



## Characterization the Removal of Phenol from Aqueous Solution in Fluidized Bed Column by Rice Husk Adsorbent

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### Abstract

Pollution deals with the modification of the natural physical and chemical environment of the earth of human activity. Phenol is one of the most toxic industrial pollutants and even at low concentration it is reported to affect water quality and is harmful to human health. The objective of this study is to contribute in the search for less expensive adsorbents and their utilization possibilities for agricultural waste by – products such as rice husk, for the elimination of phenol from aqueous solution. Experiments have been conducted to examine the liquid – phase adsorption of phenol from aqueous solution by rice husk RH and activated rice husk ARH(pretreated by 13 M  $H_2SO_4$  with 0.5 NaOH and thermally treated at 573k). Fluidized bed column kinetic and isotherm studies were carried out evaluate the effect pH 2 -10, initial phenol concentration 50 – 200 mg/l ,different bed height 5 – 30 cm , different temperature 298 – 338 K and different flow rate 1 – 7 cm<sup>3</sup>/s , with 90 min operated the system. The sorption data obtained from studies at optimized conditions have been subject Langmuir and Freundlich isotherm studies. The data fits well to both the Langmuir and Freundish isotherm models indicating favorable and monolayer adsorption. X – ray diffraction analysis , which indicates that the RH and ARH mainly consist of amorphous materials. The adsorbent were characterized using FTIR, it was found that the pretreatment of rice husk increase the specific surface area and changed the functional groups , therefore leads to increase the capacity of adsorption.

**Keywords:** Catalytic rice husk, activated rice husk, phenol, isotherm, fluidized bed column.

### Introduction

Discharging different kinds of wastewater and polluted waters such as domestic, industrial and agricultural wastewaters into environment, especially to surface water, can cause heavy pollution of this body sources with regard to increase effluent discharge standards to the environment<sup>1</sup>. Contamination of ground and surface water by different organic pollutant from various activities include industrial and agricultural, becoming high concern by authorities<sup>2</sup>. Fresh water in already a limiting resource in many parts of the world, in the next century, it will become even limiting due to increased population. Urbanization, climate change and pollution in fresh water ecosystems by liquid and solid water produced by human settlements and industrial activities; thus leading to water crisis and serious consequences on the environment<sup>3</sup>.

The most important technology for eliminating organic pollutant from water and wastewater are, chemical precipitation, electrofloatation, reverse osmosis, adsorption ion- exchange<sup>4</sup>. Among those methods, adsorption is highly efficiency technique which has gained considerable attention for treating compounds from aqueous streams. The efficiency of the adsorption process is mainly due to the characteristic of the adsorbent such as high surface area, high

adsorption capacity, microporous structure and special surface reactivity. Despite of these advantage adsorption process is expensive due to the high cost of the adsorbents and the regeneration process. Consequently, there has been a growing interest in developing and implementing various potential adsorbents for the removal of organic compounds from water and researchers are always in a hunt for developing more suitable, efficient, cheap and easily accessible types of adsorbent, particularly from the waste materials<sup>5,6</sup>.

In recent years, special attention has been focused on the use of natural adsorbents as an alternative to replace the conventional adsorbents<sup>7</sup>. Natural materials such as rice husk that are available in large quantities, or certain waste products from industrial or agricultural operations , may have potential as inexpensive sorbents<sup>8</sup>. Rice husk is easily available low cost agriculture waste biomass. Approximately 80 million tons of rice husk are obtained annually in the world<sup>9</sup>. Rice husk is generated as a waste during the first stage of rice milling, when rough rice or paddy rice is husked (husk is separated from the rest of the grain), it contains about 20% silica in combination with a large amount of the structural polymer<sup>10</sup>. The concept of up flow fluidized – beds a result the engineering development has not progressed as far as fixed - beds and moving technology. The fluidized –

beds offer a high available surface area, since there is no contact between particles, intimate contact of the entire surface with the waste stream is assured<sup>11</sup>. This work aimed to study the feasibility of using rice husk RH and chemically modified rice husk ARH for removal organic compounds form water.

