



## Modification and Characterization of Biosorbent Developed from Coconut Shell

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### Article Information

Article # 10012

Received: 25<sup>th</sup> Sept. 2023

1<sup>st</sup> Revision: 14<sup>th</sup> Jan. 2024

2<sup>nd</sup> Revision: 16<sup>th</sup> Feb. 2024

Acceptance: 5<sup>th</sup> April 2024

Available online:

10<sup>th</sup> April 2024.

### Key Words

Physicochemical,  
Activated carbon,  
Modification,  
Surface area,  
Bulk density,

### Abstract

The need to develop a more efficient adsorbent comparable to commercially available activated carbon is attracting significant interest as a promising precursor for the adsorption phenomenon. The study focused on the characterization and modification of adsorbent generated from coconut shell (CNSA) using standard methods (ASTM). The physicochemical and proximate compositions of CNSA were determined for both unmodified (UCNSA) and modified (MCNSA) adsorbents such as pH, Moisture content (MC), ash content (AC), volatile matter (VM), fixed carbon (FC), surface area (SA), bulk density (BD), and particle size. Fourier transform infrared spectrometer (FT-IR) and scanning electron microscope (SEM) were used to determine the surface functional groups and surface morphology respectively. The results revealed the following range: pH ( $7.10 \pm 0.101 - 6.60 \pm 0.110\%$ ), MC ( $6.20 \pm 0.100 - 3.50 \pm 0.110\%$ ), VM ( $10.00 \pm 0.011 - 9.00 \pm 0.012\%$ ), AC ( $16.42 \pm 0.111 - 15.10 \pm 0.110\%$ ), FC ( $71.08 \pm 0.001 - 68.70 \pm 0.01\%$ ), BD ( $0.769 \pm 0.000 - 0.720 \pm 0.000 \text{ g/cm}^3$ ), SA ( $1120.00 \pm 0.000 - 1100.000 \text{ m}^2/\text{g}$ ), and PS ( $300.00 \pm 0.000 - 300.00 \pm 0.000 \text{ }\mu\text{m}$ ). The biosorbents possessed high fixed carbon contents with low inorganic, in addition to large surface area which make them viable adsorbents. The FTIR analysis revealed the presence of oxygen surface complexes such as carbonyls and OH groups which are potential adsorption sites with concomitant good pore structures from SEM studies, while EDX revealed the presence of elements like carbon, oxygen, calcium, magnesium and silicon in percent weights. Overall, the study proposes both UCNSA and MCNSA as efficient, low-cost and eco-friendly biosorbents for the purification of wastewater, and industrial effluents together with waste cooking oil regeneration.

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### Introduction

Generation and disposal of waste have been a major concern globally and many studies have validated these assertions (Babayemi, 2017; Abdel-Shafy and Mansour, 2018; Loizides *et al.*, 2019; Ferronato, 2019; Biswas *et al.*, 2020; Pandit *et al.*, 2021; Kondoh, *et al.*, 2021; Abdullahi *et al.*, 2022). Most of these wastes are generated from different sources, such as agriculture and/or industry, regenerating, recovering, and recycling these wastes to useful material would be of huge benefit to man, and his environment and generate income to the society at large (Onawumi *et al.*, 2017; Boadu *et al.*, 2018; Biswas *et al.*, 2020, Ushedo *et al.*, 2022; Abdullahi *et al.*, 2022). Wastes generated from the agricultural sector are many, they constitute a nuisance to the immediate environment where they are found, agricultural wastes are usually in large quantities, some of these wastes are known for their offensive odour, and their decayed matter have

the ability to alter soil pH (Bello *et al.*, 2017). Recently, different research works have been carried out, to prepare adsorbents from these agricultural wastes used as an alternative to commercially available activated carbons, which are expensive. Presently, agricultural waste materials have been proposed as economic and eco-friendly (Kumar and Kumar, 2014; Oladoja *et al.*, 2014; Abdullahi *et al.*, 2022). These protect the environment by getting rid of the danger such wastes pose to the environment, most especially where disposal of agricultural wastes has become a major problem (Bello *et al.*, 2017). There are different types of adsorbents derived from various forms of biomass which 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 groundnut and egg shells (Onawumi *et al.*, 2021), bagasse (Ali and Anany,

