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Generation and Characterization of Adsorbents Derivedfrom Unmodified Peanut and Egg Shells

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Abstract: ***Efficient adsorbents equivalent to industrially obtainable activated carbon is drawing huge attention as a proficient precursor foradsorption process. The current study is zeroed on the generation and characterization of unmodified adsorbents from peanut shell (PNS) and Eggshell (EGS) by employing standard methods (ASTM and AOAC).The physicochemical characteristics were investigated up for both PNS and EGS adsorbents, and the outcome revealed the following biomass range: moisture content (15.20 ± 0.101 to14.50 ± 0.110)%, volatile matter (11.20 ± 0.110 to10.30 ± 0.011)%, ash content (9.10 ± 0.111 to8.80 ± 0.110)%, pH (6.50 ± 0.010 to6.40 ± 0.011) fixed carbon (66.40 ± 0.010 to 64.50 ± 0.110)%, particle size (300.00 ± 0.000) µm, bulk density (1.29 ± 0.000 to 0.540 ± 0.000)g/cm^3, and surface area (750.00 ± 0.000 to 680.00 ± 0.100)m^2/g.Scanning electron microscope (SEM), and Fourier transform infrared spectrometer (FT-IR) were utilized to study the surface morphology and functional groups accordingly. The Electron dispersive X-ray spectrometer uncovered the elemental components of the adsorbents. The adsorbents had high fixed carbon contentsandlow inorganic, in conjunctionwith high surface area, making them valuable adsorbents. The FTIR examinationrevealedthepresence of functional groups such asNH, C≡C,C=O, C-H and OH which are potential adsorption sitesin addition to the well-developedpore structures from SEM studies.In contrast, EDX uncovered the presence of components like carbon, oxygen, calcium, magnesium and silicon in percent weightswith PNS and EGS having carbon contents of 91.12 and 40.11% respectively. Generally, the study adsorbents possess the potential to be efficientand eco-friendly precursors fortheadsorption process.***

Keywords: Adsorbent, adsorption, activated carbon, biomass, FTIR, precursor,

I. INTRODUCTION

Agricultural wastes had been utilized effectively in current waste treatment plants for water filtration and detoxification treatment of polluted waters (Jacob *et al*., 2017; Ajala and Ali, 2020), and effluent including waste treatment (Ajayi-Banji *et al*., 2015; Marichelvan and Azhagurajam, 2018).Wastes produced from agro-wastes are numerous, they createirritation to the place where they are found. Agro-wastes are generally in enormous amount, a portion of these wastes are known for their hostile smell,and their rotted mattercan modify soil pH (Bello *et al*., 2017). Lately, different investigations have reported the preparation ofadsorbents from the agro-wastes as a substitutefor commercial activated carbons (ACs), which are expensive. Agro-waste materials have been proposed as conservative and eco-accommodating optionsforACs (Oladoja*et al*., 2014; Abdullahi *et al*., 2022).The peanut shell (PNS) also known as groundnut shell, is the protective outer covering of the peanut (PN). It is a thin, hard, and fibrous materialsurroundingtheedible peanut kernel (PNK).The PN is typically oval or elongated in shape and varies in colour from light tan to dark brown, depending on the variety of PN. It consists of two main parts: the outer shell and the inner seed coat. (Onawumi*et al*., 2021). The outer shell is tougher and more fibrous ~~layer~~, while the seed coat is thinner and smoother layer that directly encloses the PNK. The shell serves as a natural protective barrier for the PNK, shielding it from environmental factors, pests and diseases. It helps to maintain the freshness and quality of the kernel inside. However, the shell itself is not consumed by humans as it is generally considered inedible due to its tough and fibrous nature (Ajala and Ali, 2020). The PNS have various uses beyond their roles in protecting the peanuts (PNs). They are commonly used as a good source of biomass fuel; they can be used as animal feed or as a component in livestock bedding material. In some cases, the shells are utilized for industrial purposes such as production of particle boards, mulch, and compost. PNS can also be used as an adsorbent intheadsorption phenomenon in the removal of dyes, heavy metals and other impurities from wastewater, andeffluents including remediation of waste cooking oil (Onawumi*et al*., 2021). Further, aneggshell (EGS) is the outer covering of an egg, which is usually thin, fragile, and bristle. It is primarily composed of calcium carbonate, along with small amounts of protein and other minerals (Onawumi*et al*., 2021). The colour of an EGS varies, depending on the species of bird that laid it, but it is commonly white or off-white (Ahmed *et al*., 2021).The EGS serves as a protective barrier that encases the egg's content, providing support and shielding the delicate internal structure.

Despite its delicate appearance, the EGs isquite strong and capable of withstanding the weight of an incubating bird sitting on it (Ahmed *et al*., 2019a). However, it is still susceptible to cracking or breaking under pressure (Onawumi*et al*., 2021). The surface of an EGS is covered with tiny pores that allow for the exchange of gases. These pores enable the developing embryos inside the egg to breathe by allowing oxygen to enter and carbon dioxidetoexit. In terms of texture, the outer surface of an EGS can feel smooth, but it may also have a slight roughness or a grainy texture (Onawumi*et al*., 2021). The texture can vary depending on the species of bird and individuals. Eggshells (EGSs) have been utilized for various purposes beyond their role in protecting the developing embryos. Theyare sometimes crushed into a fine powder and used as a calcium supplement or fertilizer. EGSs have also been employed in art and craft, as well as in traditional remedies and folk practices (Ahmed *et al*., 2021). Finally, EGSs have been employed in various environmental remediation technologies such as theremoval of heavy metals, adsorption of radioactive meals, andadsorption of total nitrogen,fluoride and phosphorus from wastewater (Lu et al., 2017; Ahmed *et al*., 2021; Onawumi*et al*., 2021).

