

## Original Research Article

Scalability of Palm Kernel Shell Derived Activated Carbon for the Remediation of Pb<sup>2+</sup> and 2, 4-DichlorophenolMustapha Omenesa Idris<sup>1\*</sup>, Edegebe Esosa Williams<sup>2</sup>, Abdulrahman Itopa Suleiman<sup>3</sup>, Felix Ebhodaghe Okieimen<sup>2</sup><sup>1</sup>Department of Pure and Industrial Chemistry, Kogi State University, PMB 1008, Anyigba, Nigeria<sup>2</sup>Department of Chemistry, University of Benin, PMB 1154, Benin City, Nigeria<sup>3</sup>Department of Biochemistry, Kogi State University, PMB 1008, Anyigba, Nigeria

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**Abstract:** Activated carbon is a widely used industrial adsorbent mainly composed of carbonaceous material with relatively high surface area and porous structure. This attributes allows it to preferentially adsorb organic materials and other non-polar compounds from a gas or liquid stream resulting to its significant application in many industrial processes. This study investigates the adsorption efficiency of acidic treated activated carbon and its untreated activated carbon on the adsorption of lead (Pb) and 2,4-Dichlorophenol (2,4-DCP). The activated carbon was prepared from palm kernel shell as the precursor and was characterized based on its porosity having value of 0.18, iodine number of 440.95 mg/g, surface area of 433.65 m<sup>2</sup>/g and surface acidity value of 0.20 mmol H<sup>+</sup>/g. The palm kernel shell activated carbon was subjected to various treatment conditions of (40°C, 60°C, 80°C) temperatures, (30 minutes, 1 hour, 2 hours) treatment time and (1 M, 2 M, 3 M) concentrations of HNO<sub>3</sub>. The most suitable condition for the acidic treatment was determined by subjecting each of the treated activated carbon to surface acidity analysis. The highest surface acidity value of 2.55 mmol H<sup>+</sup>/g was obtained at treatment condition of 80°C, 2 hours and using 3 M concentration of the acid while the lowest value of 0.65 mmol H<sup>+</sup>/g was obtained at 40°C, 2 hours and 1 M concentration. Effect of the surface acidic treatment of the adsorbent on adsorption was investigated by comparing the adsorption capacity of the highly treated adsorbent with the untreated adsorbent for the adsorption of lead and 2,4-DCP. It was revealed that the treated adsorbent is better for the adsorption of lead while the untreated adsorbent is better for the adsorption of 2,4-DCP.

**Keywords:** Palm Kernel Scalability Carbon.

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## 1.0 INTRODUCTION

The processing of agro-forest products generates substantial amounts of waste which can be harnessed using appropriate technologies that reduce, reuse, and recycle waste to churn out useful products like activated carbon [1]. Activated carbon, a widely used industrial adsorbent, is mainly composed of carbonaceous material with high surface area and porous structure [2]. This is due to its relatively high adsorptive capacity and porous structure which allows it to preferentially adsorb organic materials and other non-polar compounds from a gas or liquid stream, it has been found to have significant application in many industrial processes. These include, gold mining operations, carbonated drink manufacturing processes, edible oil treatment operations and wastewater treatment plant. These processing industries in Ghana however depend on supply of activated carbon from outside the country and this increases the cost of

production. The most commonly used raw materials for the production of activated carbon are coal (anthracite, bituminous and lignite) and wastes of vegetable origin (such as coconut shell, palm kernel shell, corn cobs etc), resources which are abundant in Nigeria. The fixed carbon content of these vegetable waste materials is quite high and hence can be used as feedstock for the production of activated carbon [3]. Activated carbon is produced by either the physical (steam) activation process or the chemical activation process. The physical activation process occurs in two stages: the carbonization of the precursor in an inert atmosphere and subsequently followed by activation of the crude char in the presence of a reactive gas like carbon dioxide or steam at elevated temperatures of about 900°C. This method accounts for the majority of activated carbon used in the world and the preferred raw material is coal. Chemical activation on the other hand is a one stage process involving the use of a