2014; Ezeonuegbu *et al.*, 2021), rice husk (Siqueira *et al.*, 2020; 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 material with a large surface area (500-1500 m<sup>2</sup>/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 (Onawumi *et al.*, 2021; Mansour *et al.*, 2020; Wan *et al.*, 2010; Hidayat and Sutrisno, 2017). However, the performance properties of biomass-activated carbon (AC) depend largely on raw material source (Sivakumar *et al.*, 2012). Adsorption of pollutants using agro-waste 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 (Onawumi *et al.*, 2021; Mansour *et al.*, 2020; Amirza *et al.*, 2017; Reza *et al.*, 2020; Abdullahi *et al.*, 2022). The Adsorption process has been proven to be one of the best pollutant treatment technologies globally, activated carbon is undoubtedly considered a universal solution for the removal of different types of pollution in the environment (Onawumi *et al.*, 2021; Bello *et al.*, 2017). Coconut is a multi-benefit plant that is quite familiar to most Nigerians. This plant is used in almost all parts by human, so it is considered a multipurpose plant (Saputro *et al.*, 2020). Coconut (*Cocos nucifera*) is widely grown in most of the island and reaches a height of 30 m. Utilization of coconuts for various daily food needs as well as for agricultural-industrial commodities, leaving waste in the form of shell called coconut shell (Figure 1a) (Sujiono *et al.*, 2022). This study focused on the production, modification and characterization of biosorbents generated from coconut shells, unmodified (UCNSA) and modified (MCNSA).

## Materials and Methods

**Biomass samples:** Coconut shell samples were procured from a farm settlement at Idi-Osan, Iragbiji, Boriye local government area, Osun State, Nigeria. The samples were placed in polythene bags and brought to the herbarium for Identification in the

Biological Sciences Laboratory unit of Federal University Otuoke.

**Sample preparation:** The method described by Onawumi *et al.* (2021) was adopted, 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 105 °C for 5 hours and allowed to cool in desiccators. The dried samples were pulverized and sieved to a desired particle size.

**Adsorbent modification:** The method described by Bello *et al.* (2017) was slightly modified by increasing the molarity of orthophosphoric acid from 0.3 M to 0.5 M. A carefully weighed 14.0 ± 0.01 g of raw samples was put into a beaker containing 250 cm<sup>3</sup> of 0.5 M orthophosphoric acid (H<sub>3</sub>PO<sub>4</sub>). The content of the beaker was thoroughly mixed and heated on a hot plate until a thick paste was formed. The pastes of CNSA samples were transferred into a crucible which was placed in a furnace and heated at 500 °C for 1 hour. Thereafter, the samples were allowed to cool and then washed severally with distilled water to the desired pH, 7.10 ± 0.101. Subsequently, oven dried at 105°C for 5 hours, sieved to desired particle size and stored in an air-tight container for further analysis and usage (Bello *et al.* 2017; Onawumi *et al.*, 2021). The unmodified (UCNSA) after pulverization and sieving was not treated with H<sub>3</sub>PO<sub>4</sub>.

## Adsorbent Characterization

**pH determination:** 3 g of each of the adsorbent samples was weighed and soaked into 30 ml of boiling deionized water for 24 hrs. The mixture was stirred to ensure proper dilution and filtered. The pH of the resulting filtrate was determined using a digital pH meter, Jenway 3520 (ASTM: D 3838; Boadu *et al.*, 2018; Onawumi *et al.*, 2021; Ebelegi *et al.*, 2022).

**Determination of moisture content:** The moisture content of the sample was determined according to standard methods ASTM D 2974 (2014). 2 g of the samples was weighed into the clean crucible. This was oven-dried at 105°C to constant weight and kept in a desiccator. The percentage moisture content was expressed mathematically (Boadu *et al.*, 2018)

$$\%MC = \frac{C - D}{C - B} \times 100 \quad 1$$

Where:

B = Weight of crucible (g)

C = Weight of crucible plus original sample (g)

D = Weight of crucible plus dried sample

**Bulk density:** The standard procedure used in analysing bulk density was from Ijaola *et al.* (2013) and Ebelegi *et al.* (2022). 5 g of the sample was placed into a pre-weighed 5 ml measuring cylinder ( $w_1$ ). The cylinders were gently 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 were recorded as ( $w_2$ ). The bulk density was expressed as:

$$\text{Bulk Density} = \frac{w_2 - w_1}{V} \quad 2$$

Where:

$W_2$  = weight of samples and cylinder (g)

$W_1$  = weight of measuring cylinder (g)

$V$  = volume of the measuring cylinder

**Ash content:** Ash content was determined according to the standard method (ASTM: D 2866-94; Ebelegi *et al.*, 2022). 5 g of dried samples was weighed into a crucible of a known weight and heated in a Gallenkamp muffle furnace for 6hrs at 600°C. 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 was expressed as the percentage weight of the original carbon sample.

$$\% \text{Total Ash} = \frac{C - D}{C - B} \times 100 \quad 3$$

Where:

$B$  = Weight of the crucible (g)

$C$  = Weight of crucible + original sample (g)

$D$  = Weight of crucible + ashed sample (g)

#### Volatile matter

Volatile matter content was determined according to the standard method (ASTM D3175-11) and method used by Ebelegi *et al.* (2022). 1g of samples was taken in a pre-dried crucible and covered with lid, then heated in a Gallenkamp muffle furnace regulated at 950°C for 7 minutes. After heating, the plate was quickly covered, cooled in desiccators and weighed. The amount weighed was taken as volatile matter.

