

## Development and Characterization of Adsorbent from Rice Husk Ash to Bleach Vegetable Oils.

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**Abstract:** The study is aimed at characterization and development of adsorbent from rice husk ash to bleach vegetable oil. The vegetable oil bleached was groundnut oil, palm kernel oil and palm oils. Rice husk sample were pre-treated with different concentration of HCl. The characterization of the rice husk ash was tested by varying the temperature and time interval before it was subsequently calcined at 600 °C for three hours (i.e. having a constant temperature and time interval for calcinations). The optimum temperature and burning time was observed to be 600 °C for 3 hrs. A yield of 2.5M HCl was recorded as the best bleaching potential of palm kernel and palm oil and 2M HCl for groundnut oil. The result obtained showed that acid treatment improve the bleaching potential of the rice husk ash. Graphical interpretation of results obtained indicates that the unbleached oil is directly proportional to the wavelength.

**Keywords:** Characterization, Development, Adsorbent, Rice husk and Vegetable oil.

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### I. Introduction

Rice (*Oryza Sativa*) is grown over vast area of land around the world and is a major staple food for more than a half of the world population (Juliano, 1985). The Asian continent accounts for approximately 90 percent of rice production and is also the major customer. However, Rice is cultivated in virtually all the agro-ecological zones in Nigeria. Significant improvement in rice production in Nigeria occurred in 1980 when output increased to about a million tons while area cultivated and yield rose to 550 thousand hectares and 1.98tonnes per hectares respectively (Tunji, et al., 2007). About 10<sup>8</sup> tons of rice husk is generated annually in the world. In Nigeria, about 2million tones of rice is produced annually, while in Niger State, about 96000 tons of rice grains is produced in 2000 (Alhassan, 2005).

Global production of rice, the majority of which is grown in Asian, is approximately 550million tons/year. The milling of rice generates a waste material, while husk is the material surrounding the rice grain. The annual Worldwide output of rice husk is approximately 80million tons, which corresponds to 3.2 million tons of silica /year globally (Natarajan, et al., 1998).

Rice husk are rich in cellulose (28-36 %), crude fibre (34.5-45.9 %) and ash (13.2-21.0 %) (Juliano, 1985). The environmentally sound disposal and use of large quantities of hull is a challenging issue for rice processors around the world. The rice hull energy content at 14.0 % moisture content is 11.9 – 13.0MJ/Kg (5,116.5-5,589.4 Btu/lb) (Velupillai *et al.*, 1997). In developing countries, like India, where rice milling industries are small and scattered, hulls are used as a part of brick kilns, as an ingredient in dung-cake, as components in goldsmith or blacksmith furnaces or as a fuel for water heating systems (GovindRao, 1980). Managing rice husk is a problem especially in developing countries like Nigeria, where there is no any proper method of waste disposal in place. There is therefore the need to seek for way of converting the waste into hotter resources. Development of adsorbent from the rice husk is considered a perfect means of controlling the problems of rice husk as a waste.

Rice husk ash (RHA) is an end product of the combustion of rice husk. Rice husk ash has good adsorbent properties because of its high silica content (Velupillai *et al.*, 1997). Rice husk ash has been successfully applied as adsorbent for several lipid components such as phospholipids (Brown and Snyder, 1989), lute in (Proctor and Palaniappan, 1990), palmitic and oleic acids (Ooi and Leong, 1991), carotene (Liew *et al.*, 1993) and saturated fatty acids. The type of ash varies considerably depends on the techniques employed for calcinations. The silica in the ash undergoes structural transformation depending on the temperature during combustion. At 600 °C amorphous silica is formed, whereas 700 °C is a transition region from amorphous to crystalline silica while at 800 °C and above till the melting point of the ash, only crystalline silica is formed.

This study is focus on the development of adsorbent from rice husk for the bleaching of vegetable oil using various pretreated and untreated samples.

## **II. Research Methodology**

### **Preparation of the Rice Husk**

The rice husk was washed thoroughly with de- ionized water to remove dirt and then dried in an oven for three and the half hours at 130 °C.

### **Pre-treatment**

60 g of the clean and dried rice husks each was treated with different concentration of HCl in the range of 0.5-2.5M at 0.5M interval. Acid leaching was performed by boiling at 90 °C for 3 hours with acid/rice husk ratio 10 ml: 1g. After leaching, the Pre-treated rice husk were washed in plenty of distilled water and tested with pH meter for neutrality and then dried in the oven at 110°C for three and the half hours. The colors of the pre-treated rice husk were observed.