chemical activating agent (an acid or a base) before carbonizing. The chemical agent acts as a dehydrating agent thereby limiting the formation of tar during the carbonizing step [4]. Chemical activation is also carried out at relatively lower temperatures which enhance the formation of porous structure. For both methods, the carbonizing stage enriches the carbon content and creates an initial porosity while the activation stage, further develops the porosity in the final product [5]. The choice of raw material for the production of activated carbon depends on the price, purity, potential extent of activation and the availability and stability of the supply. Nigeria is among the top 20 producers of palm oil, a commodity that generates a lot of waste. For every tonne of palm oil produced, 800kg of palm kernel shell is generated [5]. A total of 96,000 tonnes of palm kernel shells were generated from palm oil producing operations in the country in 2010. Palm kernel shells are mainly used as fuel or fuel supplement in the small-scale traditional oil processing industries. In large scale industries that use a boiler, only 5% of the shells generated in the established palm oil industries are burnt together with the fibre to generate heat for the boiler [6]. In this study, palm kernel shell (PKS) is proposed as a starting material for the production of activated carbon because of the availability of substantial amount of shell all year round, the cheap cost of the raw material, the relatively high fixed carbon content and low ash content. The activated PKS was used to study the adsorption capacity on heavy metal and organic contaminant.

## 2.0 MATERIALS AND METHODS

### 2.1 Collection of Sample

The palm kernel shell used for this work was collected from palm oil processing mill site, Ugbowo, Benin City. The sample was soaked in water for 24 hours after which they were washed to remove all dirt and was sun dried for 24 hours. The samples were crushed into smaller units. They were further oven dried at 105°C to remove excess moisture content before carbonization.

### 2.2 Carbonization

The samples were carbonized on a muffle furnace at temperature of 600°C for 2 hours. After carbonizing, the sample was allowed to cool at room temperature. It was grinded into powder form by the use of wooden mortar and pestle, and was sieved through a 1.18mm mesh. This was kept safe in a closed container prior to the activation process.

### 2.3 Activation

Chemical activation with base was employed. The carbonized sample was soaked in 2M KOH solution in a ratio of 1:20 of weight of carbonized sample to volume of KOH solution for 12 hours. The mixture was filtered through a clean white silk and was allowed to drip dry for three days. This was later activated in a muffle furnace at a temperature of 400°C

for 3 hours. The activated carbon was allowed to cool at room temperature, washed and was kept in a tightly closed container before the characterization processes.

## 2.4 Characterization

### 2.4.1 Porosity Determination

2.0 gram of the activated carbon was transferred into a measuring cylinder (10 ml) and the volume of the particle was recorded. This sample was placed into a beaker containing 20 ml of distilled water and boiled for 5 minutes (to displace air in the container). The content was filtered, superficially dried and weighed. The increase in weight of the sample divided by the density of water gave pore volume. Porosity was calculated by dividing the pore volume of the particle with the total volume of particle [7].

### 2.4.2 Surface Area Determination

The Iodine number was first determined and the surface area was calculated from a mathematical relation between surface area and iodine number as reported by Mainowski *et al.* [8]. In determining the iodine number, 10 ml of 5% by weight HCl was added to 1g of activated carbon in a conical flask and was allowed to boil gently for 30 seconds to remove any impurities which may interfere with the test results. After the solution was cool at room temperature, 100 ml of 0.1N iodine solution was added into the flask. The content was shaken vigorously for 30 seconds and was filtered quickly into a beaker. 20 ml of the filtrate was titrated against 0.1N sodium thiosulphate using starch as indicator. Similarly, the quantity of sodium thiosulphate needed to titrate the blank was determined. All titration was carried out in triplicate and the average titre value obtained in each case was calculated. The iodine number was defined as the quantity of iodine adsorbed in (mg/g carbon) as residual iodine concentration.

$$\text{Iodine number} = \frac{(A - B)}{B} \times \frac{VM}{W} \quad (126.91)$$

A = Volume of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> used for blank

B = Volume of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> used for activated carbon

V = Volume of iodine solution used

M = Molarity of iodine solution used

W = Weight of activated sample

Surface Area = (0.9946) I.N-4.91 [8].

