

## Deodorization of Hydrogen Sulphide Contaminated Water by Biosorption on Coconut Fibre Activated Carbon

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### ABSTRACT

The potential of activated carbon produced from *Cocos nucifera* (coconut) fibres, to remove Hydrogen Sulphide odour from aqueous solution was evaluated in a batch process. Experiments were carried out as function of contact time, initial concentration, dosage of the adsorbent and temperature. The surface chemical structure before and after adsorption experiments was analysed using Scanning Electron Microscope (SEM) and the type of chemical bonds was characterized using Fourier transform infrared reflection (FT-IR). The odour adsorption capacity of the prepared adsorbent was analysed using Hach H<sub>2</sub>S Test Kit (HS-7). The research experiment indicated a 95% performance of the prepared adsorbent which can be improved upon subsequent time of this research. From the research carried out, the results show that activated carbon produced from coconut fibres can be used for odour removal in water.

**KEYWORDS:** Activated carbon; Biosorption; Hydrogen sulphide; Adsorption; Wastewater treatment

### I. INTRODUCTION

Carbonaceous Agricultural wastes and by-products have long been recognized as good sources of activated carbon. At present, activated carbon finds wide application in many areas. It is used especially in environmental pollution control as well as in industry for various liquid and gas phase adsorptions processes<sup>1</sup>. Activated carbon has high internal surface area and pore volume which makes it very easy to be used as adsorbent, catalyst, or catalyst supports in gas and liquid phase processes for purification and chemical recovery<sup>2</sup>.

One major property of the activated carbon which makes it unique for experimental works is its adsorptive property. Adsorption is a process in which a substance (adsorbate), in gas or liquid phase, accumulates on a solid surface. It is based on the capability of porous materials with large surfaces to selectively retain compounds on the surface of the solid (adsorbent)<sup>3</sup>. The activated carbon chemistry is very important as adsorption of the pollutant (taste and odour causing compounds) is preceded by the adsorbate displacing the water to reach the surface of the carbon.

The adsorption process of the adsorbate molecules from the bulk liquid phase into the adsorbent surface is presumed to involve the following stages:

1. Mass transfer of the adsorbate molecules across the external boundary layer towards the solid particle.
2. Adsorbate molecules transport from the particle surface into the active sites by diffusion within the pore-filled liquid and migrate along the solid surface of the pore.
3. Solute molecules adsorption on the active sites on the interior surfaces of the pores.
4. Once the molecule adsorbed, it may migrate on the pore surface through surface diffusion.

The most important property of adsorbent, which determines its usage, is the pore structure and the specific surface area<sup>4</sup>. The total number of pores, their shape and size determine the adsorption capacity and even the dynamic adsorption rate of the adsorbent.

Powdered and granular activated carbons account for 48% and 35% of the global activated carbon market respectively, while other forms of activated carbons (such as pellet, bead and impregnated carbon) share the remaining portion<sup>5</sup>.

Adsorption of substances from liquids by activated carbon has been widely applied:

In food productions and processing industries for the removal of undesirable odours, colours, unwanted components to improve quality of food materials, preparation of alcoholic beverage, decolourization of sugar, oils and fats; in chemical and pharmaceutical industries for the purification, decolourization of medicine products; in textile industries for the removal of dyes; in petroleum industries for the refining liquid fuels, separation; in environmental sectors for spill clean-up, groundwater remediation, drinking water filtration and in Medicine, activated carbon can be used to treat a variety of ailments and poisonings<sup>1,6,7,8,9</sup>.

While water treatment is currently the dominant application of activated carbon, it can be used to pull contaminants from water, effluent or drinking, an invaluable tool in helping to protect the earth's most precious resource<sup>1</sup>.

More frequent complaints from consumers to water treatment industries arise due to odour and taste in their drinking water. Consumers may also perceive a risk to their health due to taste and/or odour in their water, resulting in a loss of consumer confidence. Water should be well treated before use. Odour and Taste in portable water results from the presence of various chemical substances/compounds in water/water sources, as summarized in a drinking water taste and odour wheel by Rogers,<sup>10</sup>.

To effectively control and remove tastes and odour imparted to the raw water or portable water by blooms of certain microorganisms and chemical substances, the most logical method is to work on their sources. Adsorption with activated carbon has been shown to be more generally effective at removing a wider range of odour and taste control<sup>11</sup>.