## Material and Methods

**Preparation of Adsorbent:** The rice hush (RH) used in this study were obtained from a local mill, chemical and physical properties of rice husk are reported in table 1, was washed at several times with tap water and dried in sunlight for 5 days and cleaned with distilled water to remove any dirt remains and then oven – dried at 110 C° (WG7L, Electric Blast Dry Box ,Tiajin City Taisite Instrument Co. Ltd.), for 18 hours to remove moisture. The cleaned (RH) were milled in (Nautiunl, Juicer/ Blender Model MJ- 176NR Japan), the RH characterized for their surface area by electric shaker sieve (Electric Sieve Shaker, Germany). For the preparation of activated rice husk ARH were carried out using base, acid and organic compounds. Three different modification methods were selected.

**Table - 1**  
**Physical – chemical characteristics of rice husk and activated rice husk**

Properties	Rice Husk	Activated Rice Husk
Surface area (m <sup>2</sup> /g)	97.5	138.6
Bulk density (kg/m <sup>3</sup> )	1011	890
Pore Volume (m <sup>3</sup> /kg)×10 <sup>-4</sup>	2.32	2.83
Carbon (%)	42.3	46.5
Ash (%)	18.4	16.9

**Table-2**  
**Chemical and physical properties of phenol [13, 15]**

Formula	C <sub>6</sub> H <sub>5</sub> OH
Molecular weight (g/mol)	94.11
Melting temperature (°C)	40.9
Boiling point (°C)	181.75
Water solubility (r.t.)	9.39 phenol / 100 m H <sub>2</sub> O
pKa	9.89
Flammability limits in air (vol.)	1.7 (lower) – 8.6 (higher)
Flash point (°C)	79(closed cup)
Auto- ignition temperature (°C)	715

i. base treatment using 0.5M NaOH with stirring at room temperature, ii. acid treatment use 13 M H<sub>2</sub>SO<sub>4</sub> at 175-180°C for 20 min with stirring and iii. organic treatment by 1% formaldehyde in the ratio of (1,5 (RH, formaldehyde, w/v)) at room temperature for 24 hours with stirring. The RH was

washed with distilled water and dried at 50-60°C in electrical oven till the weight attained a steady state and constant value. Thermal treatments of RH were obtained by burning the RH at temperature of 300°C (GSL 1600 X, VAGVVM France, China) this temperature considerable amount of carbon would be produced<sup>6,9</sup>.

**Properties and Preparation the Adsorbate:** In the present investigation, sorption of phenol from water was carried. Phenol is an organic chemical usually present in industrial wastewater, chemical and physical properties of phenol are reported in table 1. Phenol was first isolated from coal tar in 1834 by the German chemist Range<sup>12</sup>. It is an aromatic hygroscopic crystalline solid at ambient temperature and pressure. When pure, solid phenol is white but is mostly colored due to the presence of impurities. Phenol is very soluble in ethyl alcohol, ether and several polar solvents, as well as in hydrocarbons such as benzene<sup>13</sup>. In water it has a limited solubility and behaves as a weak acid. Phenol compounds are poisonous to aquatic life, plants and human as a product of bioconcentration. Ingestion of phenol in concentration from 10 – 240 mg/l for a long time causes mouth irritation, diarrhea, and excretion of dark urine and vision problem<sup>14</sup>.

The test solution were prepared by diluting of stock solution of phenol to the desired concentration. A stock solution of was obtained by dissolving 1.0 g of phenol (obtained by FLUKA AG, Chemicals Fabrik CH-9474 Buchs) in 1 liter distilled water. Desired solution of phenol were prepared using appropriate subsequent dilutions of the stock solution. The rang in concentration of phenol prepared from standard solution varied between 50–200 mg/l. The PH of the solutions was adjusted to the 0.1 N NaOH solution.