I.N = Iodine number.

### 2.4.3 Surface Acidity Determination

In determining the surface acidity, 0.1N NaOH solution (100 ml) and 5 gram of the activated carbon were mixed in a conical flask. The mixture was shaken for 4 hours with a conical flask shaker to ensure that the solution had come to equilibrium. The solution was back titrated with standard 0.1N HCl solution using a pH indicator. The surface acidity of the activated carbon was calculated from the titre value [9].

Surface Acidity = The volume of NaOH used (ml) × 0.1N (the normality of the NaOH solution) – the volume of HCl used × 0.1N (the normality of NaOH solution) [9].

## 2.5 Surface Acidic Treatment

The Surface acidic treatment was carried out in three different conditions; temperature of heating, duration of heating and concentration of HNO<sub>3</sub> used. 1:20 of the weight of activated carbon to volume of HNO<sub>3</sub> was adopted for all the treatment conditions. 5 gram of the Activated carbon was weighed and dissolved in 100 ml of 1M HNO<sub>3</sub>. This was heated in a thermostatic hot plate at 40°C for 30 minutes. The mixture was allowed to cool at room temperature and filtered through a Whatmann filter paper, washed and kept tightly closed in a small plastic bottle. The same process was repeated for all conditions of temperatures; 40°C, 60°C and 80°C, all conditions of concentrations of HNO<sub>3</sub>; 1M, 2M and 3M for 30 minutes treatment time. Similarly, the whole treatment process and conditions were done for 1 hour and 2 hours treatment times respectively. After all treatments, a total twenty-seven (27) acid treated activated carbon were obtained based on different treatment conditions. Each sample was labeled based on their treatment conditions. E.g. 1M/40°C/30mins, 2M/40°C/30mins, 3M/40°C/30mins etc. Surface acidity test was conducted on the 27 samples as describe in section 2.4.3 to identify which treatment condition was more effective.

## 2.6 Adsorbtion of lead (Pb)

The treated sample with the highest surface acidity value and the untreated sample were used to study the effect of surface acidity of the coconut shell activated carbon on the adsorption of Lead. AAS was used to detect the concentration of the lead after adsorption.

## 2.7 Adsorption of 2,4-Dichlorophenol (2,4-DCP)

The treated sample with the highest surface acidity value and the untreated sample were used to study the effect of surface acidity of the coconut shell activated carbon on the adsorption of 2,4-DCP. UV-Vis spectrophotometer was used to calibrate the curve for standard concentrations of 2,4-DCP ranging from 1ppm to 20ppm. The UV-Vis spectrophotometer was also used to detect the absorbance values of the 2,4-DCP after adsorption. The concentrations were extrapolated from the standard calibration curve [11].

## 2.8 Adsorption Procedure

The acidic treated activated carbon that shows highest surface acidity test and the untreated activated carbon were used as the adsorbent. 1.0g of the adsorbents were mixed with 100 ml of 20mg/L (20ppm) lead, 100 ml of 20mg/L 2,4-dichlorophenol and 100 ml equimixture of 20mg/L lead and 20mg/L 2,4-dichlorophenol in a separate conical flask respectively. The flask was shaken with a conical flask shaker for 3 hours to ensure equilibrium adsorption. The adsorbents were then separated from the solution by filtration through a whatman filter paper. The residual concentrations of the lead and the 2,4-DCP was analysed with AAS and UV-Vis spectrophotometer respectively. The equilibrium adsorption capacity ( $q_c$ ) is defined as the amount of adsorbate per gram of adsorbent and calculated by

$$q_c = \frac{(C_1 - C_2)V}{m} \quad (\text{mg/g})$$

C<sub>1</sub> = Concentration of adsorbate before adsorption (mg/L)