In this paper, the adsorptive capability/performance of activated carbon, produced from a low cost source material (agricultural wastes such as coconut fiber) in removing/controlling residual hydrogen sulphide odour and taste in usable water was studied.

## II. MATERIAL AND METHODS

### *Preparation and Characterization of the Activated Carbon*

Coconut (*Cocos nucifera*) fibres were prepared and activated as done by Ohimor *et al.*,<sup>12</sup>. The prepared and activated sample was also characterized as carried out in Ohimor *et al.*,<sup>12</sup>.

### *Preparation of the Sulphide Stock Solution*

All the chemicals used were analytical grade reagents. Stock solutions of the sulphide were prepared in a concentration of 1M using Sodium sulphide (Na<sub>2</sub>S), that is, 78.05g of Na<sub>2</sub>S in 1000ml of distilled water.

### *Adsorption Experiment*

The effect of a specific process parameter was determined by measuring the H<sub>2</sub>S adsorption efficiency by the coconut fibre activated carbon using the H<sub>2</sub>S test kit and varying the parameter while keeping other parameters constant.

The percentage efficiency of adsorption was evaluated from the equation:

$$\% \text{ Efficiency} = \left[ \frac{O_{cb} - O_{ca}}{O_{cb}} \times 100 \right]$$

where O<sub>ca</sub> and O<sub>cb</sub> are the initial and final (equilibrium) concentrations of the H<sub>2</sub>S in the solution (mg/l).

The amount of adsorbed H<sub>2</sub>S (mg H<sub>2</sub>S /g biomass) were calculated from the decrease in the concentration of H<sub>2</sub>S in the medium by considering the adsorption volume and used amount of the biosorbent:

$$q_e = \frac{(O_{cb} - O_{ca})V}{m}$$

where q<sub>e</sub> is the amount of H<sub>2</sub>S adsorbed into unit mass of the biosorbent (mg/g) at equilibrium, V is the volume of H<sub>2</sub>S solution (L) and m is the amount of biosorbent used (g).

### *Procedure for testing the presence of H<sub>2</sub>S in the water sample using the H<sub>2</sub>S test kit*

The sample Vial was filled to the 25ml mark with the Sulphide stock solution. The H<sub>2</sub>S test paper (lead acetate paper) was placed in the Vial cap and an effervescent tablet (Calcium Citrate Vita plus) was added to the Vial containing the sample and immediately the sample Vial was covered with the cap containing the H<sub>2</sub>S test paper (lead acetate paper). The sample was observed till the tablet dissolved totally and then the Vial was open and the H<sub>2</sub>S test paper was removed and the colour change was compared with the H<sub>2</sub>S test chart and then readings were recorded in mg/L for each observation/experiment.

### *Effect of dosage on H<sub>2</sub>S adsorption*

A 2mg/l solution of the adsorbate was prepared into five (5) different test tubes each containing 25ml of the solution. 0.2g, 0.4g, 0.6g, 0.8g and 1.0g of the adsorbent were added to each of the test tubes A-E respectively and then the test tubes were corked. The mixture was placed in a water bath horizontally and incubated at 35°C for 90 minutes before it was removed and filtered. The percentage efficiency and concentration of the adsorbate before and after was determined using the H<sub>2</sub>S test kit.

### *Effect of concentration on H<sub>2</sub>S adsorption*

Five different test tubes each containing 1g of the adsorbent was prepared. 2mg/l, 1.5mg/l, 1mg/l, 0.5mg/l and 0.3mg/l of the adsorbate was added to the test tubes respectively and then the test tubes were corked. The mixture was placed in water bath horizontally and incubated at 35°C for 90 minutes before it was removed and filtered. The percentage efficiency and concentration of the adsorbate was determined using the H<sub>2</sub>S test kit.

### *Effect of contact time on H<sub>2</sub>S adsorption*

A mixture containing 0.5g of the adsorbent and 2mg/l of the adsorbate was prepared. Five (5) different test tubes each containing 25ml of the mixture were corked and placed in water bath horizontally and incubated 35°C. The test tubes were removed from the water bath and filtered at 20 minutes interval for 100 minutes. The percentage

efficiency and concentration of the adsorbate was determined using the H<sub>2</sub>S test kit immediately after each removal.

*Effect of temperature on H<sub>2</sub>S adsorption*

A mixture containing 1g of the adsorbent and 2mg/l of the adsorbate was prepared. 25ml of the mixture was pour into a test tube then it was corked and placed in a water bath and incubated at 35°C for 90 minutes. The test tube was removed from the water bath and then filtered. The percentage efficiency and concentration of the adsorbate was determined using the H<sub>2</sub>S test kit immediately. The process was repeated for temperatures at 45°C, 55°C, 65°C, 75°C and 85°C respectively.