$$\% \text{VM} = \frac{C - D}{C - B} \times 100 \quad 4$$

Where:

$B$  = Weight of the crucible (g)

$C$  = Weight of crucible + sample (g)

$D$  = Weight of crucible + volatile sample (g)

#### Particle size

Particle sizes of the ground samples alone was determined. The samples were prepared using an electric blending machine after which a sieve analysis was carried out using CONTROLS MILAND-ITALY D402-01 MATR 84000 109 sieve shaker at a rotation of 10-15 min with (75, 150, 300, 600, 750  $\mu\text{m}$ ) sieves (ASTM D-2862-97).

$$\% C = \frac{\text{Weight of carbon after sieve}}{\text{Total weight of carbon}} \quad 5$$

**Surface Area:** The surface area of the adsorbent was determined using the Sear's method, described by Ebelegi *et al.* (2022) where 0.5 g of each sample was carefully weighed into 250 ml conical flask containing 25 ml of 0.1M HCl at pH 3.50, after which 1 g of NaCl was added to raise the pH to 4, the mixture was titrated against a standard solution of 0.1M NaOH until pH 9 was achieved. The volume required to increase the pH from 4 to 9 was noted and used in computing the surface area using Eq. (6).

$$\text{Surface area (m}^2/\text{g)} = 32V - 25 \quad 6$$

where  $V$  is the volume of NaOH used to raise pH from 4 to 9.

#### Fourier transform infrared spectroscopy (FT-IR) analysis

Precisely 100 mg of potassium bromate (KBr) was weighed on a sensitive weighing balance and mixed with 2.1 mg of the adsorbents powder in a mortar and pestle. The mixture was compressed in a compressor machine until the sample was compacted. Samples were placed in a cell before fixing it in a Parkin Elmer FT-IR system BX spectrum and spectra readings (4000-400  $\text{cm}^{-1}$ ) were taken (Jabar *et al.*, 2020)

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

The morphology of the samples and elemental composition of UCNSA and MCNSA were obtained by scanning electron microscopy (SEM) using a JSM-7610F (Tokyo, Japan). The equipment is an ultra-high resolution schottky Field emission scanning electron microscopy coupled with energy dispersive x-ray/x-ray fluorescence spectrometer. The biosorbent surface was studied with a microscope operated at 10.0 kV. The samples were coated with a 10 nm thick layer of gold (Jabar *et al.*, 2020).

#### Results and Discussion

Tables 2 and 3 show the physicochemical properties and proximate compositions of unmodified (UCNSA) and modified (MCNSA) coconut shell (CNSA) adsorbents. The biomass revealed varying properties as follows:

**Adsorbent pH:** Scientific findings revealed that pH influences the charge density around adsorbent/adsorbent molecules (Ebelegi *et al.*, 2022). Thus, pH is a crucial parameter that influences the dissociation of active sites on the surface of adsorbents (Onawumi *et al.*, 2021). Tables 2 and 3 show that UCNSA has a pH ( $6.60 \pm 0.110$ ), and is a good adsorbent for anionic species while MCNSA with pH ( $7.10 \pm 0.101$ ) could take anionic, cationic, or neutral species. The pH range for the prepared biomass is within the range of those reported in literature (Ajala and Ali, 2020; Onawumi *et al.*, 2021; Abdullahi *et al.*, 2022) which suggests that the prepared adsorbents were ideal precursors for adsorption. The pH for optimum uptake of metals and organic pollutants by most adsorbents occurs between pH 6.0-9.0 (Aji *et al.*, 2015; Ebelegi *et al.*, 2022).

**Moisture Content (MC):** Moisture is the presence of liquid especially water in trace amounts. Research shows that moisture content (MC) increases linearly with Bulk density (Verla *et al.*, 2012; Ebelegi *et al.*, 2022). Therefore, high MC does not support adsorption while low MC enhances better adsorption efficiency of the adsorbent. Tables 2 and 3 revealed the range of MC ( $3.50 \pm 0.110$ - $6.20 \pm 0.100\%$ ) which suggests that MCNSA possesses a better adsorption efficiency than the UCNSA. Further, adsorbents with low MC have a longer shelf-life than the ones with high MC (Onawumi *et al.*, 2021). Hence, biosorbents with high MC should be further subjected to mild heat for some time before they could be used as this would have reduced the MC, and thus enhance an improved adsorption capacity (Ebelegi *et al.*, 2022).

**Volatile Matter:** Volatile matter (VM) is the amount of combustible material in biosorbents. The VM values were displayed in Tables 2 and 3 in the range of ( $16.42 \pm 0.111$  -  $15.10 \pm 0.110\%$ ) for both UCNSA and MCNSA which was considerably high, possibly due to the biomass origin (Onawumi *et al.*, 2021).