C<sub>2</sub> = Concentration of adsorbate after adsorption (mg/L)

V = Volume of the solution

m = Mass of the adsorbent (g) [10]

## 3.0 RESULTS AND DISCUSSION

**Table-3.0: Characteristics of the untreated palm kernel shell activated carbon**

S/no	Parameters	Results
1	Porosity	0.18
2	Iodine Number (mg/g)	440.95
3	Surface Area (m <sup>2</sup> /g)	433.65
4	Surface Acidity (mmol H <sup>+</sup> /g)	0.20

## 3.1 Physico-chemical Properties of the coconut shell Activated Carbon

Some physical and chemical properties of the untreated palm kernel shell activated carbon are given in table 3.0. The porosity of the activated carbon was found to be 0.18. The porosity was determined from the pore volume. Large pore volume enhances adsorption of materials[12] The iodine number which is used to estimate the surface area was found to be 440.95 mg/g. Iodine number gives an estimate or approximate value of the surface area, the surface area was determined from their relation as reported by Mianowski *et al*. [8]

to be 433.65 m<sup>2</sup>/g. The value of the surface area suggests a fairly large area surface for the carbon. Generally, the larger the surface area of the adsorbent, the better its adsorption performance.

It was also reported by Bantista *et al*. [13] that micropores are responsible for large surface area. The higher the iodine number, the higher the surface area and the better the adsorption capacity. The surface acidity of the untreated carbon was determined to be very low at 0.31 mmol H<sup>+</sup>/g which is a very small value compared to the various values after various treatment

conditions. The surface groups play a key role in the surface chemistry of activated carbon and adsorption is

depended upon their surface chemistry [13].

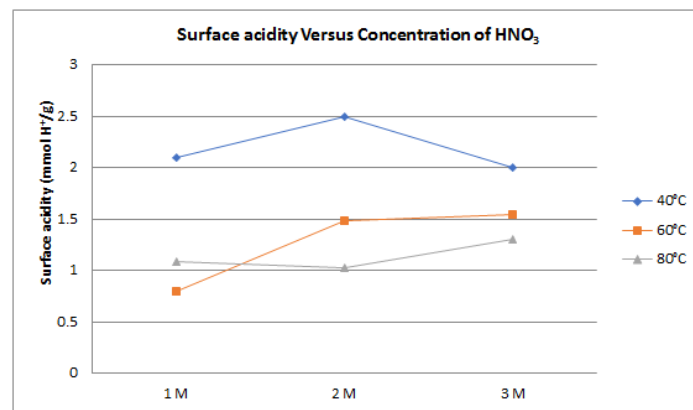
**Table-3.1: Acidic treatment for 30 minutes**

Temp (°C)	Conc. (M)	Surface Acidity (mmol H <sup>+</sup> /g)
40	1	2.10
	2	2.50
	3	2.00
60	1	0.80
	2	1.49
	3	1.54
80	1	1.09
	2	1.03
	3	1.31

**3.2 Effect of Acidic Treatment on the Surface Acidity**

The surface acidities of the adsorbent at various treatment conditions for 30 minutes duration of treatment are shown in Table 3.1. It reveals that for 30 minutes treatment, 2 M HNO<sub>3</sub> and treatment temperature of 40°C gave the highest surface acidity value of 2.50 mmol H<sup>+</sup>/g and lowest acidity value of 0.80 mmol H<sup>+</sup>/g at treatment temperature of 60°C and 1 M HNO<sub>3</sub>.

The effect of 30 minutes treatment on the surface acidity was explained in figure 3.0. It was observed that all treatments at 40°C gave favourable effect on the surface acidity. Treatments carried out at 60°C and 80°C were not as effective as that done at lower temperature. It therefore implies that at 30 minutes duration of treatment, 40°C treatment temperature increases the surface acidic group of the activated carbon much better than at higher temperature.



