAC, (2019), (ASTM: D 2974-2014, Boadu *et al*., 2018).

$$\% MC = \frac{Wfs - Wds}{Wfs} \times \frac{100}{1}$$

Where Wfs = weight of fresh sample; Wds = weight of dry sample

Volatile matter: Volatile matter content was determined according to standard method (ASTM: D 2974-2014, Boadu *et al*., 2018). 1g of samples was taken in a pre-dried crucible and covered with lid, the heated in a Gallenkamp muffle furnace regulated at 950^0C for 30 minutes. After heating, the plate was quickly covered, cooled in desiccators and weighed. The amount weighed was taken as volatile matter.

Determination of ash content: Three crucibles were cleaned with ethanol, dried, labelled A, B & C and pre-weighed using an analytical weighing balance 2g of biomass was weighed in each crucible. The sample was dried in the furnace at a temperature of 650^0C for 4 hours, cooled in desiccators and weighed (AOAC, 2019).

$$\%\ Ash\ Content = \frac{Mc + Fs - MC}{Mc + Fs} \times \frac{100}{1}$$

Where Mc = mass of crucible; Fs =fresh sample

Ash content was determined according to standard method (ASTM: D 2974-2014, Boadu *et al*., 2018). 5g of dried samples were weighed into a crucible of a known weight and heated in a Gallenkamp muffle furnace for 6hrs at 600^0C. When constant weight was achieved, the crucibles was allowed to cool in desiccators. The mass of the ashed carbon was determined. The weight of ashed carbon is expressed as the percentage weight of the original carbon sample.

$$\%\ \text{Total Ash} = \frac{D-C}{D-B} \text{x}100$$

Where: B = Weight of the crucible (g); C = Weight of crucible + original sample (g); D = Weight of crucible + ashed sample (g)

Determination of fixed carbon: The fixed carbon was determined by subtracting the sum of the moisture content, ash content and volatile matter content in percentage composition from hundred. The value of fixed carbon content obtained is expressed in percentage

FC = 100% - (MC+AC+VM) %

The value for the fixed carbon content should be equal or greater than 65% for a good activated carbon (Olayiwola *et al*., 2015)

Bulk density: The standard procedure used in analysing bulk density was from Akpapunam and Markakis (1981). 5g of the sample was placed into a pre-weighed 5ml measuring cylinders (w_1). The cylinders was gentle tapped to eliminate air spaces within the samples in the cylinders to give a possible close pack (PBD). The volume occupied by the samples and the added weight in the cylinders were determined using analytical weighing balance and will be recorded as (w_2). The bulk density is expressed as:

$$\textit{Bulk Density} = \frac{W2-W1}{Volume\ of\ cylinder}$$

Where: W_2 = weight of samples and cylinder (g); W_1= weight of measuring cylinder (g)

Particle size: Particle sizes of the ground samples alone was determined. The samples were prepared using electric blending machine after which a sieve analysis was carried out using CONTROLS MILAND-ITALY D402-01 MATR 84000 109 sieve shaker at rotation of 10-15 min with (2-36mm, 1.18mm, 0.6mm, 212µm) sieves (ASTM D-2862-97).

$$\%\ C = \frac{Weight\ of\ carbon\ after\ sieve}{Total\ weight\ of\ carbon}$$

Fourier Transform Infrared Spectroscopy (FT-IR) Analysis: FTIR is an instrument used in determining the surface functional group of a material. The FTIR spectroscopy method was employed in this study in order to observe the functional groups of the different agricultural waste and probably deduce their surface chemistry and hence their structures. It also gives information on the possibilities of the functional groups of chemical activated adsorbents. FTIR spectra was obtained with dried powder of different agro-waste samples under consideration. 100 mg of potassium bromated (KBr) was weighed on a sensible weighing balance and mixed with 2.1mg of adsorbents powder in a mortar and pestle. The mixture was compressed in a compressor machine until the sample was compacted. Samples was placed in a cell before fixing it in a Parkin Elmer FT-IR system BX spectrum and spectra reading will be taken (Munagapati and Dong-Su, 2016, Munagapati *et al*., 2018; Zhao, 2018, Ridha *et al*., 2018 and Naba and Sumana, 2018)

Scanning Electron Microscopy/ Energy Dispersive X-ray Spectroscopy (SEM/EDS) Analysis: The surface morphology of the adsorbent can be demonstrated by SEM photograph, using a JSM_7610F (Tokyo, Japan). SEM is a type of electron microscope that produces images of a sample by scanning it with a focussed beam of electrons. The electrons interact with atoms in the sample, producing various signals that can be detected and that contain information about the sample's surface topology and composition. The electron beam is generally scanned in a raster scan pattern, and the beam's position is combined with the detected signal to produce an image. SEM can achieve resolution better than one nanometer. Specimen can be observed in high vacuum, low vacuum, wet conditions (in environmental SEM), and at a wide range of cryogenic or elevated temperature (Vent Kat and Vijay Babu, 2013; Munagapati and Dong-Su, 2016; Munagapati *et al*., 2018 and Naba and Sumana, 2018, Boadu *et al*., 2018).