**Ash Content:** Ash content (AC) is a measure of the total amount of minerals present within a material, whereas, the mineral content is a measure of the amount of specific inorganic component present within a sample. The results of the AC revealed that UCNSA was  $15.10 \pm 0.110\%$ , and MCNSA ( $16.42 \pm 0.111\%$ ). Reports have it that high levels of AC lessen the general activity of adsorbents, hence it reduces the effectiveness of the adsorbents, in terms of re-use. Thus, the results obtained suggest the presence of an appreciable amount of ash in the biosorbents, and this could hinder their surface development (Ebelegi *et al.*, 2022). However, researchers have reported AC values

similar to what was obtained in the present studies (Boadu *et al.*, 2018)

**Fixed Carbon:** Fixed carbon (FC) gives information on the amount of char formation in a thermochemical conversion process (Ebelegi *et al.*, 2022). It is the solid combustible residue that remains after volatile matters are driven off. The higher the FC, the higher the char production in the thermochemical process as a product yield (Onawumi *et al.*, 2021). Tables 2 and 3 reveal that the values of UCNSA and MCNSA are  $68.70 \pm 0.010$  and  $71.08 \pm 0.001\%$  respectively. The higher the FC, the better the adsorption efficiency of the adsorbent, hence, MCNSA would be a better adsorbent than UCNSA due to its higher FC. Reports have it that a good biosorbent should have a  $FC \geq 65\%$  (Olayiwola *et al.*, 2015; Onawumi *et al.*, 2021). The FC values in the present studies fall within the range reported in the literature, hence, the biosorbents prepared are best for the adsorption process.

**Bulk Density:** Bulk density (BD) is a measure of the amount of adsorbate the adsorbent can hold per unit volume in  $g/cm^3$  (Onawumi *et al.*, 2021). Tables 2 and 3 showed the values of BD for UCNSA ( $0.720 \pm 0.000$ ), and MCNSA ( $0.769 \pm 0.000 g/cm^3$ ). It could be seen that MCNSA has a higher BD while UCNSA has the least value. The results of this study indicate that the biosorbents investigated are in the same range of BD found in previous studies (Onawumi *et al.*, 2021). Therefore, the BD values obtained for the adsorbents are within the ideal values widely reported in the literature making them excellent precursors for adsorption. However, since BD is inversely related to the surface area (SA) ( $BD \propto 1/SA$ ), hence, the biosorbent with lower BD would have better adsorption performance. Therefore, UCNSA would do better than MCNSA in relation to BD.

**Surface Area:** Surface area is a prominent factor influencing the adsorption process. The higher the SA, the better the adsorption due to the availability of many adsorption sites. Tables 2 and 3 presented the SA value of  $1100.00 \pm 0.000 m^2/g$  for UCNSA, while MCNSA has  $1120.00 \pm 0.000 m^2/g$ . Hence, MCNSA depicts a better adsorbent because of its higher SA which connotes the availability of its vacant adsorption sites than UCNSA. Reports have that activation promotes better porosity and high surface area (Onawumi *et al.*, 2021), and biosorbents with SA of 500-1500  $m^2/g$  are ideal for adsorption (Ajala and Ali, 2020; Wang *et al.*, 2020).

**Particle Size:** Particle size (PS) has a direct relationship with the surface area. Smaller particle size has direct correlations to the promotion of larger

surface area, providing more sites for adsorption (Ebelegi *et al.*, 2022). The value of particle size ( $300.00 \pm 0.000 \mu\text{m}$ ), and other physicochemical properties of the biosorbents under consideration conform to those reported in the literature (Ajala and

Ali, 2020; Abdullahi *et al.*, 2022), and in conformity with a quality threshold value of activated carbon as recommended by National Industrial Standard of Indonesia (SII) No. 0258-79, and National Standard of Indonesia (SNI) No. 06-3730-1995 (Table 1).

**Table 1:** Quality threshold standard for activated carbon

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

**Table 2:** Physiochemical properties and proximate compositions of unmodified coconut shell adsorbent

S/no	Parameters	Mean $\pm$ SE
1	pH	$6.60 \pm 0.110$
2	Moisture content (%)	$6.20 \pm 0.100$
3	Volatile matter (%)	$10.00 \pm 0.011$
4	Ash content (%)	$15.10 \pm 0.110$
5	Fixed carbon (%)	$68.70 \pm 0.010$
6	Bulk density ( $\text{g}/\text{cm}^3$ )	$0.720 \pm 0.000$
7	Surface area ( $\text{m}^2/\text{g}$ )	$1100.00 \pm 0.000$
8	Particle size ( $\mu\text{m}$ )	$300.00 \pm 0.000$