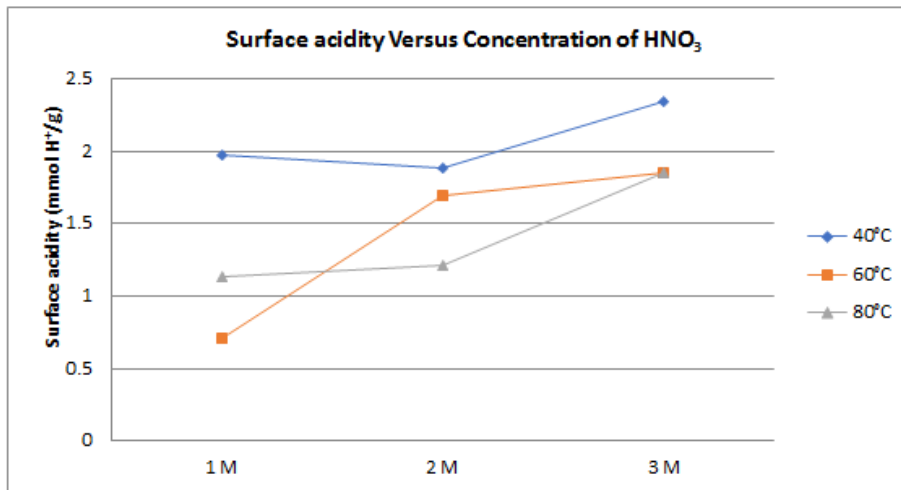
**Fig-3.0: Effect of 30 minutes treatment on the surface acidity**

**Table-3.2: Acidic treatment for 1 hour**

Temp (°C)	Conc. (M)	Surface Acidity (mmol H <sup>+</sup> /g)
40	1	1.98
	2	1.89
	3	2.35
60	1	0.71
	2	1.70
	3	1.85
80	1	1.13
	2	1.21
	3	1.85

Table 3.2 reveals that 40°C treatment temperature and 3 M HNO<sub>3</sub> gave the highest surface acidity of 2.35 mmol H<sup>+</sup>/g. The lowest surface acidity of 0.71 occurs at 60°C treatment temperature and 1 M HNO<sub>3</sub>.

Figure 3.1 explains the effect of 1 hour treatment on the surface acidity. It shows that adsorbents treated with 3 M HNO<sub>3</sub> at the three temperatures gave a fairly good effect on their surface acidity. It also reveals that all treatment condition gave better surface acidity value for 40°C.



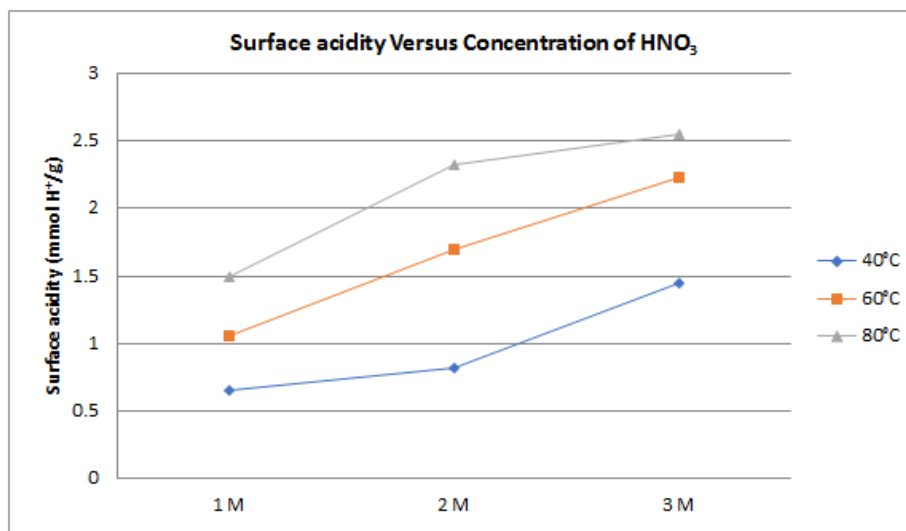
**Fig-3.1: Effect of 1 hour treatment on the surface acidity**