Adsorbent characterization: Tables 1 and 2 showed the physicochemical and proximate analyses carried out on the prepared activated carbon indicated varying properties. The moisture content shows lower value of 12.90±0.110% for ESAC than GSAC which has value of 14.10±0.101 %. Activated carbon with lower moisture content has been reported to have more adsorption efficacy (Elelu, *et al*., 2019). The moisture

content of ESAC revealed that it will be more effective than GSAC. The pH showed a higher value in ESAC with 7.80±0.011 as against 7.10±0.000 in GSAC, pH of carbon is important to the adsorption of pollutant in solution and pH range of the produced activated carbons falls within the range of most agricultural wastes as reported by other literatures. GSAC gave higher values of moisture and ash contents with 14.10±0.101, 8.98±0.111 respectively calculated in percentage composition than ESAC. Therefore, the result indicates a higher value of fixed carbon content for ESAC as compared to GSAC with values of 71.40±0.001 and 67.70±0.010 respectively this is in agreement with the results reported by Aji *et al*. (2015), Aji *et al*. (2017), Malik *et al*. (2006), adsorbents with fixed carbon ≥ 65 is considered suitable for adsorption (Olayiwola *et al*, 2015). Furthermore, according to Xiong *et al*. (2013) as reported by Bello *et al*. (2017), only samples with high carbon content can be efficient adsorbent based on the carbon content in the removal of pollutants is in order ESAC>GSAC (Tables 1 and 2). Results show that the more effective adsorbent between those prepared is ESAC, followed by GSAC. The bulk density of a generated activated carbon plays a great role on the adsorbate uptake and the range was 370.00±0.000−380.00±0.000 m^2/g.

Table 1: Physicochemical properties and the proximate compositions groundnut shell AC

S/No	Parameters	Mean ± SE
1	pH	7.10 ± 0.101
2	Moisture content (%)	14.10 ± 0.101
3	Volatile matter (%)	9.20 ± 0.112
4	Ash content (%)	8.98 ± 0.111
5	Fixed Carbon	67.70 ± 0.010
6	Bulk density (g/L)	370.00 ± 0.000
7	**Surface area (m^2/g)**	**880.00 ± 0.100**

Table 2: Physiochemical properties and proximate compositions of egg shell AC

S/no	Parameters	Mean ± SE
1	pH	7.80 ± 0.011
2	Moisture content (%)	12.90 ± 0.110
3	Volatile matter (%)	9.90 ± 0.012
4	Ash content (%)	5.80 ± 0.111
5	Fixed carbon (%)	71.40 ± 0.001
6	Bulk density (g/L)	380.00 ± 0.000
7	Surface area (m^2/g)	800.00 ± 0.000