**Table 3:** Physiochemical properties and proximate compositions of modified coconut shell adsorbent (

S/no	Parameters	Mean $\pm$ SE
1	pH	$7.10 \pm 0.101$
2	Moisture content (%)	$14.10 \pm 0.101$
3	Volatile matter (%)	$9.20 \pm 0.112$
4	Ash content (%)	$8.98 \pm 0.111$
5	Fixed carbon (%)	$67.70 \pm 0.010$
6	Bulk density ( $\text{g}/\text{cm}^3$ )	$0.508 \pm 0.000$
7	Surface area ( $\text{m}^2/\text{g}$ )	$880.00 \pm 0.100$
8	Particle size ( $\mu\text{m}$ )	$300.00 \pm 0.000$

**Fourier transform infrared spectroscopy (FT-IR)**

**analysis:** Figures 1 and 2 revealed the spectra of UCNSA and MCNSA, while Tables 4 and 5 show the tabular interpretation. The spectra of the adsorbent samples showed the presence of some functional groups that are potential adsorption sites. The spectra revealed a reduction, broadening, disappearance and appearance of new peaks after the process of activation with  $\text{H}_3\text{PO}_4$  (Onawumi *et al.*, 2021). The shift in spectra authenticates the effect of activation on the adsorbents. The prominent bands after activation are indications that the prepared adsorbents would be effective in the removal of inorganic and organic pollutants (Bello *et al.*, 2017; Ajala and Ali, 2020; Abdullahi *et al.*, 2022). For both UCNSA and MCNSA biosorbents, the peak representing the bands were observed at  $3533.71$ ,  $3228.81$ ,  $1637.69 \text{ cm}^{-1}$ , and  $3429.55$ ,  $2926.11$ ,  $2004.11$ ,  $1732.13$ ,  $1633.17$ ,  $1464.02$ ,  $1246.06$ ,  $1033.88 \text{ cm}^{-1}$  respectively. The bands at  $3533.88$  and  $3429.55 \text{ cm}^{-1}$  represent the stretching of OH of lignocellulosic in MCNSA and

UCNSA. The peak at  $3286.8 \text{ cm}^{-1}$  represents the OH of carboxylic acid, while the peak at  $1637.69 \text{ cm}^{-1}$  may be due to the presence of a C=C stretching ring from lignin while that of  $2926.11$  and  $2004.11 \text{ cm}^{-1}$  may be due to saturated C-H stretching, and functional group due to C=C respectively. The bands at  $1732.13$ ,  $1633.76$ ,  $1246.06$  and  $1033.88$  may be due to functional groups such as C=O of aldehydes (-CHO) and ester (-COOR), C=C stretching ring from lignin, and C-O stretching of esters and carboxylic acids respectively. This is in agreement with the work of other authors (Nandiyanto *et al.*, 2019). Functional groups of adsorbents not only affect the adsorption behaviour, but also dominate the adsorption mechanism (Zheng *et al.*, 2014, Onawumi *et al.*, 2021). The peaks appearing in the FT-IR spectrum were assigned to various functional groups according to their respective wave numbers.

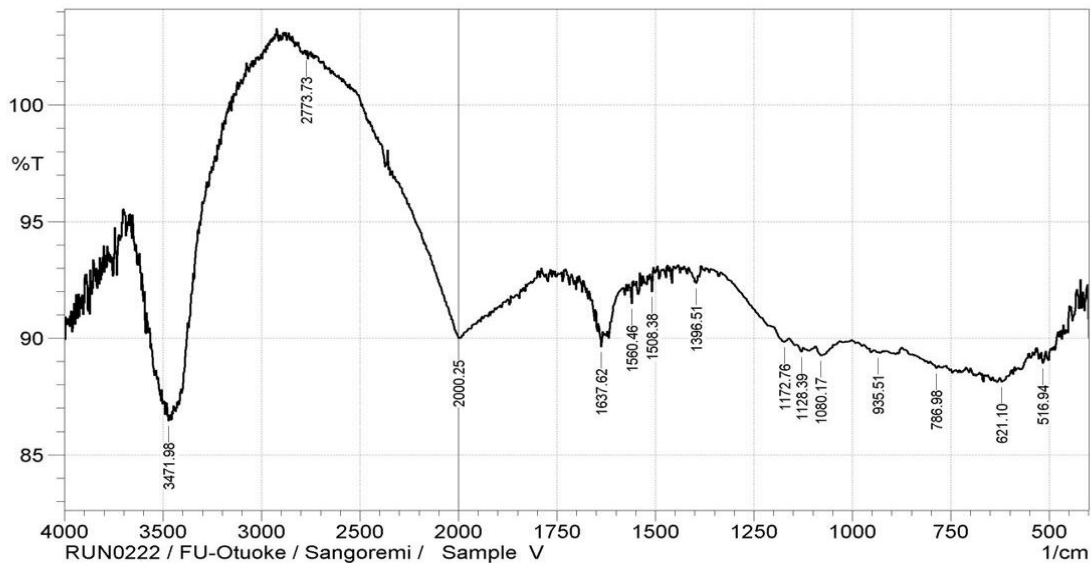


Figure 1: FTIR Spectrum of unmodified coconut shell adsorbent (UCNSA)