**Column Adsorption Studies:** Fluidized bed column was constructed by connecting glass tubes to contain the adsorbent figure 1, by using water from water network in the feed container , the three – way valve was opened in the vent direction. The solution was pumped through the rotameter to the vent opening for two – three min to allow bubbles to be moved out. The direction of the three – way valve was converted to the column which contained the bed of adsorbents. The experiments continue for 90min, shutdown the process (switch off the pump), 25ml of the sample was taken from outlet of the column (effluent tank), direct injection of the sample into the UV – visible spectrophometer (Hitachi, U- 2001). The amount of adsorbent per unit mass calculated using following equation.

$$q_e = \frac{(C_0 - C_e)V}{m} \quad (1)$$

Where, q<sub>e</sub>, the amount of adsorbent (mg/g), (C<sub>0</sub> and C<sub>e</sub>) are the initial and equilibrium concentration of phenol, respectively. V is the volume of solution (ml) and m is the amount of the RH (g). The % sorption was measured by the following equation -

$$\text{Sorption \%} = \frac{(C_0 - C_e)v}{C_0} \times 100 \quad (2)$$

## Results and Discussion

**Effect of pH:** It is well known that pH of the solution in one of the prime factors influencing the sorption's efficiency of the system. The effect of pH on the adsorption characteristic of rice husk RH and activated rice husk ARH were determined in the pH 2 – 10, and constant other parameters (T = 298k, flow rate = 4 cm<sup>3</sup>/s, bed height = 15 cm and initial concentration of phenol = 100mg/L). The adsorption of phenol from aqueous solution is dependent on the pH of the solution which affects the surface charge of the adsorbent, degree of ionization and speciation of the adsorbate species<sup>16</sup>. The results are displayed in figure 2. As was expected, the adsorbed amount decreases with increasing the pH value. This can be explained on the basis that positively charged surface is formed on the adsorbents at a lower pH due to adsorption of hydrogen ions on the surface of adsorbents<sup>3-17</sup>. From the other hand at low pH, the concentration of H<sup>+</sup> ions increased and the surface of the biomass become positively charged. A strong electrostatic interaction develops between positively charged biomass surface and negatively charged sites on the biosorbent<sup>9</sup>.

**Effect the Initial Concentration:** The adsorption studies were carried out at fixed parameters (T=298k, pH=8, bed height=15 cm, flow rate=4 cm<sup>3</sup>/s) and different initial concentration (50 – 200 mg/L). The equilibrium sorption capacities of the sorbents obtained from experimental are presented in figure 3. As seen from results, the sorption capacities of the sorbents increase with increasing phenol concentration while the adsorption yields of phenol showed the opposite trend. Increasing the mass transfer driving force and therefore the rate at which phenol molecules pass from the bulk solution to the particle surface<sup>18</sup>. The equilibrium uptake and adsorption yield were highest for ARH, which was expected because of the greater specific surface area and the microporous structure of ARH compared with RH<sup>16</sup>.

**The Effect of Different Bed Height,** The experiments were carried out with different RH and ARH bed heights (5 – 30 cm). The other initial parameters were kept constant [k=298 k, ph=8, initial concentration=100 mg/l and flow rate=4 cm<sup>3</sup>/s].

The curves for the above experiments are plotted as showed in figure 4, examining this figure, it can be seen that the capacity of adsorption of phenol on RH and ARH increased as the bed height increased. The increasing of bed height will provide an extra surface area for the adsorption process with the increasing of the RH and ARH bed depth at constant flow rate of solution and constant linear velocity, therefore the contact time will increase at bed depth increasing. The

increasing of the bed height will increase the adsorption capacity, because additional space will be available for the phenol molecules to be adsorbed on these unoccupied areas, furthermore, increasing the bed height will give a sufficient contact time for these molecules to be adsorbed on the RH and ARH surfaces<sup>3-11</sup>.