*Fourier Transform Infrared Spectroscopy Analysis (FT-IR)*

The FT-IR analysis was carried out in order to determine the chemical composition, functional group and the type of bond present in the prepared adsorbent material. The adsorbent material before and after sorption experiment was examined using Infrared Spectrophotometer (Transmittance method) in the range of 650 – 4000 cm<sup>-1</sup> (wavelength).

*Scanning Electron Microscope (SEM) Analysis*

In order to determine the surface morphological composition and the element present in the prepared adsorbent before and after adsorption experiment, the SEM analysis was carried out. The morphological composition of the prepared adsorbent before and after adsorption experiment was examined using Scanning Electron Microscope magnification X-1000.

### III. RESULTS AND DISCUSSION

*Results of Carbonization and Activation*

**Table 1: Results of Carbonization and activation**

Precursor	Coconut fibres (husk)
Initial weight (g)	3200
Weight after carbonization (g)	704
% Weight loss after carbonization	78%
Weight of sample activated (g)	600
Weight after activation (g)	570
Percentage yield (%)	21
Carbonization temperature (°C)	450
Activating agent	KOH
Activation temperature (°C)	700

*Results of Characterization of the Activated Carbon*

**Table 2: Properties of the activated carbon**

S/N	PARAMETER	VALUE
1	pH	7.03
2	Pore Volume	0.78E3
3	Percentage Weight Loss (%)	78
4	Bulk Density (g/cm <sup>3</sup> )	0.16
5	Ash content	0.89
6	Moisture content (%)	11.76
7	Colour	Black
8	Particle size (µm)	63

*Results of Adsorption Experiment*

Figure 1a and b is a sample of the H<sub>2</sub>S test paper (lead acetate paper) before and after use.



**Figure 1: a)** H<sub>2</sub>S test paper before use

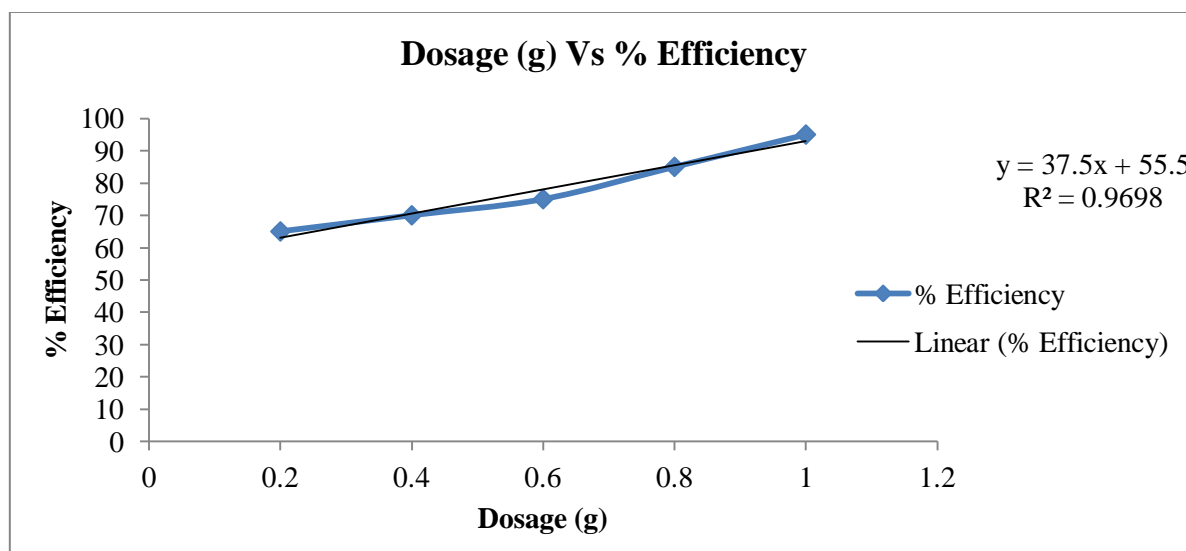


**b)** H<sub>2</sub>S test paper after use

*Effect of dosage on H<sub>2</sub>S adsorption*

The effect of dose of adsorbent on the efficiency (percentage removal) of H<sub>2</sub>S (adsorbate) from the water sample was investigated at H<sub>2</sub>S adsorption time of 90 minutes and the maximum equilibration speed was about 125rpm. It was found from the Figure 2 that the percentage removal of H<sub>2</sub>S was increased by increasing adsorbent (activated carbon) dosage. This is because with increase in adsorbent dosage the numbers of available adsorption sites were increased for H<sub>2</sub>S sorption<sup>13,14</sup>.