II. MATERIALS AND METHODS

A. Agro-waste samples procurement

PNS samples wereobtainedfrom a farm settlement at Idi-Osan, Iragbiji, Boripe L.G.A, while the EGS were collectedfrom an eatery at Ogbonna region, Osogbo, Osun State, Nigeria. The samples were placed in polythene packs and brought to the herbarium for proof of identity at theLife Sciences research facility of FUOtuoke, Bayelsa State, Nigeria (Sangoremi*et al*., 2024).

B. Sample preparation

The procedureofOnawumi*et al*. (2021) wasused.The samples were washed with steady water in the laboratory, and washed severally with distilled water to remove stones, dirt and trash. The samples were sun-dried for 24 hr and oven-dried at 105^{o}C for 5 hr and allowed to cool in desiccators. The dried samples were squashed and sieved to 300 µm size with amechanical sifter and kept in a sealed glass container beforeusage.

C. Adsorbent Characterization

1) pH determination

Exactly 3 g of the adsorbent was measured and soakedin 30 ml of hot distilled water for 24 hr. The combination was mixed to guarantee good dilution and filtered. The pH of the filtrate was determined using a computerized pH meter, Jenway 3520 (Ebelegi*et a*l., 2022; Onawumi*et al*., 2021; ASTM: D 3838).

2) Determination of moisture content

The moisture content (MC) wasdetermined by standard techniques ASTM D 2974 (2014). About2 g of the models was measured into a crucible. This was dried at 105 ^{o}C to consistent mass and kept in a desiccator. The percentage MC was determined mathematically (Boadu *et al*., 2018)using the relationship below**:**

$$MC = \frac{C - D}{C - B} x100 \qquad 1$$

D = Mass of crucible in addition to dried example
B = Mass of crucible (g)
C = Mass of crucible in addition to initial example (g)

3) Bulk density

The common procedure used in calculating the bulk density (BD)was weighing 5 g of the sample and placed on a pre-weighed 5 ml measuring cylinder (w_1). The cylinders were gently tapped to eliminate air spaces inside the samples in the cylinder to give a potential close pack (PBD). The volume required by the samples and the additional load of the cylinder wererecorded using a weighing balance as (w_2), (Ijaola*et al.*2013, and Ebelegi*et al*. (2022).The mass BD was determined as:

$$BD = \frac{W2 - W1}{V} \qquad 2$$

W1 = mass of cylinder (g)
V = size of the cylinder
W2 = mass of tests and cylinder (g)

4) Ash content profile

Ash content (AC) was calculated as per standard technique (Ebelegi*et al.*, 2022; ASTM: D 2866-94). Exactly 5 g of dried samples were measured into a crucible of a known weight and heated in a muffle furnace for 6 hr at 600 ^{o}C. Precisely when consistent weight was accomplished, the crucible wasallowedto cool in desiccators. The mass of the ashed carbon was determined as the percentage of the initial sample.

$$AC = \frac{C - D}{C - B} x100 \qquad 3$$

D = Mass of crucible + ashed sample (g)
C = Mass of crucible + initial sample (g)
B = Mass of the crucible (g)

5) Volatile matter

Volatile matter (VM) was determinedby the standard techniques (ASTM D3175-11 and Ebelegi*et al*. 2022). Exactly 1g ofthe samplewastaken in a pre-dried crucible and covered with a lid,and placed in a muffle furnace controlled at 950^{o}C for 7 min. During heating, the crucible was promptly covered, cooled in desiccators and weighed. The sum weighed was taken as VM.

$$VM = \frac{C - D}{C - B} x100 \,(4)$$

D = Mass of the crucible + volatile ample (g)
C = Mass of crucible + test sample (g)
B = Mass of the crucible (g)

6) Particle size

The particle size (PS) of the ground samples were examined. The samples were made by mean of an electric blending machine after which a sifter examination was done using Controls Miland-Italy D402-01 Matr 84000 109 sieve shaker at a rotation of 10-15 min with (300 µm) sieve.

$$\%C = \frac{A}{B} \,(5)$$

A = Mass of carbon after sieve
B = Total mass of carbon

7) Surface Area

The surface area (SA) of the biosorbent was determinedusing the Sae's technique, (Ebelegi*et al.*,2022) where 0.5 g of each sample was carefully weighed into 250 ml Erlenmeyer flaskcontaining 25 ml of 0.1M HCl at pH 3.50. Thereafter, 1 g of NaCl wasadded to increase the pH to 4, andthe mixture was titrated against a standard solution of 0.1M NaOH until pH 9 was achieved.
The volume expected to extend the pH from 4 to 9 was noted and applied in working out the SA utilizing Eq. (6).
SA (m2/g) = 32V− 25 (6)
V = volume of NaOH used to increase pH from 4 to 9. The surface area of the biosorbent was resolved by using the Sae's technique, portrayed by Ebelegi*et al.* (2022) where 0.5 g of each sample was carefully weighed into 250 ml erlenmeyer flask holding 25 ml of 0.1M HCl at pH 3.50, after which 1 g of NaCl was enhanced to increase the pH to 4, the mixture was titrated against a standard solution of 0.1M NaOH until pH 9 was achieved.
The volume expected to extend the pH from 4 to 9 was noted and applied in working out the SA utilizing Eq. (6).
SA (m2/g) = 32V− 25 (6)
V = volume of NaOH used to increase pH from 4 to 9.