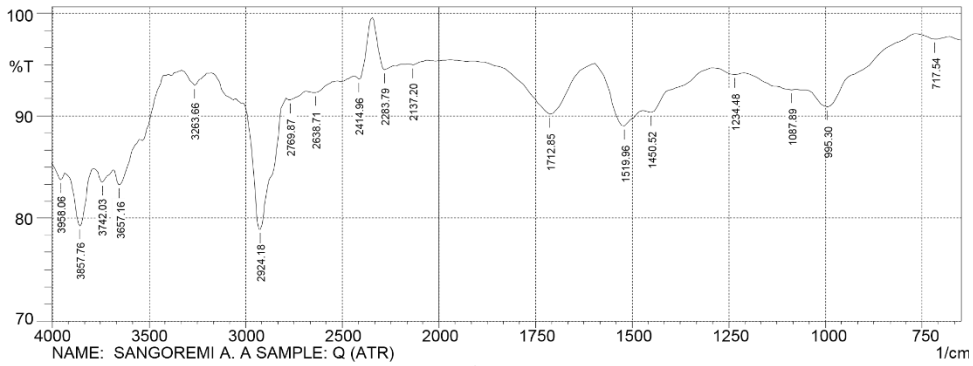

Fig. 1: Unmodified groundnut shell

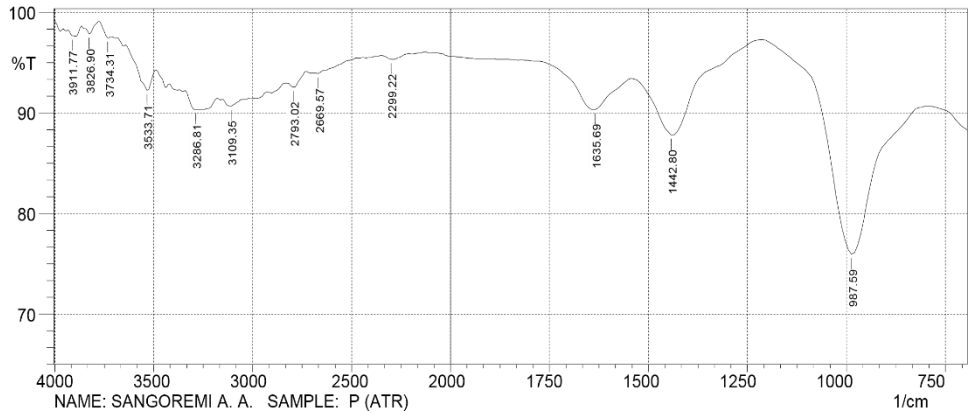

Fig. 2: Modified groundnut shell

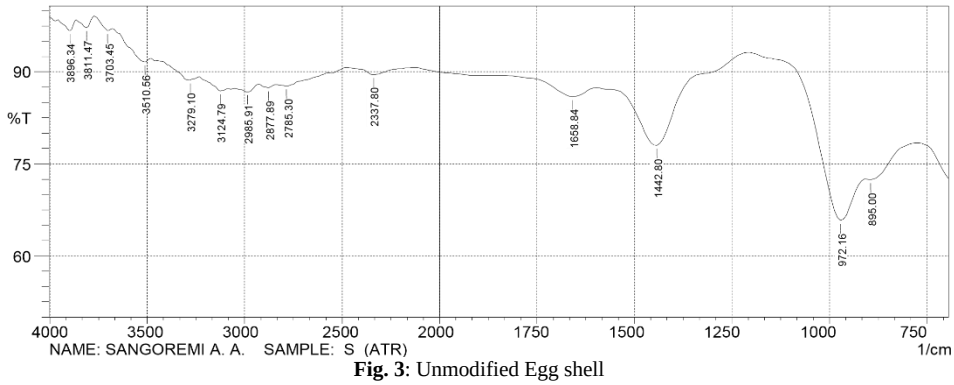

Fig. 3: Unmodified Egg shell

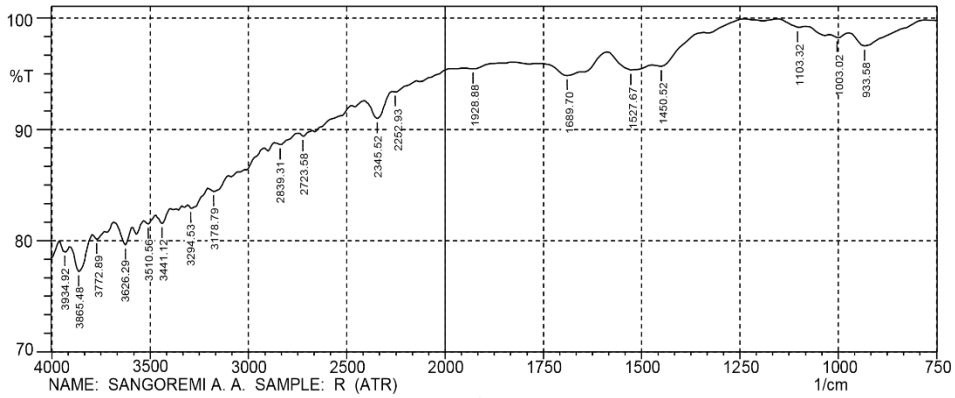

Fig. 4: Modified Egg shell

Figures 1, 2, 3 and 4 show the FTIR spectra of unmodified groundnut shell, modified groundnut shell, unmodified eggshell and modified eggshell respectively. The spectra of the samples show the presence of several functional groups. These spectra revealed a reduction, broadening, disappearance and appearance of new peaks after the process of activation. The shifts in the spectra revealed the effect of activation on these adsorbents. The prominent bands after activation are indications that the prepared adsorbent will be effective in removal of organic and inorganic pollutants. (Bello *et al*., 2017).

The FTIR spectra of modified adsorbents (figures 2 and 4), show a broad bands at 3826.90 cm^{-1}, 3865.48 cm^{-1} representing the –OH stretching of alcohol or phenol. A sharp bands observed at 3533.71 cm^{-1} and 3441.12 cm^{-1} respectively represent the –OH bonding group of alcohol or carbonyl group. The peaks around 2299.22 cm^{-1} and 2345.52 cm^{-1} for the two activated carbon correspond to C≡N stretch vibration of amines. The bands at 1928.88 and 1635.63 cm^{-1} correspond to C=O stretching acid halide. Functional groups of adsorbents not only affect the adsorption behaviour, but also dominate the adsorption mechanism (Zheng *et al.*, 2014). The peaks appearing in the FT-IR spectrum were assigned to various functional groups according to their respective wave numbers.