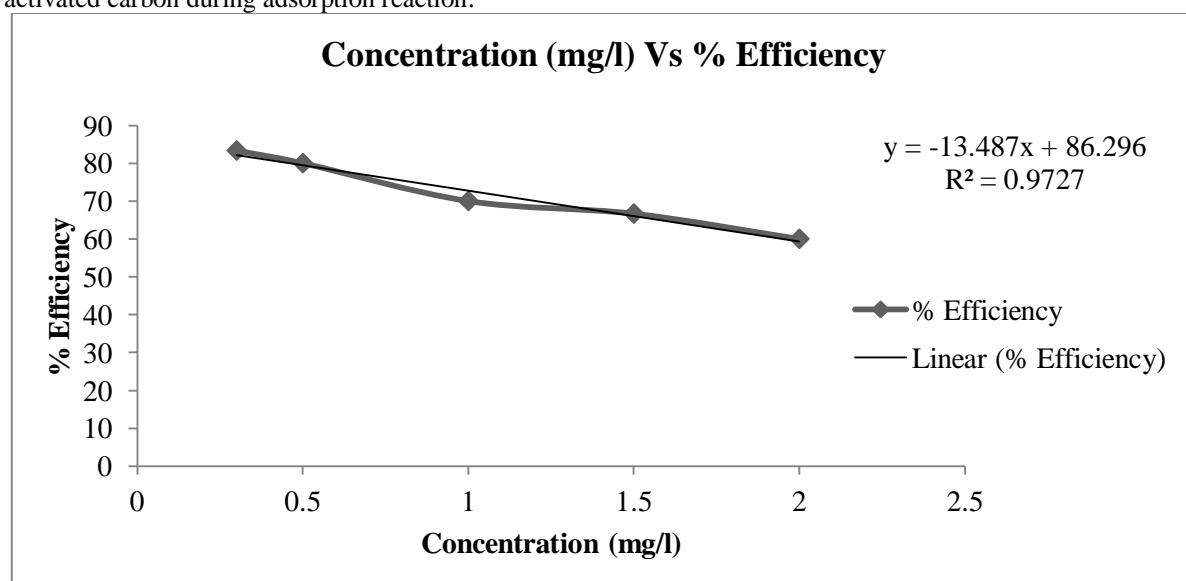
From the results, one can deduce that further increase in dosage will results to 100% efficiency in H<sub>2</sub>S removal from the water sample. That is, the adsorbent is still very much unsaturated during the adsorption reaction; hence more of the adsorbate can be adsorbed unto the adsorbent.



**Figure 2:** Effect of dosage on H<sub>2</sub>S Adsorption unto the prepared activated carbon (initial concentration = 2mg/l, time = 90mins and temperature = 35°C).

*Effect of initial concentration on H<sub>2</sub>S adsorption*

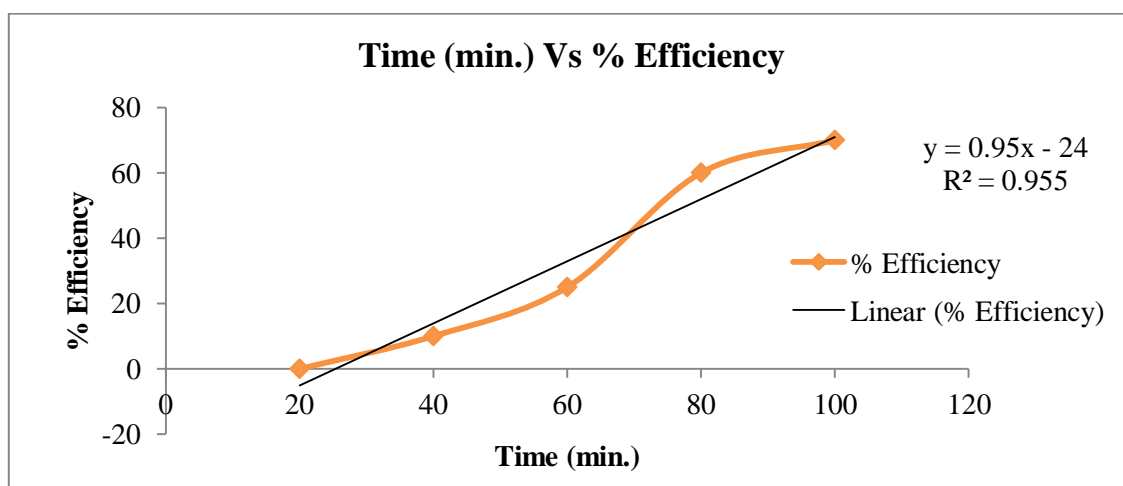
The effect of initial H<sub>2</sub>S concentration (0.3mg/l – 2.0mg/l) on the removal efficiency using prepared activated carbon of 1g was studied. It was observed from **Figure 3**, the sorption yield of the adsorbate was decreased with increasing initial H<sub>2</sub>S concentration over the studied range<sup>15</sup>. These results may be due to lack of availability of active sites for the H<sub>2</sub>S sorption onto the prepared activated carbon at high initial adsorbate concentration<sup>14</sup>. The decreased in the adsorbate sorption can also be ascribed to saturation of the prepared activated carbon during adsorption reaction.