Scanning electron microscopy/ energy dispersive X-ray spectroscopy (SEM/EDX/) analysis

The morphology of the samples and elemental configuration of PNS and EGS wereobtainedthrough SEM using a JSM-7610F (Tokyo, Japan). The hardware is a super-high goal Schotty Field emanation filtering electron microscopy joined with energy dispersive x-ray. The adsorbent surface was inspected with an amplifying lens worked at 10.0 kV. The samples were covered with a 10 nm thick layer of gold (Jabar *et al.*, 2020).

III. RESULTS AND DISCUSSION

A. Adsorbent pH

Scientific investigationsshowed that pH impacts the charge bulkiness around adsorbent/adsorbent particles (Ebelegi*et al.*, 2022; Sangoremi*et al*., 2024). Subsequently, pH is a significant parameter that impacts the dissociation of active sites on the surface of adsorbents (Onawumi*et al*., 2021). Tables 2 and 3 reveal that PNS and EGS have pH (6.50 0.010 and 6.40 0.011), and may be great adsorbent for anionic species. The pHfor the prepared biomasses iswithin the range of those stated in the literature (Ajala and Ali, 2020) which suggests that the biomasswas ideal for adsorption. The pH fortheideal take-up of metals and organic pollutants by most adsorbentsisbetween pH 6.0-9.0 (Aji *et al*., 2015; Sangoremi*et al.*, 2024).

B. Moisture Content (MC)

Research shows that moisture content (MC) increases directly with BD (Ebelegi*et al*., 2022). Tables 2 and 3 reveal the range of MC (15.20 ± 0.1010 to 14.50 ± 0.110%) which suggests that comparable to MC, EGS has anideal adsorption efficiency over the PNS. Furthermore, adsorbents with low MC have a longer shelf-life usability than the ones with high MC. Consequently, biosorbents with high MC ought to be additionally exposed to gentle heating before they could be utilized as this would have diminished the MC, and subsequently upgraded a superior adsorption ability (Ebelegi*et al.*, 2022).

C. Volatile Matter

The VM amount is shown in Tables 2 and 3 in the scope of (11.20 ± 0.110 to 10.30 ± 0.011%) for both PNS and EGS which was extensively high, conceivably because of the adsorbent source.

D. Ash Content

The outcome of AC uncovered that PNS was 9.10 ± 0.111%, and EGS (8.80 ± 0.110%). Reports have it that elevated levels of AC diminish the overall effectiveness of adsorbents, subsequently, it decreases the efficiency of the adsorbents, as far as adsorbent regeneration is concerned. Accordingly, the outcomes acquired revealed the presence of an obvious level of ash in the adsorbents, and this could prevent their surface reactivity (Ebelegi*et al*., 2022). Be that as it may, researchers have detailed AC values comparable to what was gotten in the current works (Boadu *et al.*, 2018).

E. Fixed Carbon

Tables 2 and 3 reveal that the values of FC for PNS and EGS are 64.50 ± 0.110 and 66.40 ± 0.010% respectively. The higher the FC, the better the adsorption capacity of the adsorbent, consequently, EGS would be a preferable adsorbent over PNS because of its higher FC. Reports have it that a decent adsorbent ought to have a FC ≥ 65% (Olayiwola *et al*., 2015). The FC values EGS adsorbent in the current work fall within the range of adsorbents as stated in the literature, while PNS was 0.5% lesser, by and large, the adsorbents synthesized are suitable for the adsorption process.

F. Bulk Density

Tables 2 and 3 presented the results of BD for PNS (0.540 ± 0.000), and EGS (1.29 ± 0.000 g/cm3). It is observed that EGS has a higher BD while PNS has the least. In this way, the BD values got for the adsorbents conformed to the values widely reported in the literature, making them suitable for adsorption (Ajala and Ali, 2020). Be that as it may, since BD is inversely correlated to the surface area, (SA) (BD α 1/SA), subsequently, the adsorbent with lower BD will have better adsorption efficiency. Hence, PNS shows improvement over EGS based on BD.

G. Surface Area

Tables 2 and 3 presented the surface area (SA) of 680.00 ± 0.100 m^2/g for PNS, while EGS has 750.00 ± 0.000 m^2/g. Consequently, EGS portrays a superior adsorbent in view of its higher SA which means the accessibility of more empty adsorption sites than PNS. Reports have that activation advances better porosity and high surface area (Onawumi*et al.*, 2021), and adsorbents with SA of 500-1500 m^2/g are great for adsorption.

H. Particle Size

Particle size (PS) has a close relationship with SA. Decreased PS has direct connections to the progression of greater SA, enhancing more adsorption ofempty sites within the adsorbents (Ebelegi*et al*., 2022).

The worth of PS (300.00 ± 0.000 µm), and other physicochemical properties of the adsorbents aligned to those detailed by different authors (Ajala and Ali, 2020; Sangoremi*et al.*, 2024).

Table 1: Standard threshold s for ACs

Parameter	SII No. 02587-79	SNI 06-3730-1995
Fixed Carbon (%)	Max -	Max 65
Volatile matter (%)	Max 15	Max 25
Ash content (%)	Max 2.5	Max 10
Iodine (mg/g)	Min 200	Min 750
Moisture (%)	Max 10	Max 15

Min: minimum, Max: maximu

Table 2: Physicochemical properties of Peanut shell (PNS)

S/no	Parameters	$\bar{x}$ ± SE
1	Moisture content (%)	15.20 ± 0.101
2	Volatile matter (%)	11.20 ± 0.110
3	Ash content (%)	9.10 ± 0.111
4	pH	6.50 ± 0.010
5	Fixed carbon (%)	64.50 ± 0.110
6	Particle size (µm)	300.00 ± 0.000
7	Bulk density (g/cm^3)	0.540 ± 0.000
8	Surface area (m^2/g)	680.00 ± 0.100

Table 3: Physicochemical properties of eggshell a(EGS)