**The Effect of Different Temperature:** The results regarding the effect of temperature on the biosorption of phenol by RH and ARH are shown in Fig.(5). The results indicated that, biosorption organic compounds increase with increase in temperature from (298 – 338k) at constant at other parameters (pH = 8, initial concentration = 100 mg/L, bed height = 15cm and flow rate = 4 cm<sup>3</sup>/s). The biosorption of phenol was favorable at high temperature indicating the endothermic nature of the process. This might be due to the increase in the number of pores on the biomass surface at high temperature. The high temperature reduce the thickness of outer surface of the biosorbent and increase the kinetic energy of phenol molecules, as a result, the phenol molecules biosorbed easily in to the surface of the biomass<sup>9, 19</sup>.

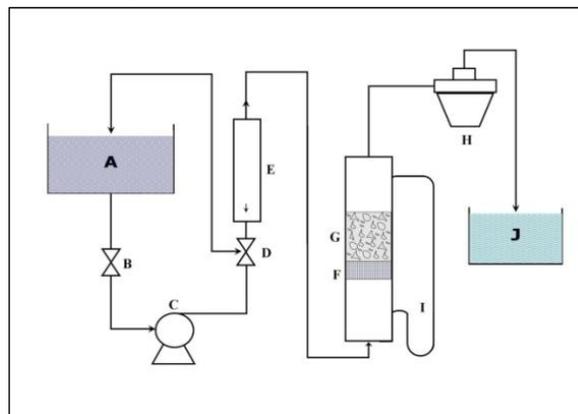


Figure - 1  
 Schematic Representation of Experimental Equipments.(A) Feed Tank,(B) Valve,(C) Pump,(D) Valve,(E) Rotameter,(F) Stainless Steel,(G)Packed Bed Absorber,(H) Cyclone,(I) Manometer,(J) Effluent Tank

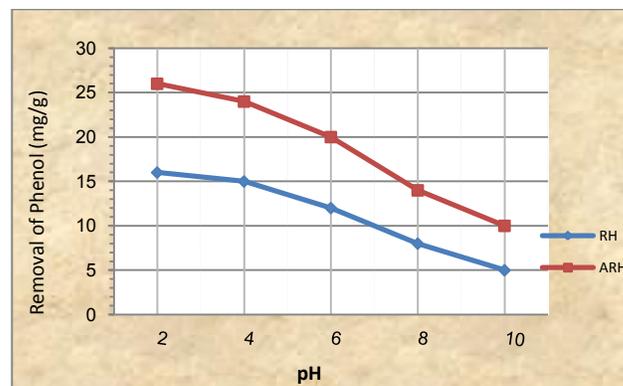


Figure-2  
 The effect of pH in the removal of heave oil by RH and ARH at 90 min

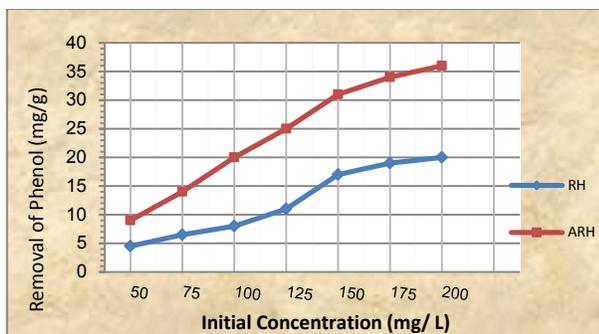


Figure-3

The effect of different initial concentration on the removal of phenol by RH and ARH at 90 min

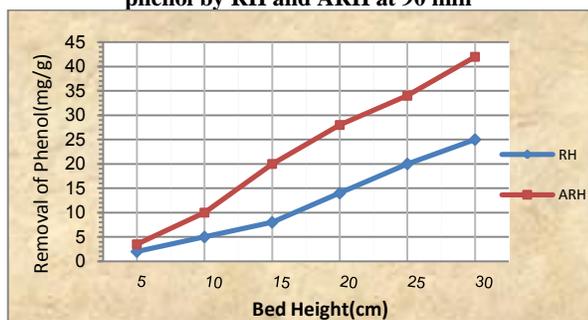


Figure-4

The effect of different bed height on the removal of phenol by RH and ARH at 90 min