**Figure 3:** Effect of initial concentration on H<sub>2</sub>S Adsorption unto the prepared activated carbon (dosage = 1g, time = 90mins and temperature = 35°C).

*Effect of variation of contact time on H<sub>2</sub>S adsorption*

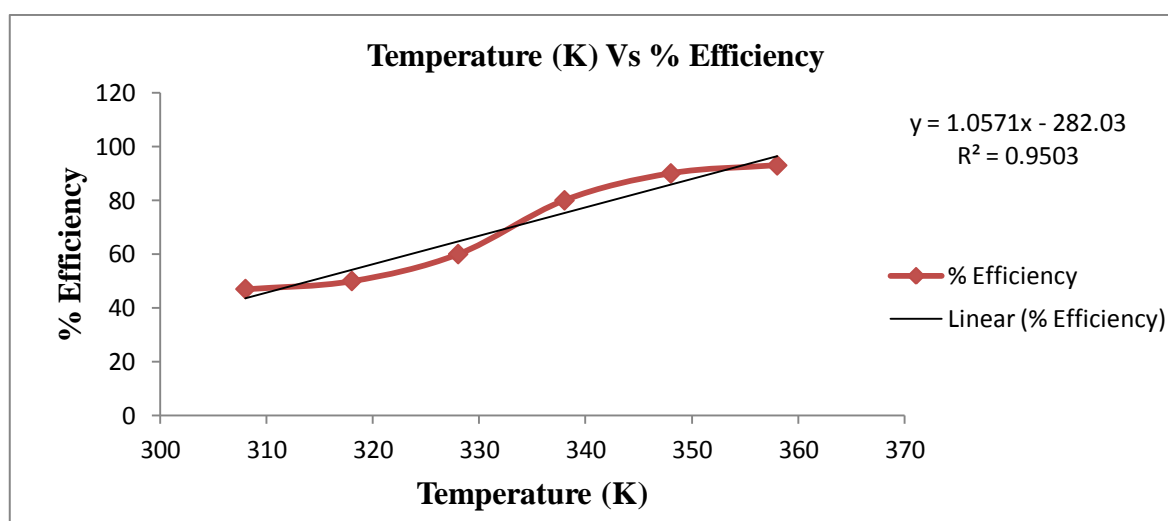
The effect of contact time on the efficiency (percentage removal) of the adsorbate onto the prepared activated carbon was investigated. From the result **Figure 4**, the percentage removal of adsorbate from the water sample increases with increase in contact time with the adsorbent. Though, the equilibrium time for the sorption process for the prepared adsorbent material was not investigated but from the result, it can be deduced that there will be an equilibrium sorption time for the prepared activated carbon material since the rate of sorption from 40 – 80 minutes is higher compared to 80–100minutes, which means that the adsorption process is tending to an equilibrium. Similarly with increase in time duration the percentage removal efficiency of Methylene blue increases after sufficient time (9-10hours) and under equilibrium condition the removal efficiency of Methylene blue shows >90%<sup>16</sup>.



**Figure 4:** Effect of variation of contact time on H<sub>2</sub>S Adsorption unto the prepared activated carbon (initial concentration = 2mg/l, dosage = 1g and temperature = 35°C).