**Table-3.3: Acidic treatment for 2 hours**

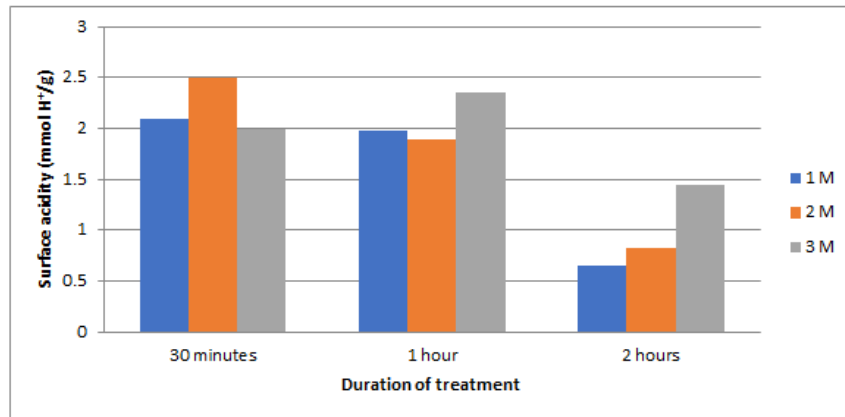
Temp (°C)	Conc. (M)	Surface Acidity (mmol H <sup>+</sup> /g)
40	1	0.65
	2	0.82
	3	1.45
60	1	1.06
	2	1.70
	3	2.23
80	1	1.50
	2	2.32
	3	2.55

The surface acidities of the adsorbent at various treatment conditions of 2 hours are shown in Table 3.3. In the treatment, it was revealed that for 2 hours treatment, 3 M HNO<sub>3</sub> and treatment temperature of 80°C gave the highest surface acidity value of 2.55 mmol H<sup>+</sup>/g and lowest acidity value of 0.65 mmol H<sup>+</sup>/g at treatment temperature of 40°C and 1 M HNO<sub>3</sub>. Figure 3.2 explains the effect 2 hours treatment on the surface

acidity. It can be observed that all treatments at 3 M HNO<sub>3</sub> impacted better surface acidity on the surface of the activated carbon, but that of 2 M at 80°C and 3 M at 60°C also gave a good surface acidity value. It is suggested that at 2 hours duration of treatment, 3 M HNO<sub>3</sub> is very effective in increasing the surface acidic group of the activated carbon much better than using 2 M and 1 M concentrations of the acid.



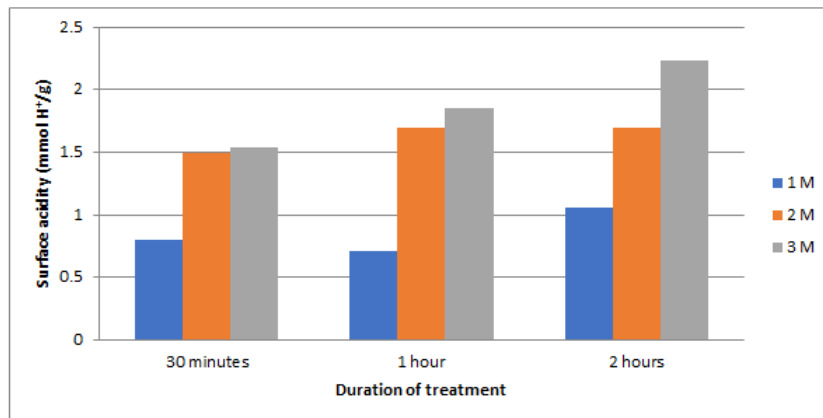
**Fig-3.2: Effect of 2 hours treatment on the surface acidity**



**Fig-3.3 : Effect of 40°C treatment temperature on the surface acidity**

The chart representations of the effect of 40°C treatment temperature on the surface acidity of the adsorbent are given in Figure 3.3. It was observed that among the three treatment times, treatment time of 30 minutes and 1 hour gave higher surface acidity values.