Table 4: FTIR spectrum interpretations of unmodified coconut shell adsorbent (UCNSA)

S.No	Wave number (cm <sup>-1</sup> )	Frequency range (cm <sup>-1</sup> )	Functional group assignment
1	3533.71	> 3500	Non bonded , O-H stretching
2	3286.81	3300-2400 (m)	OH stretching for -COOH
3	1637.69	1690-1640	C=N stretching

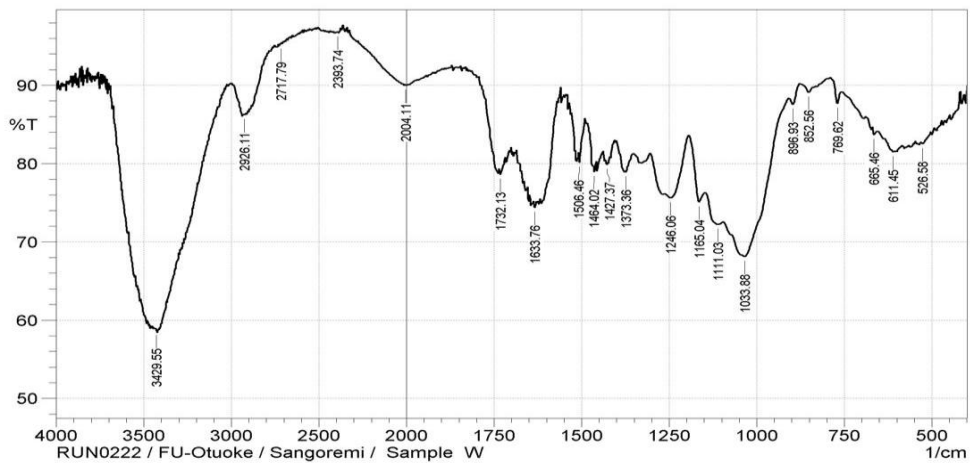


Figure 2: FTIR Spectrum of modified coconut shell activated carbon (MCNSA)

**Table 5:** FTIR spectrum interpretations of modified coconut shell (MCNSA)

S.No	Wave number (cm <sup>-1</sup> )	Frequency range (cm <sup>-1</sup> )	Functional group assignment
1	3429.55	3500-3200 (m)	O-H, hydrogen bonded
2	2926.11	3000-2850 (s)	C-H, alkane
3	2004.11	2100-1800	Carbonyl compounds
4	1732.13	1750-1730 (s)	Esters –COOR, Saturated aldehydes - CHO
5	1633.76	1680-1620	Non conjugated C=C
6	1464.02		-CH <sub>2</sub> -
7	1246.06	1320-1000 (s)	C-O stretching of esters, carboxylic acids
8	1033.88	1120-1030	CH <sub>3</sub> symmetric stretching

**Scanning Electron Micrographs (SEM) :** Figures 3 and 4 revealed the Scanning electron micrographs (SEM) of unmodified coconut shell (UCNSA), and modified coconut shell (MCNSA) adsorbents respectively. The surface morphology of the samples evidently shows that UCNSA surface pores were poorly developed and irregular as well as rough with heterogenous pore structures, but due to release of volatiles within the microstructure as a result of activation, several pores were formed, and distributed over the surface of precursors after acid treatment as seen on MCNSA micrograph. This shows that H<sub>3</sub>PO<sub>4</sub> was effective in creating well-developed pores on the surface of the precursors, hence, leading to active

carbon with a large surface area and additional porous exterior. This is in agreement with the works of other researchers (Bello *et al.*, 2015; Bello *et al.*, 2017; Ajala and Ali, 2020; Onawumi *et al.*, 2021; Abdullahi *et al.*, 2022; Ebelegi *et al.*, 2022). The availability of pores and internal surface is requisite for effective adsorption (Bello *et al.*, 2017). Thus, the porous nature of the prepared adsorbents helps in adsorbate uptake which will be advantageous in the adsorption process. These pores provide a good surface area for effluent treatment, remediation of organically polluted sites and regeneration of waste oils (Ahmad *et al.*, 2015a, b; Bello *et al.*, 2017; Ajala and Ali, 2020; Abdullahi *et al.*, 2022; Ebelegi *et al.*, 2022).