*Effect of temperature on H<sub>2</sub>S adsorption*

The effect of temperature (35°C – 85°C) on the H<sub>2</sub>S percentage removal using prepared activated carbon of 1g was studied. From the result **Figure 5**, the percentage adsorption of the adsorbate was increasing with increased in temperature of the mixture. This may be due to saturation of the prepared activated carbon during adsorption reaction<sup>14</sup>. Adsorption efficiency is generally a function of system temperature which affects both the rate of diffusion within the liquid phase and the final adsorption equilibrium. Hence, decreasing viscosity and increasing molecular movement at higher temperature allow the organic molecules to enter the carbon pores easily, resulting to increased adsorption rate and total adsorbate adsorbed<sup>17</sup>.



**Figure 5:** Effect of temperature on H<sub>2</sub>S Adsorption unto the prepared activated carbon (initial concentration = 2mg/l, time = 90mins and dosage = 1g).

FT-IR analysis

The coconut fibres adsorbent, before it was used for adsorption experiment, shows the broad band 3775.8, 3123.5, 2325.9, 2105.9, 1986.7 and 1401.5 $\text{cm}^{-1}$  as shown in Figure 6. At peak 3123.5 $\text{cm}^{-1}$ , OH acid (carboxylic acid) stretch was observed. Peak 2325.9  $\text{cm}^{-1}$  shows the presence of the carbonate ( $\text{CO}_2$ ) and peaks 2105.9, 1986.7 and 1401.5  $\text{cm}^{-1}$  depicts the presence of an alkyne ( $\text{-C}\equiv\text{C-}$ ), transitional metals and an aromatic ( $\text{-C=C-}$ ) respectively <sup>18</sup>.

From Figure 6, wavelengths corresponding to 400 $\text{cm}^{-1}$  - 1500 $\text{cm}^{-1}$  are considered as fingerprint region. In this region, strong absorption intensity is usually observed <sup>19</sup>. The highest peak with frequency (3123.5 $\text{cm}^{-1}$ ) suggests OH acid (carboxylic acid) is the main functional group present in the prepared adsorbent <sup>14</sup>.

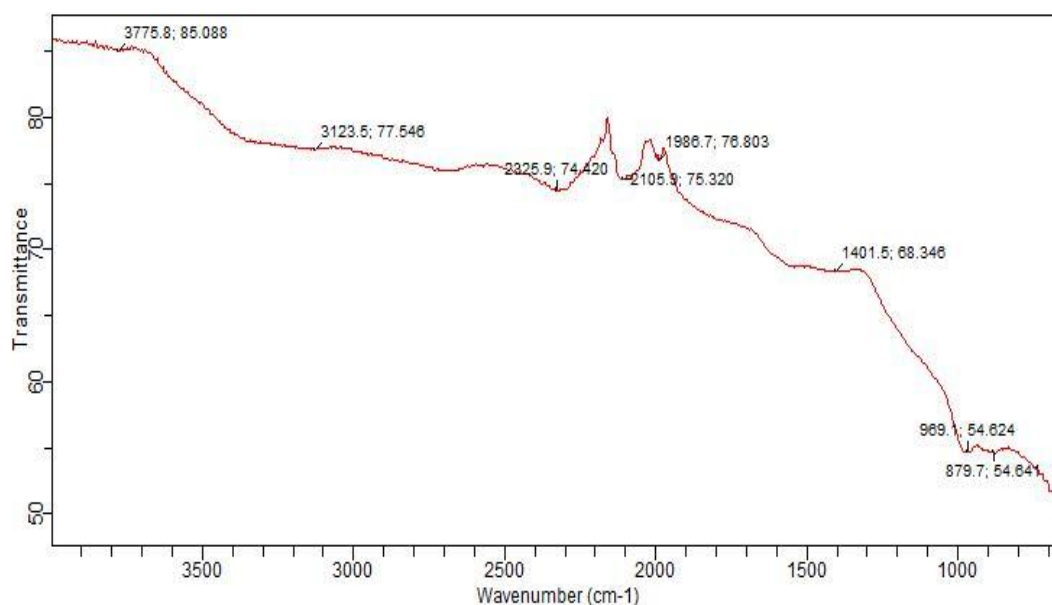


Figure 6: FT-IR Spectrum for coconut fibres before adsorption

The coconut fibres adsorbent, after adsorption with the adsorbate ( $\text{H}_2\text{S}$ ), the biosorbent showed the broad band 3652.8, 2657.6, 2325.9, 2109.7, 1990.4 and 1774.2 $\text{cm}^{-1}$  as shown in figure 4.6 below. At peak 3652.8 $\text{cm}^{-1}$ , OH (alcohol OH stretch) was observed. Peak 2657.6 $\text{cm}^{-1}$  shows the presence of OH acid (carboxylic acid) and peaks 2109.7, 1990.4 and 1774.2 $\text{cm}^{-1}$  shows the presence of an alkyne ( $\text{-C}\equiv\text{C-}$ ), an alkene ( $\text{=C=C=C=}$ ), and an anhydride ( $\text{-C=O-}$ ) respectively <sup>18</sup>.