### **Calcination process**

The pre-treated samples plus untreated samples of the rice husks were charred in the oven at 110°C for four hours. The ash was prepared by burning the rice husks inside a muffle furnace at 600 °C for 3 hours which was obtained by variation of temperature from 500-800 °C at an interval of 100 °C and time of the ash (1, 2 and 3 hours), in a porcelain crucible placed in an electric furnace and then cooled at room temperature. The selected temperature and time (600 °C for 3 hour) gave the best characterization results in terms of ash content, fixed carbon, and moisture content and from literature since we are interested in amorphous silica and not crystalline silica in terms of ash content.

### **Bleaching of Vegetable Oils**

0.5 g of the powdered ash from each sample was used to bleach 20 ml each of groundnut oil, palm oils and palm kernel oil. Bleaching was carried out at a constant temperature and time at 80 °C for 30 minutes respectively with constant stirring using magnetic stirrer on a magnetic hotplate. It was also followed by filtering the slurry using a vacuum pump. The filtered oil samples were then analyzed using a spectrophotometer at different wavelengths at the visible region between 380 nm-800 nm.

### **Characterization of Rice Husk Ash, Bulk Density Determination**

A measuring cylinder (ml) was used to measure a certain amount of water. Another measuring cylinder is then used to measure the rice husk ash which is then poured into the water in the first measuring cylinder, the volume of the water increases. The volume in the water formerly in the cylinder ( $V_1$ ) then subtracted from the new volume of the water ( $V_2$ ) and the change in the volume is then divided by known weight of the rice husk ash, then the bulk density is obtained. (Oyetola and Abdullahi, 2006)

### **Characterization of the Developed Adsorbent**

The following test was carried out in order to fully specify the characterization of activated carbon for the rice husk.

### **Determination of Ash Content**

This was carried out in accordance to standard ASTM D 2866 (1987) method. 20 g of the dry pre-treated sample was placed in a porcelain crucible, weigh of crucible plus weight of dry rice husk was noted and recorded as  $W_2$ . The sample was then transferred into a muffle furnace (Apex, 1999) set at a temperature of 600 °C for 3 hours after which it was removed and allowed to cool at room temperature. The crucible and its content were reweighed and the weight was recorded as  $W_3$ . This process was carried out for all samples. The ash content was calculated using the formula;

$$\text{Ash content} = \frac{W_{ash}}{W_{RH}} \times 100 = \frac{W_2 - W_3}{W_0} \times 100$$

Where  $W_{ash}$  = weight of ash after heating =  $W_2 - W_3$ ,  $W_0$  = Original weight of sample.

### **Moisture Content Determination**

This procedure is in accordance with standard ASTM (1987) method. 20 g of sample were weighed into a paddy dish and the weight of the sample plus paddy dish was noted as  $W_2$ . The sample is then placed in a porcelain crucible and charred into the muffle furnace at a temperature of about 600 °C for 3 hours. It was then cooled at room temperature and it was then placed back into a paddy dish and reweighed.

$$\text{Moisture content (MC)} = \frac{W_2 - W_3}{W_2 - W_1} \times 100$$

Where  $W_2$  = Original weight of sample + Crucible.

$W_1$  = Weight of Crucible.

$W_3$  = Dried weight of sample + Crucible.

#### **Yield of charcoal**

This was obtained by calculation (Ekanem, 1996)

$$\text{Yield (Ych)} = \frac{(\text{wt of S+C})b - (\text{wt of S+C})a}{(\text{wt of S+C})b} \times 100$$

Where (wt of S+C)b = Weight of sample +Crucible before ashing.

(Wt of S+C)a = Weight of sample + Crucible after ashing.