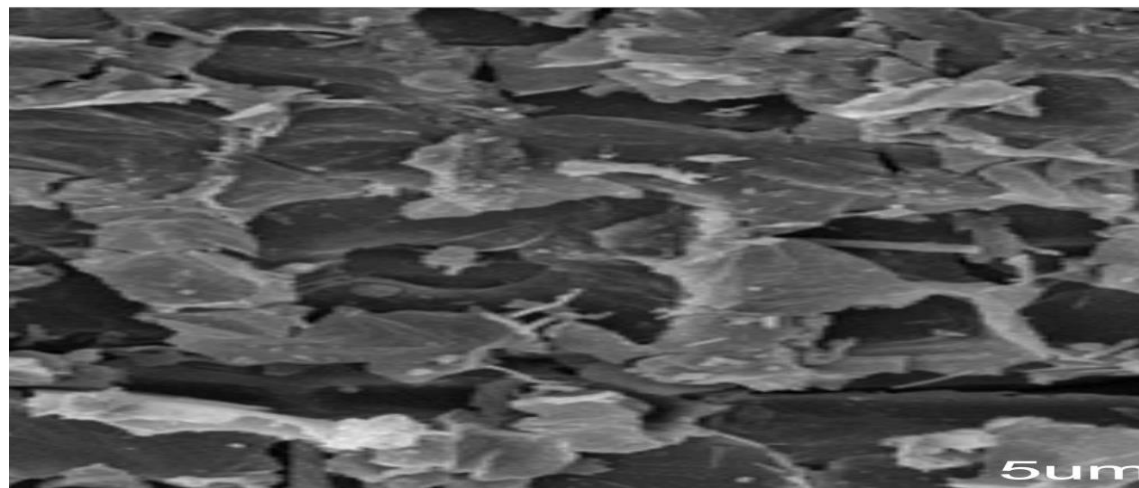
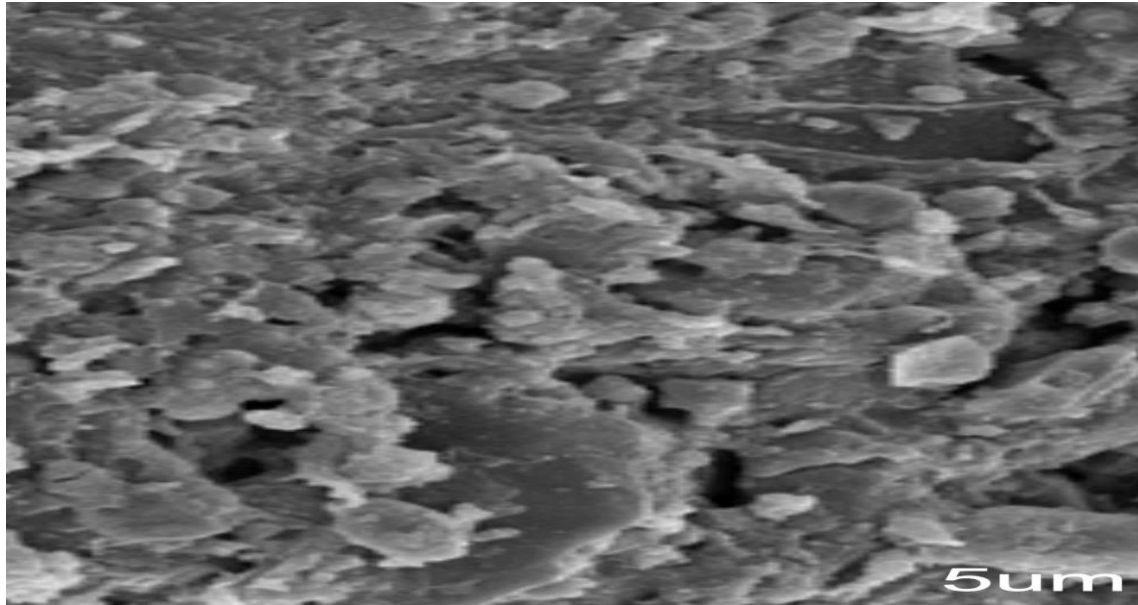