S.N	Parameters	$\bar{x}$ ± SE
1	Moisture (%)	14.50 ± 0.110
2	Volatile matter (%)	10.30 ± 0.011
3	Ash content (%)	8.80 ± 0.110
4	pH	6.40 ± 0.011
5	Fixed carbon (%)	66.40 ± 0.010
6	Particle size (µm)	300.00 ± 0.000
7	Bulk density (g/cm^3)	1.29 ± 0.000
8	Surface area (m^2/g)	750.00 ± 0.000

Fourier transform infrared spectroscopy (FT-IR) analysis

Figures 1 and 2 showed the spectra of PNS and EGS, while Tables 4 and 5 revealed the functional configuration. The spectra uncovered functional groups that are probable sites for adsorption. For both PNS and EGS adsorbents, the bands representing the peaks were seen at 3657.76, 3657.16, 2924.18, 1712.85, 1519.96, 995.30 cm-1, and 3896.34, 3811.47, 3703.15, 3510.56, 3124.79, 2337.80, 1658.84, 1442.52, 972.16 cm-1 separately. The peaks at 3896.34, 3811.47 and 3703.15 cm-1 represent the stretching of OH, and the peaks at 3510.56, 3124.79, 2337.80, 1658.84, 1442.52, 972.16 connote N-H, C-H, C≡C, C=O, CH_3, C-H stretch separately in the EGS adsorbent. The bands at 3657.76 and 3657.16 cm-1 address the N-H extending, while the peaks at 2924.18, 1712.85, 1519.96 and 995.30 cm-1 signify CH, C=O, C=C and NH2 extending separately in PNS adsorbent. The adsorbents' functional groups not only influence the adsorption process, but in addition, govern the adsorption phenomenon.

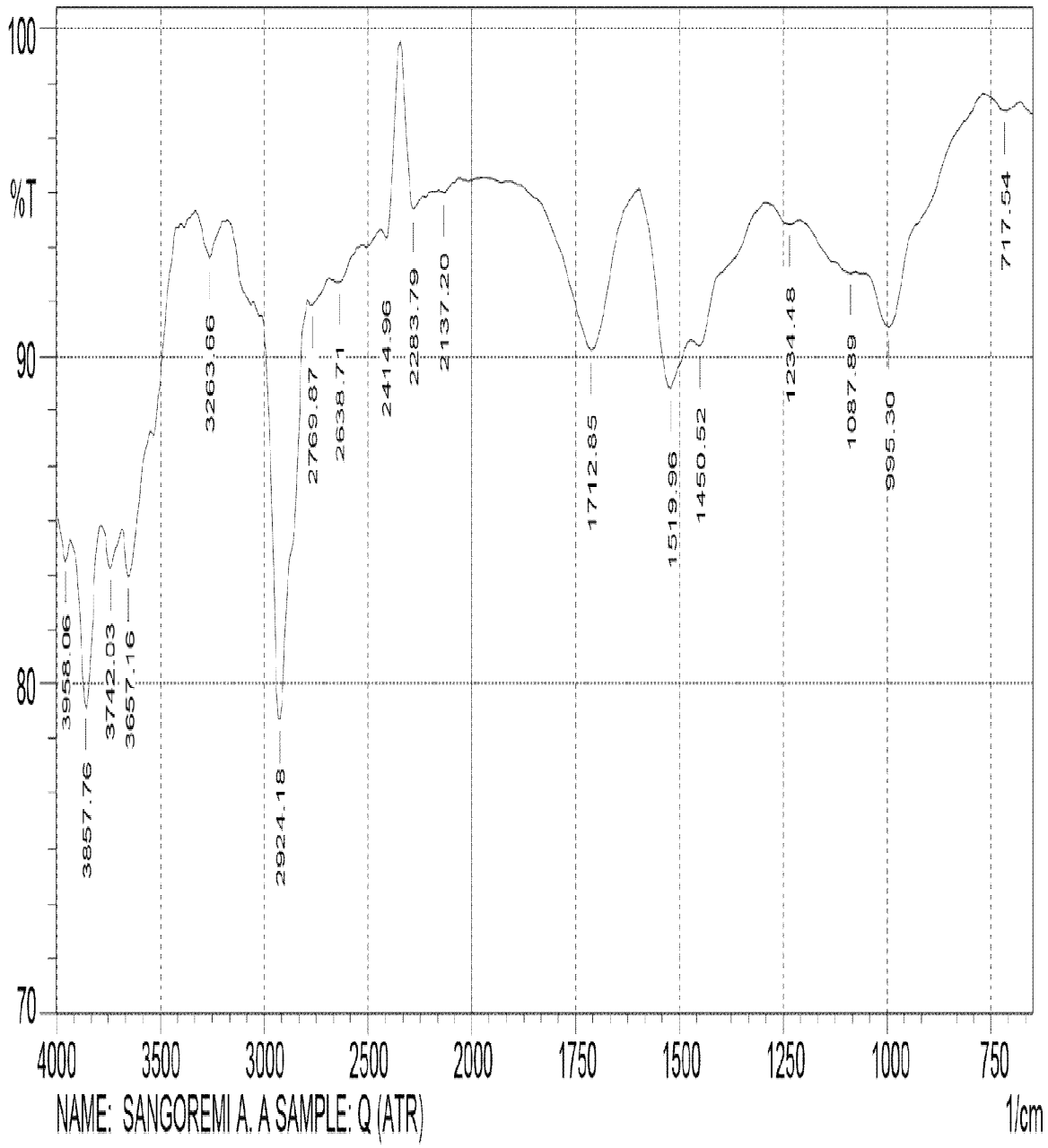

Figure 1: FTIR Spectrum of peanut (PNS)