From figure 7, wavelengths corresponding to 400 $\text{cm}^{-1}$  - 1500 $\text{cm}^{-1}$  are considered as fingerprint region. In this region, strong absorption intensity is usually observed <sup>19</sup>. The highest peak with frequency (3652.8 $\text{cm}^{-1}$ ) suggests OH bond (Alcohol OH stretch) is the main functional group present in the prepared adsorbent <sup>14</sup>.

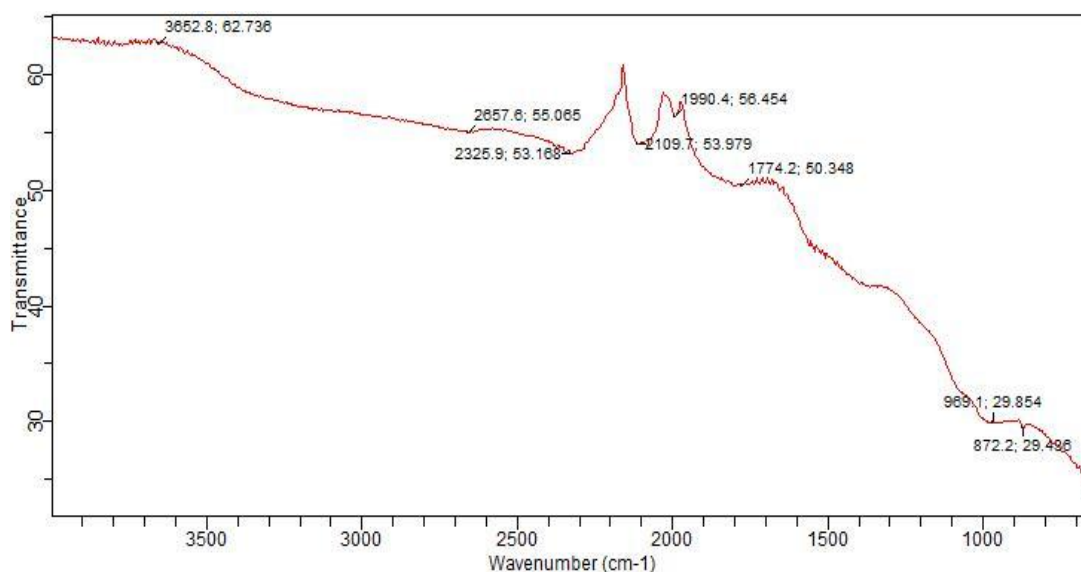
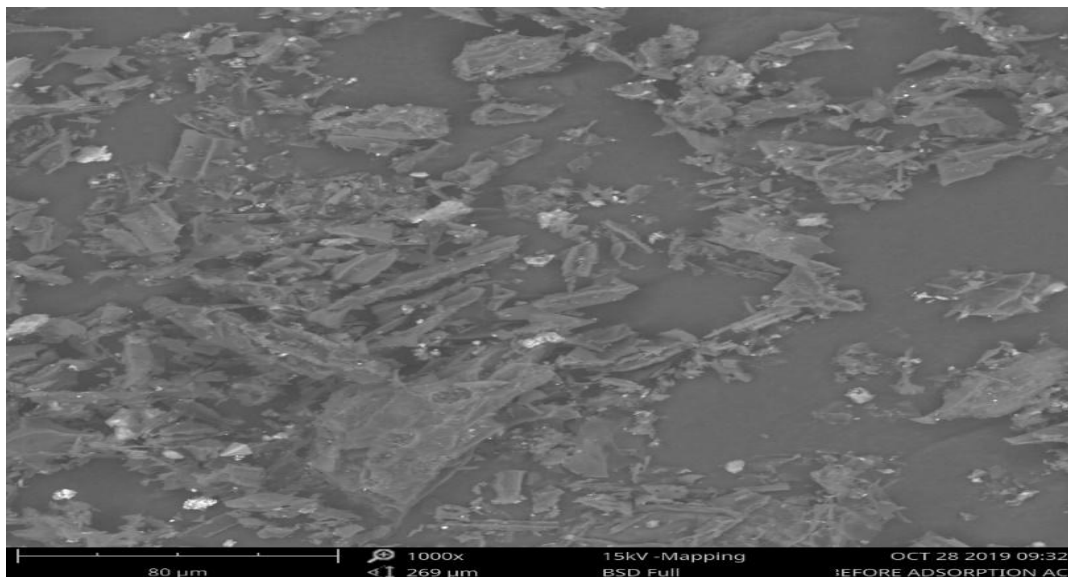