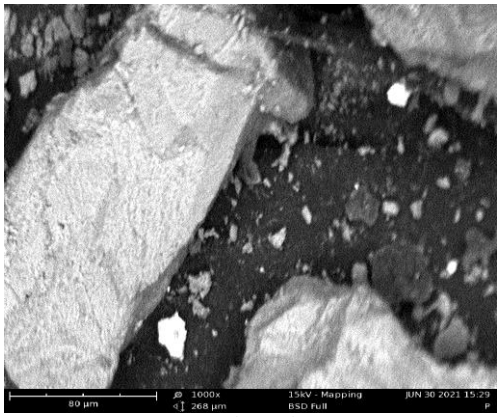

Fig. 5: SEM micrograph of modified groundnut shell

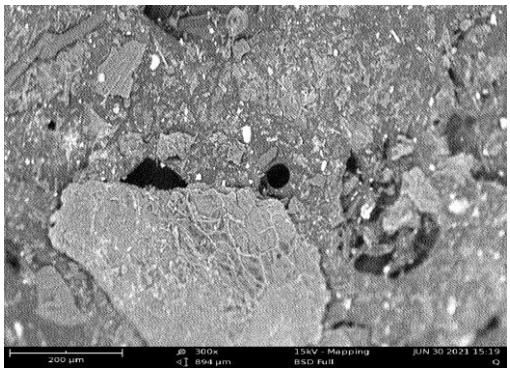

Fig. 6: SEM micrograph of unmodified groundnut shell (AC)

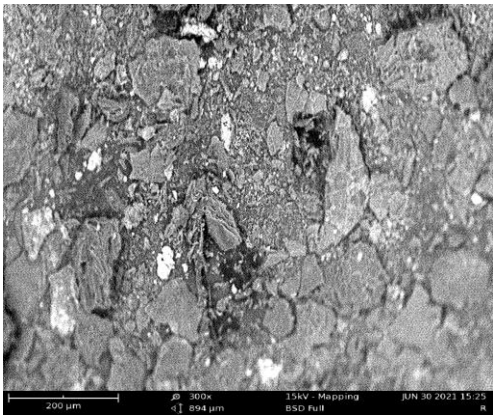

Fig. 7: SEM micrograph of modified Egg shell AC

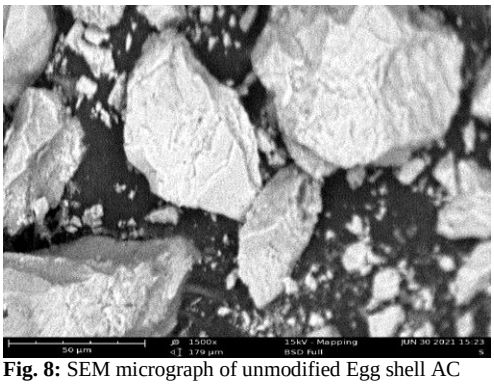

Fig. 8: SEM micrograph of unmodified Egg shell AC

Figures 5-8 revealed the Scanning electron micrographs (SEM) of modified groundnut shell activated carbon (MGSAC), unmodified groundnut shell activated carbon (UGSAC), modified eggshell activated carbon (MESAC) and unmodified eggshell activated carbon (UESAC) respectively. The surface morphology of the designated samples evidently shows that UGSAC and UESAC surface pores were properly developed, rough with fairly irregular cavities due to release of volatiles within the microstructure and several pores formed, and are distributed over surface of precursors after acid treatment as seen on MGSAC and MESAC micrographs. This is a signature that H_3SO_4 was effective in creating well-developed pores on surface of the precursors, hence, leading to active carbon with large surface area and porous exterior. This is agreement with the works of other researchers (Ajala and Ali, 2020; Bello *et al*., 2017, Bello *et al*., 2015; Abdul-Khalil *et al*., 2013; Sivakumar *et al*, 2012). The availability of pores and internal surface is requisite for effective adsorbent (Bello *et al*., 2017). Thus, the porous nature of the prepared adsorbents, helps in adsorbates uptake which will be advantageous in adsorption process. These pores will provide a good surface area for effluent treatment and remediation organically polluted sites (Ahmad *et al*, 2015a, b; Bello *et al*., 2017; Ajala and Ali, 2020).

Conclusion: Owing to the high cost of commercially available activated carbon, the alternative use of cheaper, eco-friendly and abundant agricultural wastes would be a timely intervention. Agro-wastes are readily available at little or no cost and could be a good replacement to the commercial ACs in addition to eliminating the environmental nuisance agricultural wastes constitute to the environment. The ACs produced will be good adsorption precursors in the recovery of used (waste) oils, waste water and effluent treatment.

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