Table 4: The FTIR spectrum of peanut shell

S. N	Wave number (cm^{-1})	Bands (cm^{-1})	Functional group
1	3657.76	> 3500	N-H stretch
2	3657.16	> 3500	N-H stretch
3	2924.18	2960 - 2700	CH stretch
4	1712.85	1730-1700 (s)	C=O stretch
5	1519.96	1550-1475	C=C stretch
6	1450.52	1510-1450	CH_3or CH_2
7	995.30	1000-665 (s)	NH_2

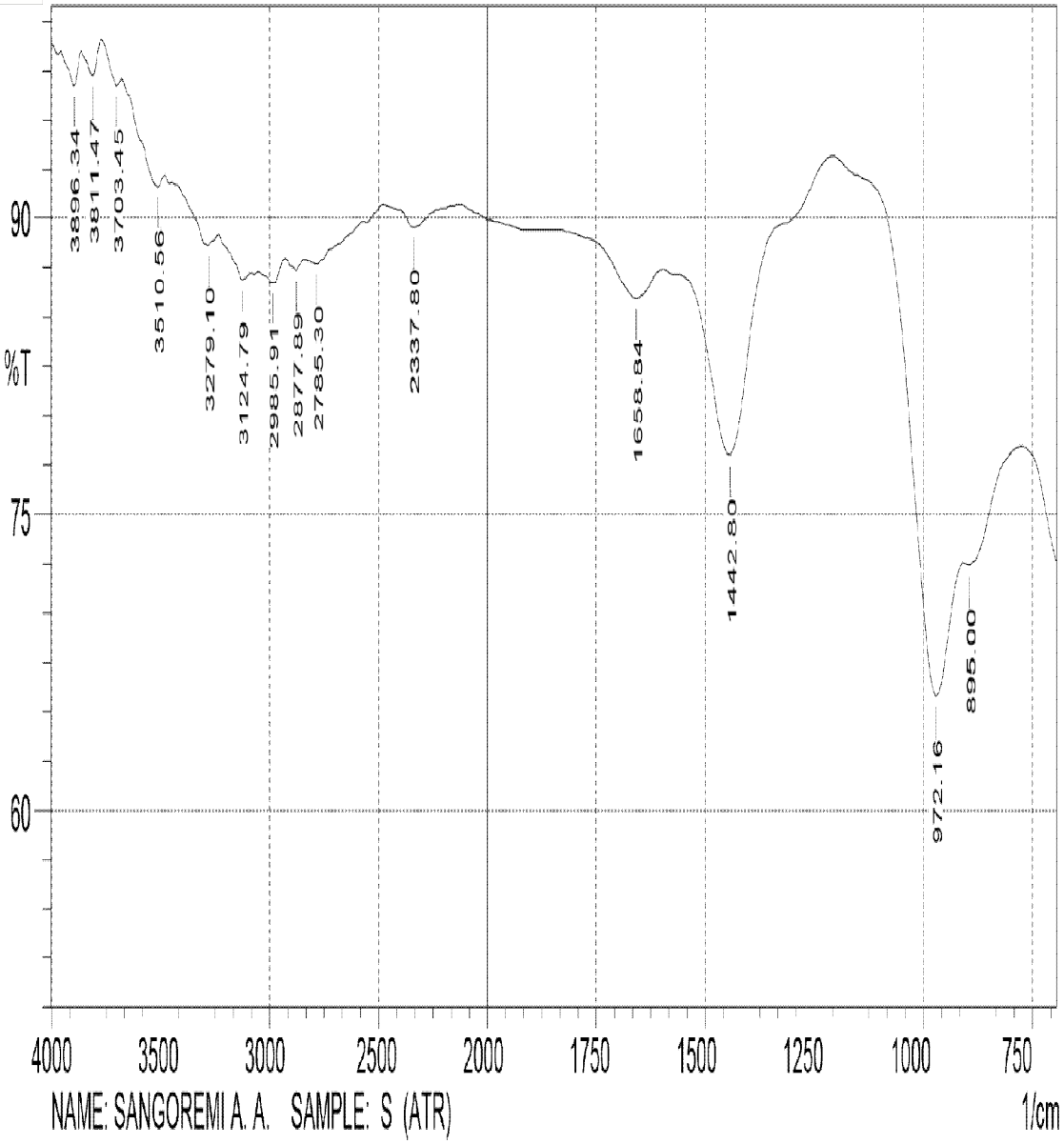

Figure 2: FTIR Spectrum of eggshell (EGS)

Table 5: FTIR spectrum interpretationsof eggshell adsorbent

S. No	Wave number (cm^{-1})	Frequency (cm^{-1})	Functional group
1	3896.34	> 3500	O-H
2	3811.47	> 3500	O-H
3	3703.15	> 3500	O-H
4	3510.56	> 3500	NH stretch
5	3279.10	3300 - 2500	NH
6	3124.79	3300 - 2500	C-H
7	2337.80	2376 - 2248	C≡C
8	1658.84	1680 - 1620	C=O stretch
9	1442.52	1510 - 1450	CH_3 (methyl group)
10	972.16	1000 665 (s)	C-H out of plane bending vibration.

Scanning Electron Micrographs (SEM)

Figures 3 and 4 showed the Scanning electron micrographs (SEM) of PNS and EGS adsorbents individually. The surface morphology of the samples clearly shows that the surface pores were well developed**,** andsmooth with regular pore structures which are prerequisites to good adsorption efficiency. Other researchers made similar reports (Ebelegi*et al*., 2022). The accessibility of pores and internal surfaces is fundamental for successful adsorption. In this way, the porous properties of the synthesized biomassassist in the adsorption process. These pores give a decent SA forimpurities removal, and remediation of waste oils (Ebelegi*et al*., 2022).