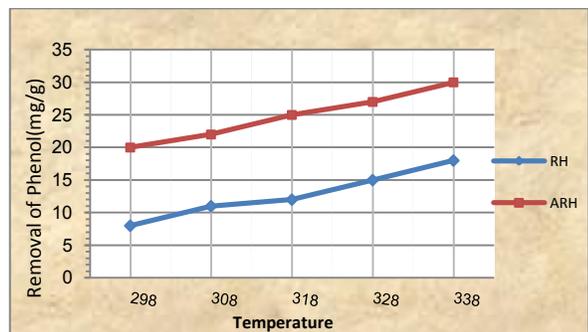


Figure-5

The effect of different temperature on the removal of phenol by RH and ARH at 90 min

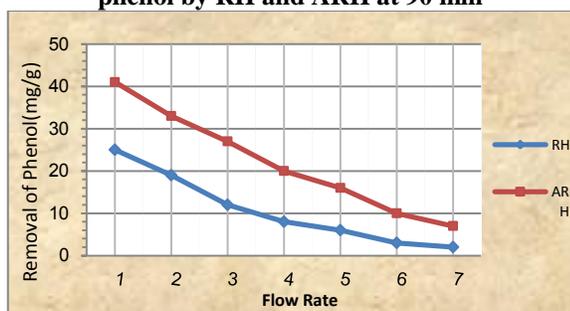


Figure -6

The effect of different flow rate on the removal of phenol by RH and ARH at 90 min

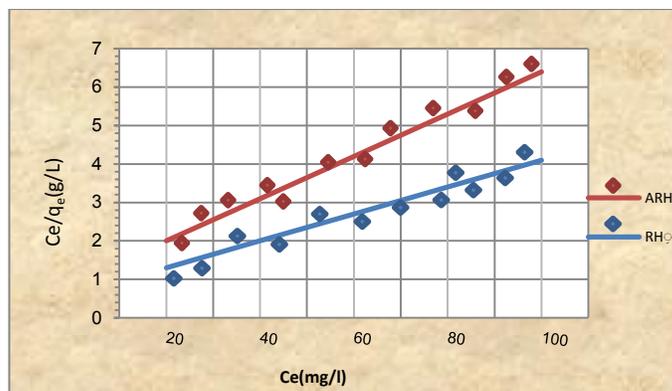


Figure-7

Langmuir plot for adsorption of phenol by RH and ARH, pH=8, T=298k, bed height =15 cm and flow rate =4 cm<sup>3</sup>/s



Figure-8

Freundlich plot for adsorption of phenol by RH and ARH, pH=8, T=298k, bed height =15 cm and flow rate =4 cm<sup>3</sup>/s

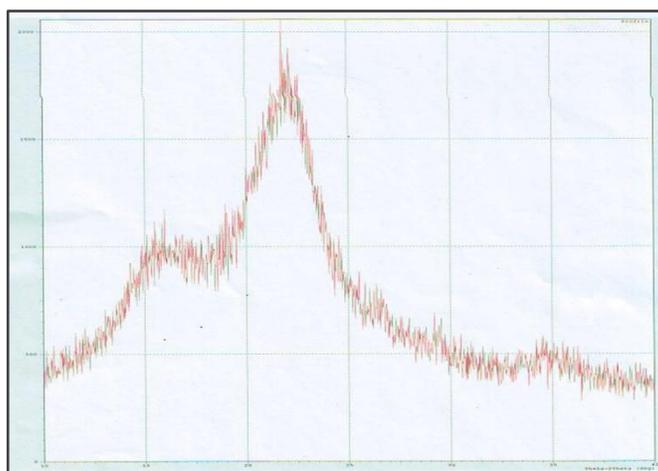
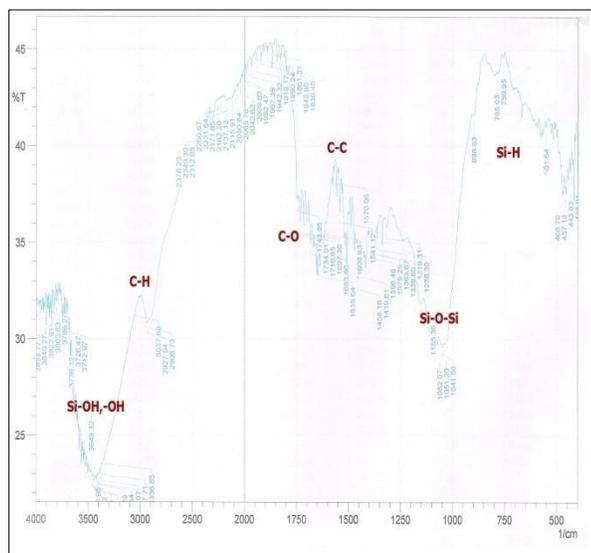