#### **Fixed carbon determination**

This was conducted according to the procedure in ASTM (1987). The fixed carbon content was determined by;

$$\text{Fixed carbon content} = \frac{Y_{ch} - A_c - M_c}{Y_{ch}} \times 100$$

#### **Pore volume determination**

The pore volume was determined according to smith (1981). 20 g of the sample was weighed into a paddy dish and the weight of the sample plus paddy dish was noted and recorded as  $W_0$ . During pre-treatment some amount of acid was poured into the sample and it was boiled for about 3hours for a temperature of 90 °C. After the air in the pore had been displaced, the weight was recorded as W. This procedure was carried out for all samples.

$$\text{Pore volume (cm}^3\text{/g)} = (W_{D_s} / \rho_w) \text{ or } \left( \frac{W_0 - W_1}{W - W_1} \right) / \text{density of water.}$$

Where  $W_1$ =weight of paddy dish,  $W_0$ = initial weight of sample and paddy dish, W= final weight of sample and paddy dish,  $\rho_w$ =density of water 1 g/cm<sup>3</sup>,  $W_{D_s}$ =weight of dry sample.

#### **Determination of Porosity and Void Volume**

The porosity is determined according to smith (1981) by calculation.

$$\text{Porosity (P}_0\text{)} = V_v / V_t$$

Where  $V_t$ = Total volume and  $V_v$  is the void volume.

The void volume was obtained first by determining the total volume of the cylinder ( $V_t = \pi r^2 h$ ) used for the experiment and also the volume of the solid is determined i.e. the activated carbon/ash used.

$$V_s = \frac{M_s}{G_s \rho_w}$$

Where r = radius

h= height

$M_s$ = Mass of solid

$G_s$ = Specific gravity

$\rho_w$ = Density of water

Then Void Volume ( $V_v$ ) was obtained as

$$V_v = V_t - V_s$$

### **III. Results And Discussions**

The results of the characterization and development of adsorbent from rice husk ash to bleach vegetable oil are presented in tabular and graphical forms.

#### **Characterization of rice husk carbonized ash**

Characterization of rice husk carbonized ash is characterized at different temperature ranging from 500-800 °C and at different time interval. The results obtained on various analysis conducted in this study are presented below.

**Table 1: Characterization of Rice Husk Carbonized Ash at Different Temperature (Constant Time=1hour and mass of sample=20 g)**

Temperature °C	Ash content (g)	Porosity	Moisture content (%)	Fixed carbon (%)	Charcoal yield (%)
500	6.78	0.938	7.16	86.50	66.10
600	5.88	0.939	7.10	87.82	70.60
700	5.72	0.940	7.08	87.94	71.40

**Table 2: Characterization of Rice Husk Carbonized Ash at Different Temperature (Constant Time=2 hours)**

Temperature °C	Ash content (g)	Porosity	Moisture content (%)	Fixed carbon (%)	Charcoal yield (%)
500	5.451	0.942	7.08	82.81	72.75
600	5.740	0.942	7.03	82.09	71.30
700	5.540	0.945	6.99	82.67	72.30

**Table 3: Characterization of Rice Husk Carbonized Ash at Different Temperature (Constant Time=3 hours)**

Temperature °C	Ash content (g)	Porosity	Moisture content (%)	Fixed carbon (%)	Charcoal yield (%)
500	5.420	0.949	7.28	82.58	72.90
600	5.999	0.951	6.63	81.95	70.01
700	5.131	0.950	6.63	84.17	74.35

**Table 4: Characterization of Rice Husk Carbonized Ash at Different Time Interval (Constant Temperature=600 °C)**

Time Hours	Ash content (g)	Porosity	Moisture content (%)	Fixed carbon (%)	Charcoal yield (%)
1	5.880	0.939	7.10	87.82	70.60
2	5.740	0.942	7.03	82.09	71.30
3	5.999	0.951	6.63	81.95	70.01

**Table 5: Characterization of the Developed Adsorbent using HCl as Activating Agent**

Properties	0.5M	1M	1.5M	2M	2.5M
Ash Content (g)	10.03	10.47	10.86	10.85	10.83
Moisture Content (%)	3.01	3.11	3.24	3.24	3.14
Yield of Charcoal (%)	75.95	76.1	76.57	76.61	75.83
Fixed Carbon (%)	82.83	82.16	81.59	81.61	81.58
Porosity	0.935	0.935	0.937	0.936	0.937
Bulk Density (g/cm <sup>3</sup> )	0.37	0.36	0.34	0.30	0.34