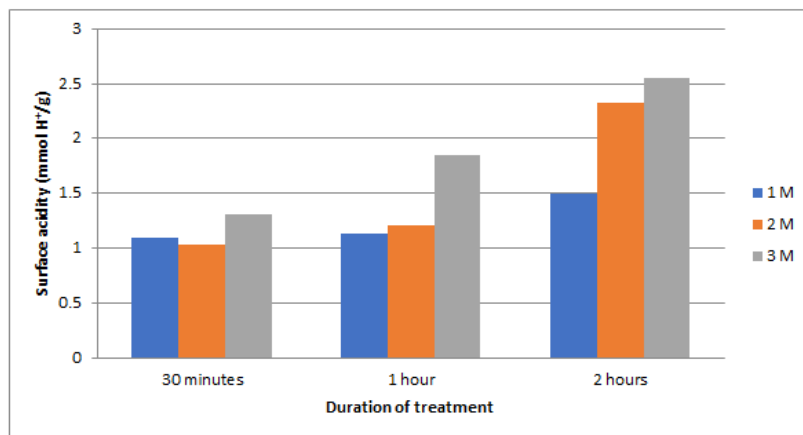
It was observed that 2 hours treatment at this temperature was not very effective in impacting acidity on the surfaces of the palm kernel shell activated carbon. But at this temperature for 2 hours using 3 M of the acid, fairly good surface acidity value was obtained.



**Fig-3.4: Effect of 60°C treatment temperature on the surface acidity**

Figure 3.4 shows the effect of 60°C treatment temperature on the surface acidity of the adsorbent. It was observed that using 3 M HNO<sub>3</sub> for the treatment for 30 minutes, 1 hour and 2 hours gave an increasing order of surface acidity on the adsorbent. 30 minutes

treatment at this temperature was determined not to be very effective in increasing the acidic surface of the adsorbent. 1 hour using 3 M and 2 hours using 3 M and 2 M of the acid respectively gave a fairly good surface acidity value.



**Fig-3.5: Effect of 80°C treatment temperature on the surface acidity**

Figure 3.5 shows the effect of 80°C treatment temperature on the surface acidity. The surface acidity of the adsorbent was found to be highest using 3 M of the acid at this condition. It suggests that for effective surface acidic treatment of the adsorbent to be obtained, 3 M of the acid will be more favourable at this condition.