Figure-9

X-ray powder diffraction of RH



**Figure-10**  
**Infrared vibration spectrum FTIR of RH with phenol**

**The Effect Different Flow Rate:** Investigated the effect of adsorbent for the removal of phenol from aqueous solution. The experiments were carried out with different flow rate (1-7 cm<sup>3</sup>/s), with keeping other parameters are constant (T = 298k, pH= 8, bed height = 15 cm and initial concentration = 100 mg/l). Examining figure 6, the sorption capacity with different flow rate. It can be seen that there is a marked increase in the rate of adsorption and the capacity of the RH and ARH with decreasing flow rate. This was due to, a good available contact time will affect the amount of the capacity, that for low flow rate the aqueous solution molecules would have a sufficient contact time to occupy the space within the particles<sup>3,11</sup>.

**Adsorption Isotherm:** The isotherms are important and basic requirement for the analysis of adsorption systems, further more isotherms are used for design purposes. A series of experiments were performed to evaluate the biosorption of rice husk (RH) and activated rice husk (ARH) and further verified by using the Langmuir and Freund-lich isotherms.

**Langmuir Isotherm:** The Langmuir adsorption<sup>9,20</sup> is frequently applied for the biosorption of organic and pollutants (phenol) from aqueous solution. This model suggests that the biosorption onto the adsorbent surface is homogeneous in nature. According to Langmuir isotherm, the biosorbent surface occurred as monolayer biosorption on the homogeneous number of exchanging sites. This phenomenon describes the uniform biosorption energy on the biosorbent surface. The non- linear Langmuir isotherm equation can be presented in the following equation -

$$q_e = \frac{q_{max} \cdot C_e}{1 + b_1 \cdot C_e} \quad (3)$$

Equation (3) can be linearized as in equation (4)

$$\frac{C_e}{q_e} = \frac{1}{q_{max} \cdot b_1} + \frac{C_e}{q_{max}} \quad (4)$$

Where,  $q_e$  (mg/g) equilibrium capacity of sorption,  $C_e$  (mg/100 ml water) equilibrium concentration of heavy oil,  $q_{max}$  (mg/g) maximum sorption capacity and  $b_1$  (mg/g) constant related to affinity and energy binding sites. The plots of  $C_e$  versus  $\frac{C_e}{q_e}$  for phenol by RH and ARH as shown in figure 7. A comparison of isotherm parameters for both adsorbents RH and ARH are indicated that the adsorption favorable with the Langmuir isotherm model and showed that the ARH have greater ability to absorb.