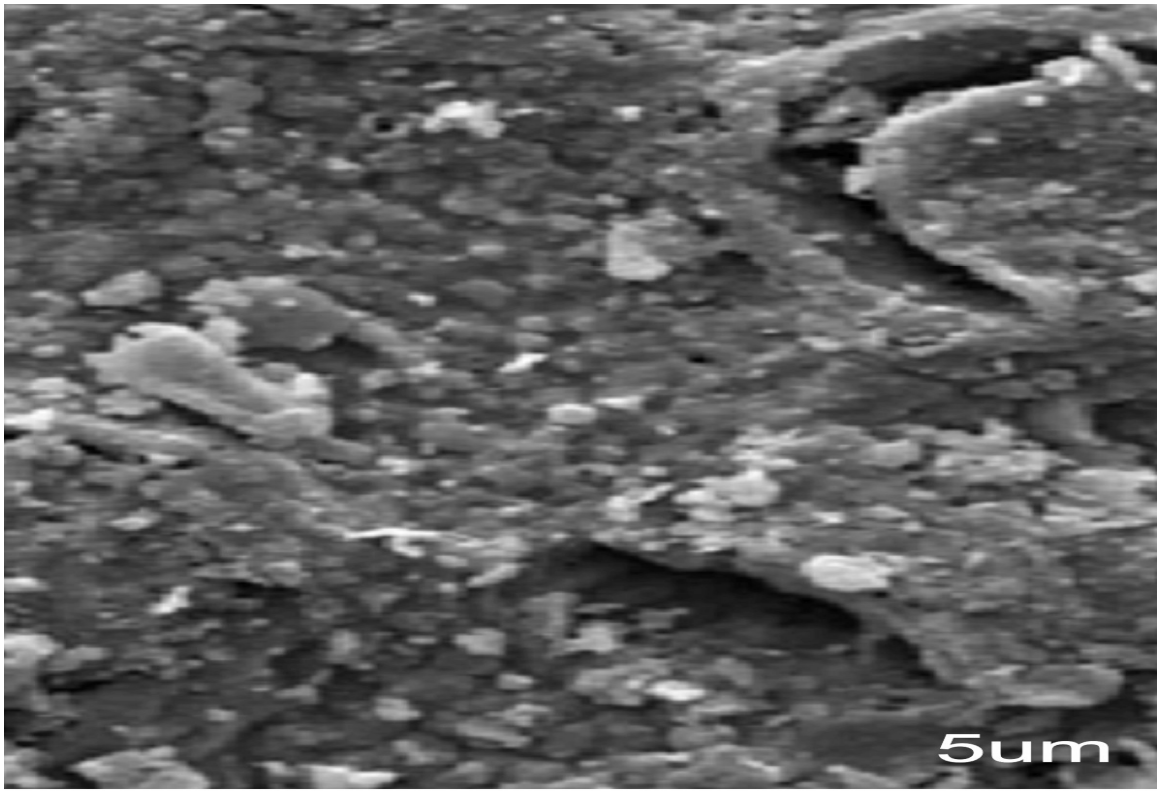

Figure 3: SEM micrograph peanut shell adsorbent

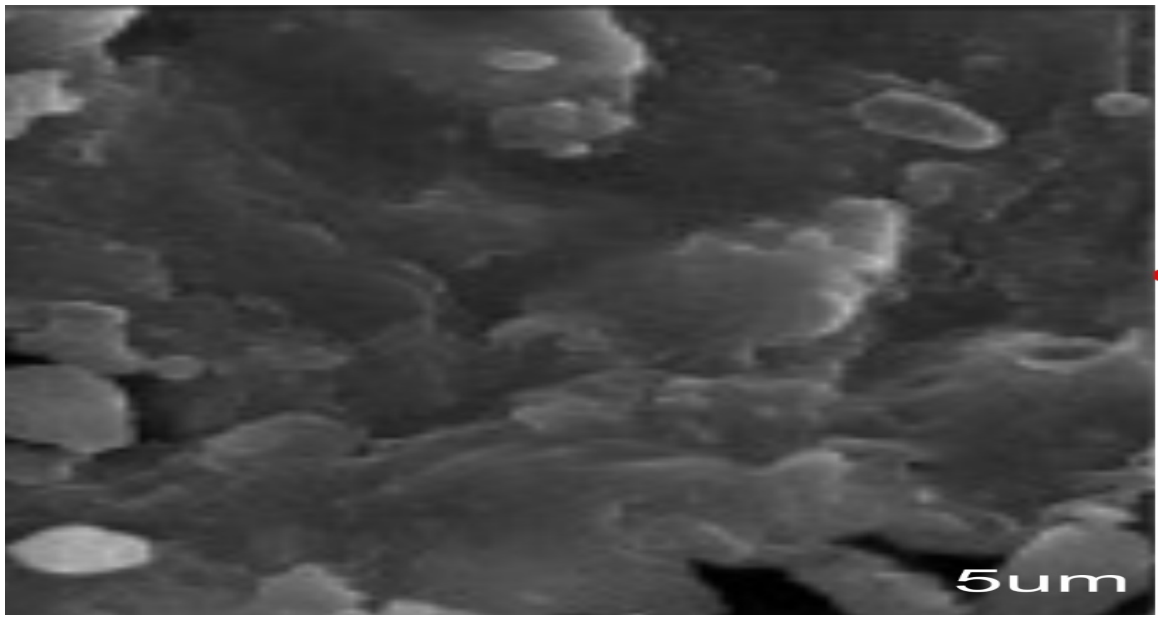

Figure 4: SEM micrograph eggshell adsorbent

Electron Dispersive X-ray Spectroscopy (EDX)

The EDX study is a technique for elemental examination in Figures 5 & 6 showed the EDX spectra of PNS and EGS adsorbents, while Tables 6 and 7 showed the elemental configuration of the adsorbents individually. The carbon contents for PNS and EGS are 91.12 and 45.41% respectively.Additional elements present according to percent atomic weight comprise: Mg (0.55%),O (42.92%), Ca (1.28%), N (5.64%),P (1.63, 1.28%) (Ushedo*et al*., 2022).