Figure 7: FT-IR Spectrum for coconut fibres after adsorption



*SEM results for the adsorbent before adsorption experiment*

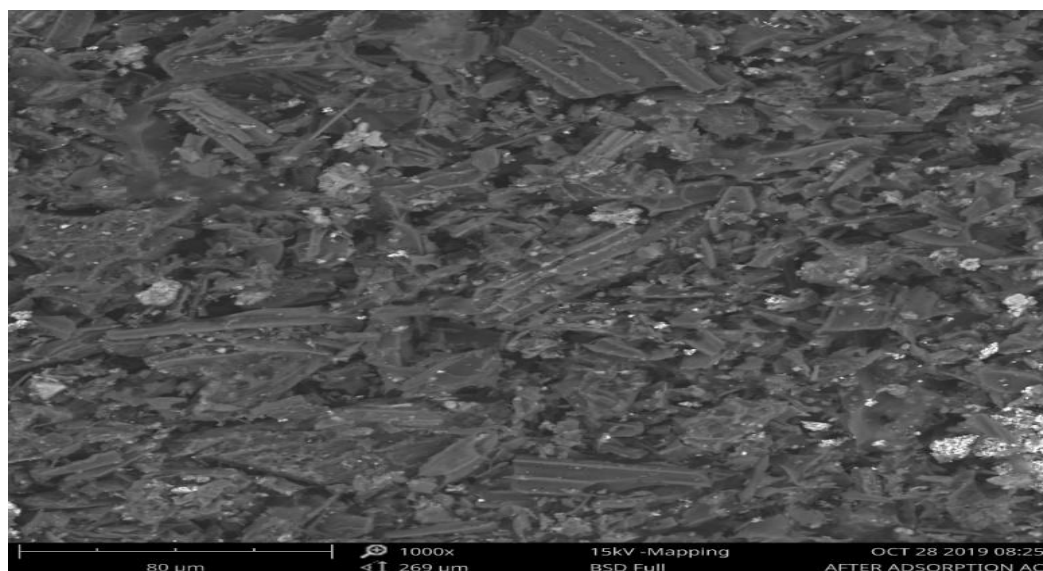
The surface morphology of the prepared adsorbent before it was used for the adsorption shows a wide variety of pores, some shiny surface as spotted in the SEM Figure 8 with X-1000 magnifications. This may be due to the reaction of the impregnating agent (KOH). The particle sizes are irregular, containing high surface area available for adsorption of the adsorbate.



**Figure 8:** SEM Visualization of the microstructure of the coconut fibres (before adsorption) at X-1000 magnification

*SEM results for the adsorbent after H<sub>2</sub>S adsorbent*

The SEM was used to observe the morphological composition of the adsorbent (activated carbon produced from coconut fibres). The SEM images showed that the surface of the adsorbent was irregular, rough and highly porous, which indicates the adsorbent to be a good material for adsorption study. From Figure 9, the effect of the adsorbed adsorbate is clearly shown



**Figure 9:** SEM Visualization of the microstructure of the coconut fibres (after adsorption) at X-1000 magnification

#### IV. CONCLUSION

The prepared activated carbon was effective upon application to the water sample containing the adsorbate (H<sub>2</sub>S, the odour causing compound in the water). From the sorption experiment carried out, the maximum efficiency of the prepared adsorbent was 95% which can be improved upon in subsequent research. The goal of this research was reached from the experimental and analytical results obtained.

From the research carried out, it was concluded that activated carbon produced from coconut fibres can be used for odour removal in water.

In order to carry out an elaborate study of the adsorptive capacity of the coconut fibre activated carbon, it is recommended that other pollutants apart from the hydrogen sulphides be used.

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