Figure 3: Scanning electron micrograph of unmodified coconut shell adsorbent

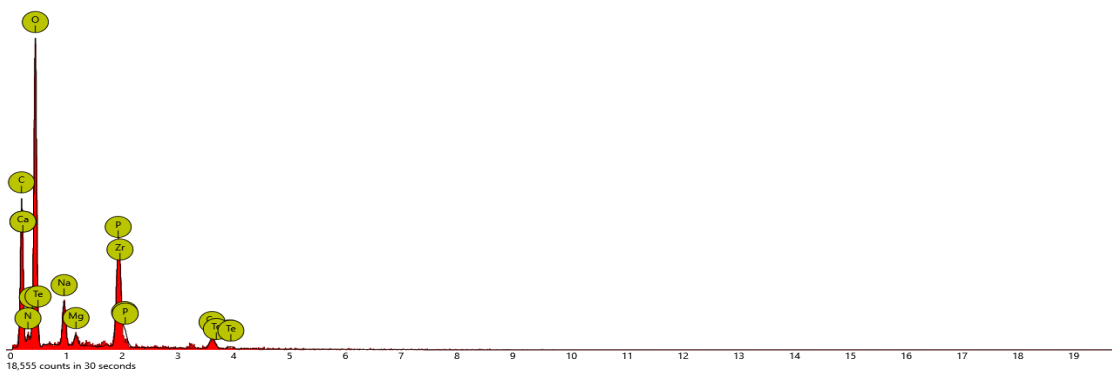


**Figure 4:** Scanning electron micrograph of modified coconut shell adsorbent

**Electron Dispersive X-ray Spectroscopy Analysis (EDX):**

The EDX analysis is a method of elemental analysis associated with electron microscopy based on the generation of characteristic x-rays that divulge the presence of elements present in a sample (Scimeca *et al.*, 2018). Figures 5 and 6 revealed the EDX spectra of UCNSA and MCNSA biosorbents, while Tables 6 and 7 revealed the elemental composition of the

adsorbents respectively. The carbon content increased from 40.32 to 65 % which demonstrates that the process of activation has enriched the carbon content in the adsorbents. Other elements present in percentage atomic weight include oxygen (42.92%), Nitrogen (5.64%), calcium (1.28%), Magnesium (0.55%), phosphorous (4.71%) (Sujiono *et al.*, 2022; Vaddi, *et al.*, 2022; Ushedo *et al.*, 2022).



**Figure 5:** EDX spectral of unmodified coconut shell adsorbent

**Table 6:** Elemental composition of unmodified coconut shell adsorbent

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
8	O	Oxygen	42.92	39.25
6	C	Carbon	40.32	30.11
7	N	Nitrogen	5.64	4.54
15	P	Phosphorus	4.71	8.39
11	Na	Sodium	2.56	3.39
40	Zr	Zirconium	2.02	10.59
20	Ca	Calcium	1.28	2.95
12	Mg	Magnesium	0.55	0.78

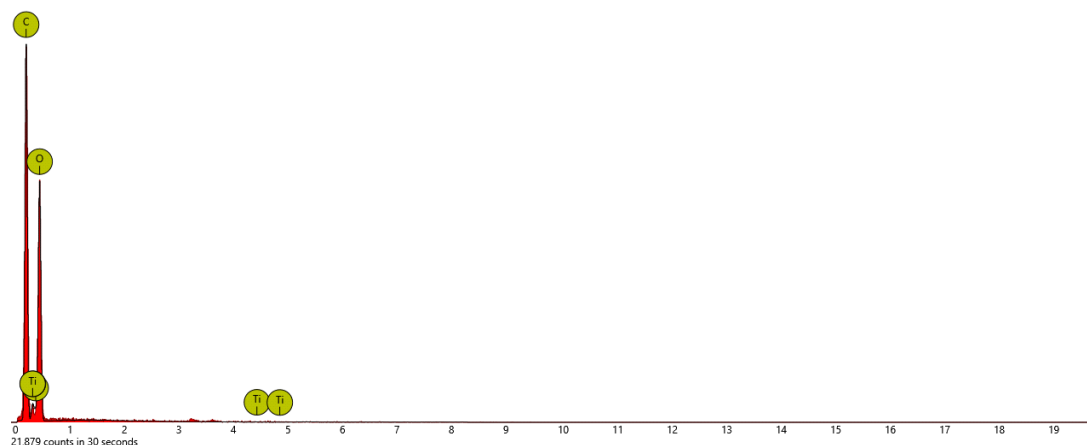


Figure 6: EDX spectral of modified coconut shell adsorbent

**Table 7:** Elemental composition of modified coconut shell adsorbent

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
6	C	Carbon	65.00	56.11
8	O	Oxygen	34.80	43.77
22	Ti	Titanium	0.20	0.12

**Conclusion**

The prepared biosorbents, UCNSA and MCNSA investigated in this study were observed to have low moisture contents, and ideal pH together with high fixed carbon including high surface area which are indices of excellent biosorbents. The results of the FTIR study indicated the reduction, broadening, appearance and disappearance of adsorption peaks, which showed that modification of the adsorbents with H<sub>3</sub>PO<sub>4</sub> was effective. The SEM results accurately explained the surface morphology of the prepared biosorbents, and MCNSA appeared to have favourable characteristics over UCNSA. The overall features

revealed that the prepared adsorbents could be considered excellent precursors for the adsorption process.

**Acknowledgments:** The authors wish to thank the Tertiary Education Trust Fund of Federal Republic of Nigeria for AST&D grant to carry out this research.

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