**Table-3.4: Adsorption of lead from 20mg/L lead solution**

Absorbents	q <sub>c</sub> (mg/g)
TA <sub>S</sub>	1.930
UA <sub>O</sub>	1.863

TA<sub>S</sub> = Treated adsorbent with the highest surface acidity

UA<sub>O</sub> = Untreated adsorbent

q<sub>c</sub> = Equilibrium adsorption capacity

**Table-3.5: Adsorption of 2,4-DCP from 20mg/L 2,4-DCP solution**

Absorbents	q <sub>c</sub> (mg/g)
TA <sub>S</sub>	1.820
UA <sub>O</sub>	1.900

TA<sub>S</sub> = Treated adsorbent with the highest surface acidity

UA<sub>O</sub> = Untreated adsorbent

q<sub>c</sub> = Equilibrium adsorption capacity

**Table-3.6: Adsorption of lead from 20mg/L equimixture lead/2,4-DCP solution**

Absorbents	q <sub>c</sub> (mg/g)
TA <sub>S</sub>	0.982
UA <sub>O</sub>	0.982

TA<sub>S</sub> = Treated adsorbent with the highest surface acidity

UA<sub>O</sub> = Untreated adsorbent

q<sub>c</sub> = Equilibrium adsorption capacity

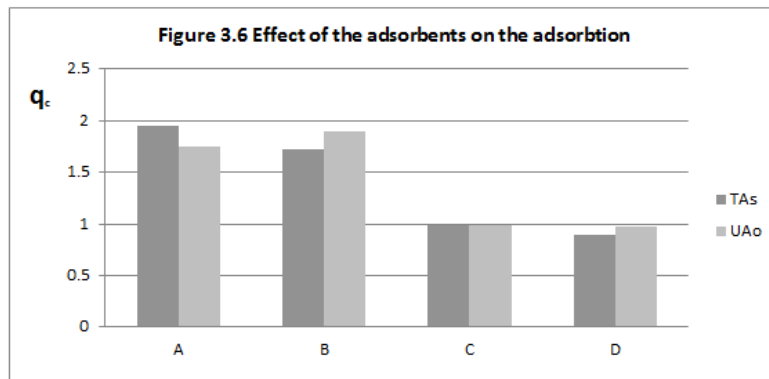
**Table-3.7: Adsorption of 2,4-DCP from 20mg/L equimixture lead/2,4-DCP solution**

Absorbents	q <sub>c</sub> (mg/g)
TA <sub>S</sub>	0.900
UA <sub>O</sub>	0.960

TA<sub>S</sub> = Treated adsorbent with the highest surface acidity

UA<sub>O</sub> = Untreated adsorbent

q<sub>c</sub> = Equilibrium adsorption capacity



**Fig-3.6 : Effects on Adsorptions**

TA<sub>S</sub> = Treated adsorbent with the highest surface acidity

UA<sub>O</sub> = Untreated adsorbent

q<sub>c</sub> = Equilibrium adsorption capacity

A = Adsorption of lead from 20mg/L lead solution

B = Adsorption of 2,4-DCP from 20mg/L 2,4-DCP solution

C = Adsorption of lead from 20mg/L equimixture lead/2,4-DCP solution

D = Adsorption of 2, 4-DCP from 20mg/L equimixture lead/2,4-DCP solution

### 3.3 Effect of the Acidic Treatment on Adsorption

The adsorption capacity of the treated and the untreated adsorbents on lead and 2,4-DCP are shown in figure 3.6 above. For Lead adsorption, it was observed that the treated adsorbent adsorbed better than the

untreated. This suggests that acidic treatment of palm kernel shell activated carbon increases the effect on lead adsorption. Zhao *et al.* [14] reported that treatment of activated carbon by oxidation using HNO<sub>3</sub> followed by heat treatment affects its adsorption capacity for Cr (VI) by an increasing factor of up to 1.4. Chun *et al.* [15] also reported that acidic surface functional groups on active carbon are essential for the adsorption of heavy metals due to chelating attribute of these groups where alkaline – earth metal cations can be integrated with the group to form complexes. In the adsorption of 2,4-DCP, it was observed that the untreated carbon adsorbs much better compared to the treated adsorbent. This was as reported by Bansal *et al.* [16] who attributed the decrease in phenol adsorption with increase oxidation of activated carbon to the increasing hydrophilic character of the oxidized carbon. Usually, the hydrophilic property of adsorbents is related mainly to

the polarity of adsorbents and the polarity is derived mostly from surface groups (mainly oxygen containing groups). As a result, water adsorption is promoted by acidic surface group while enhancing negative effect on 2,4-DCP adsorption. Section C in figure 3.6 and section D reveals the adsorption capacity of the treated adsorbent and the untreated adsorbent in the adsorption of lead and 2,4-DCP from equal mixture of 20mg/L of the two adsorbates. It is observed that in Lead adsorption, both the treated and the untreated adsorbent gave equal adsorption capacity. However, their adsorption capacities were low. It can be suggested that the low adsorption capacity was as a result of the fact that both lead and the 2,4-DCP were competitively adsorbed.

## CONCLUSION

Acidic treatment of activated carbon from palm kernel shell shows a more effective treatment on the surfaces at 80°C temperature and 2 hours treatment time using 3 M of the HNO<sub>3</sub>. The acidic treated adsorbent displays a higher adsorption capacity for lead adsorption than the untreated adsorbent while in the adsorption of 2,4-Dichlorophenol, the acidic treated adsorbent shows a lower adsorption capacity for 2,4-Dichlorophenol.

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