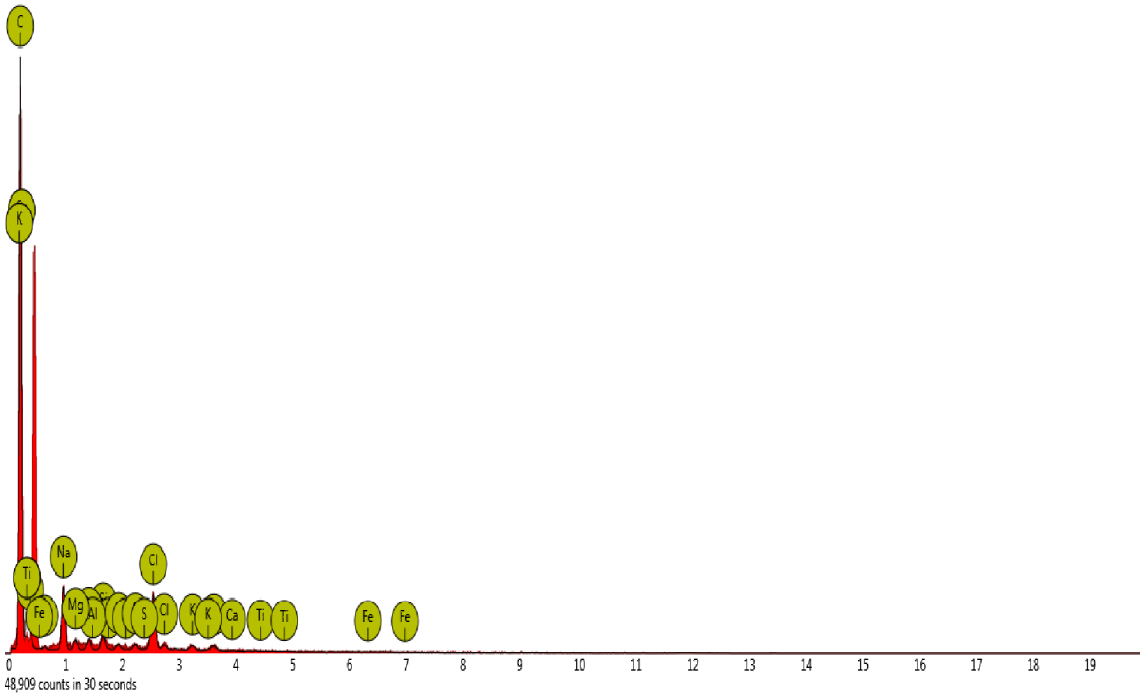

Figure 5: EDX adsorption spectrum of peanut shell

Table 6: Elementalcompositionof peanut shell

Element	Symbol	Name	Atomic Conc.	Weight Conc.
6	C	Carbon	91.12	79.15
15	P	Phosphorus	4.66	10.44
19	K	Potassium	1.63	4.60
20	Ca	Calcium	0.77	2.23
14	Si	Silicon	0.79	1.61
11	Na	Sodium	0.30	0.50
16	S	Sulfur	0.11	0.25
17	Cl	Chlorine	0.11	0.28
13	Al	Aluminum	0.23	0.46
12	Mg	Magnesium	0.28	0.49
22	Ti	Titanium	0.00	0.00
26	Fe	Iron	0.00	0.00