**Freundlich Isotherm:** The Freundlich isotherm model is based on the assumption that the biosorption process takes place by interaction of the phenol molecules on the heterogeneous surface. There is a logarithmic decline in the energy of biosorption with increase in the occupied binding sites. The Freundlich isotherm equation is given as<sup>15</sup>.

$$q_e = K_f C_e^{1/n} \quad (5)$$

Equation (5) can be linearized as in equation (6)

$$\ln(q_e) = \ln(K_f) + \frac{1}{n} \ln(C_e) \quad (6)$$

Where,  $q_e$  (mg/g) equilibrium capacity of sorption,  $C_e$  (mg/g) equilibrium concentration of heavy oil,  $K_f$  coefficient provides an indication of the sorption, capacity and  $n$  coefficient related to intensity of sorption. The values of  $K_f$  and  $n$  are obtained from the intercept and slope of the graph between  $\ln q_e$  versus  $\ln C_e$  Fig.8. The values for both  $1/n$  and  $K_f < 1$  for both RH and ARH, indicates favorable adsorption with Langmuir and the Freundlich isotherm was fitted for adsorption of phenol, from the other hand the sorbent of ARH is better with respect to RH.

**The X-ray Diffraction Analysis:** The X-ray (XRD-6000, SHIMADZO, Japan) was generated from a cu k- $\alpha$  target with a current 40 mA and a potential of 30 kV. The scan was performed between  $2\theta$  values of  $10^\circ$  and  $40^\circ$  at scan rate of 2 degrees per minute. The X-ray diffraction patterns of RH and ARH show a broad hump between  $2\theta = 17^\circ - 28^\circ$  diffraction angle, while lacking any defined figure 9. The results indicate that RH and ARH are characteristic of amorphous substance, the heating of ARH at  $300^\circ\text{C}$  did not change the structure of amorphous silicon dioxide to a crystalline structure (observed at  $2\theta = 22^\circ$ ), such as quartz, cristobalite or tridimite. This result was also reported by other researchers<sup>21,22</sup>. Major constituents of rice husk are cellulose, hemi-cellulose, lignin and crude protein<sup>23</sup>.

**Investigation of Adsorbent Using FTIR:** The chemical structure of the adsorbent of vital importance in understanding the adsorption process. The FTIR technique is an important tool to identify the characteristic functional groups, which are instrumental in adsorption of aromatic

compounds<sup>5</sup>. The FTIR spectra of RH before and after heavy oil sorption were used to determine the vibrational frequency changes in the functional groups in the adsorbent. From figure 10, the adsorption peaks between (3336.85-3462.22cm<sup>-1</sup>) indicates the existence of free hydroxyl groups, the C-H stretching vibration around (2906.73-2927.94cm<sup>-1</sup>) indicates the presence of alkenes. The peaks between (1716.65-1743.65cm<sup>-1</sup>) corresponds to the C=O stretching that may be attributed to the hemicelluloses and lignin aromatic groups<sup>9</sup>. The C=C stretching vibrations between (1541.12-1697.36cm<sup>-1</sup>) indicative of alkenes and aromatic functional groups<sup>5</sup>. The peaks around (1041.56-1155.36cm<sup>-1</sup>) and (551.64-898.83cm<sup>-1</sup>), corresponds to Si – O – Si and Si – H groups respectively. The presence of polar groups on the surface is likely to give considerable cation exchange capacity to the presence of polar groups on the surface is likely to give considerable cation exchange capacity to the adsorbent. Analysis FTIR shows that the functional groups contribute to the adsorption of heavy oil on to the surface of RH<sup>5,24,25</sup>.

## Conclusion

Rice husk RH and activated rice husk ARH (pretreated by 13 M H<sub>2</sub>SO<sub>4</sub> and thermally treated at 573k) are proven to be potentially good scavenger and low – cost adsorbent for the removal of phenol from aqueous solution, at optimum parameters. Adsorption is dependent on the pH, temperature, initial concentration of phenol, bed height and flow rate. Experiments found that, the pretreatment of rice husk has increase specific surface of the developed adsorbents. The isotherm studies showed that adsorption data fitted reasonably well Langmuir and Freundlich isotherms. X-ray diffraction analysis, which indicates that the RH mainly consist of amorphous materials. Analysis FTIR shows the surface functional groups have significant effect on the sorption of phenol on the surface of adsorbent.

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