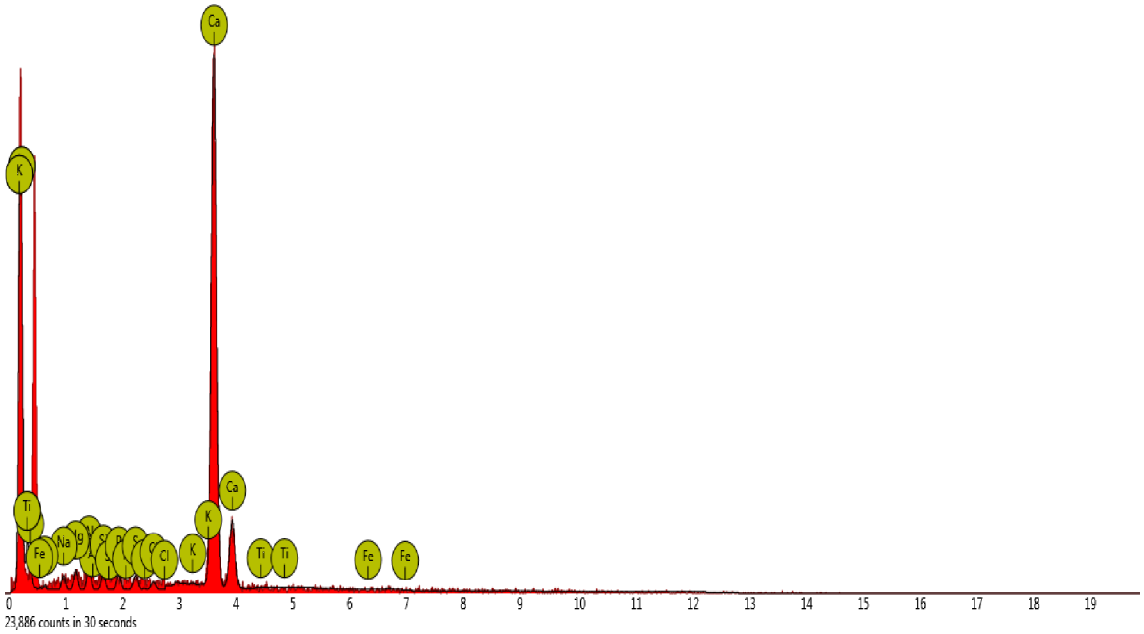

Figure 6: EDX adsorption spectrum of eggshell

Table 7: Elementalcompositionof eggshell

Element	Symbol	Name	Atomic Conc.	Weight Conc.
20	Ca	Calcium	45.41	44.66
6	C	Carbon	40.11	39.67
19	K	Potassium	5.28	4.09
13	Al	Aluminum	2.19	2.00
12	Mg	Magnesium	2.00	1.91
11	Na	Sodium	2.00	1.69
16	S	Sulfur	1.25	1.21
15	P	Phosphorus	1.28	1.20
17	Cl	Chlorine	0.38	0.33
14	Si	Silicon	1.33	1.20
26	Fe	Iron	0.00	0.00
22	Ti	Titanium	0.00	0.00

IV. CONCLUSION

The adsorbents generated, PNS, and EGS focusedin this work supposedly had moderate MC, and optimal pH alongside high fixed carbon including high SA that are pointers to outstanding adsorbents. The FTIR showed the presence of functional groups (COOH,OH,C=O, NH) that are plausible adsorption sites. The SEM results revealed the surface structure of the adsorbents (PNS and EGS)to possess the incredible features of an ideal adsorbent. The general features showed that the prepared adsorbents could be viewed as outstanding and effective precursors for the adsorption process.

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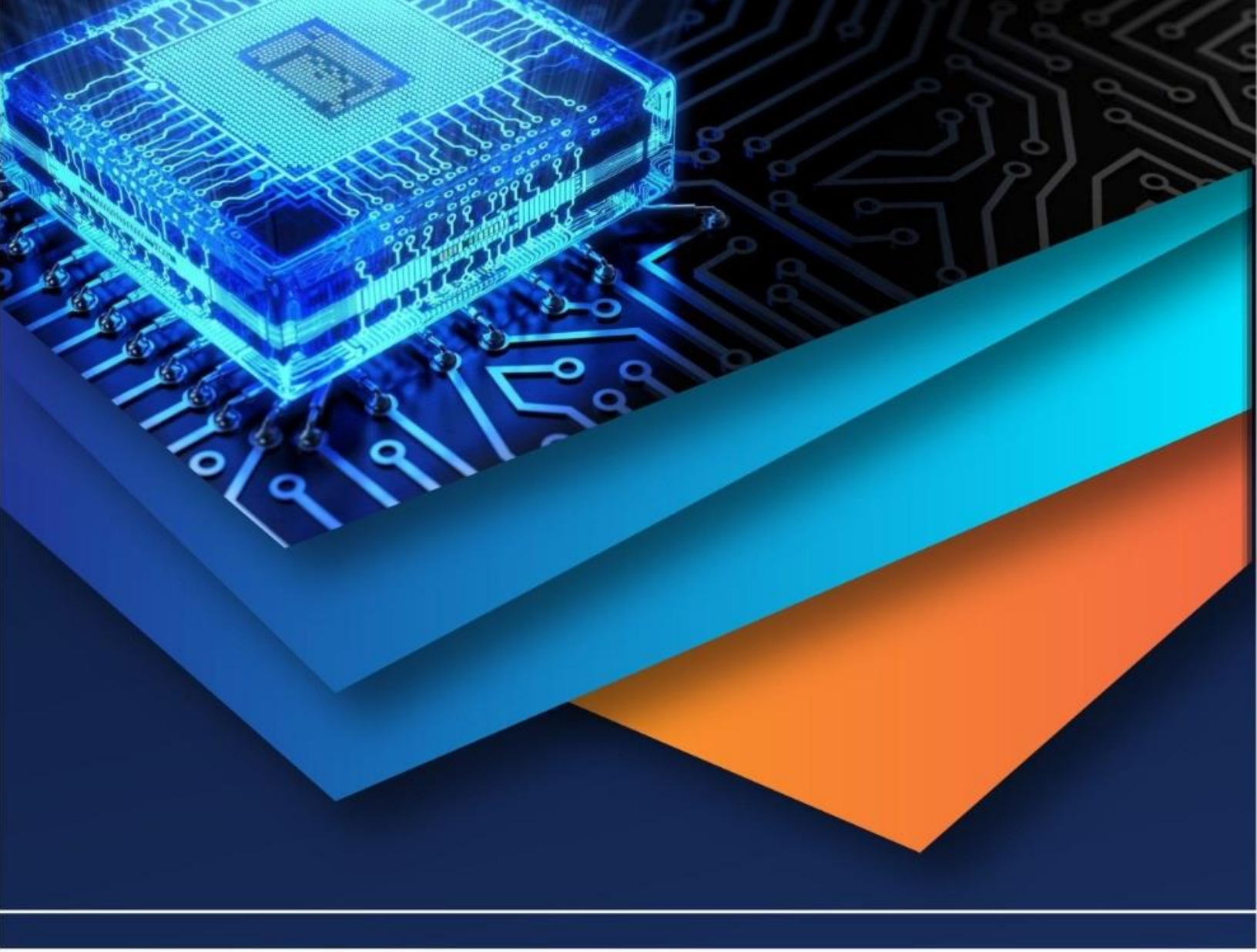

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