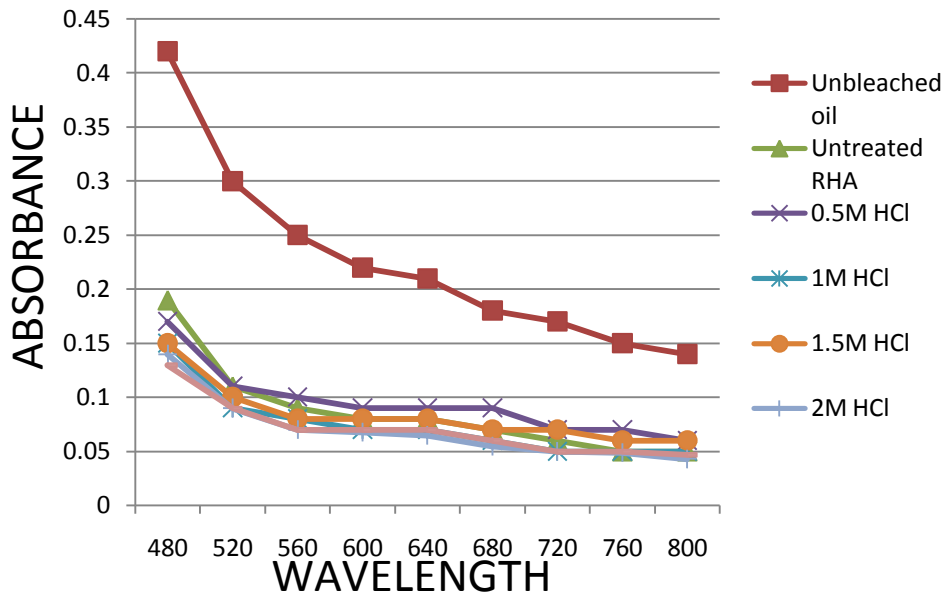


Figure 1: The Effect of Varying Concentration of HCl Acid Pre-treated RHA for Bleaching of Groundnut Oil.

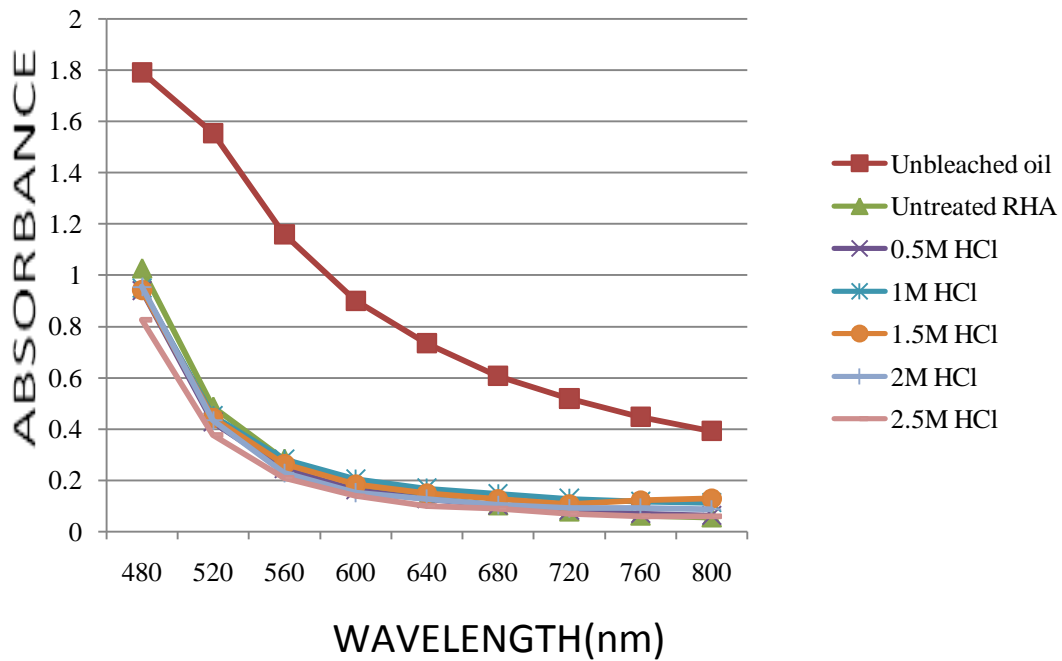


Figure 2: The Effect of Varying Concentration of HCl Acid Pre-treated RHA for Bleaching of Palm Kernel Oil.

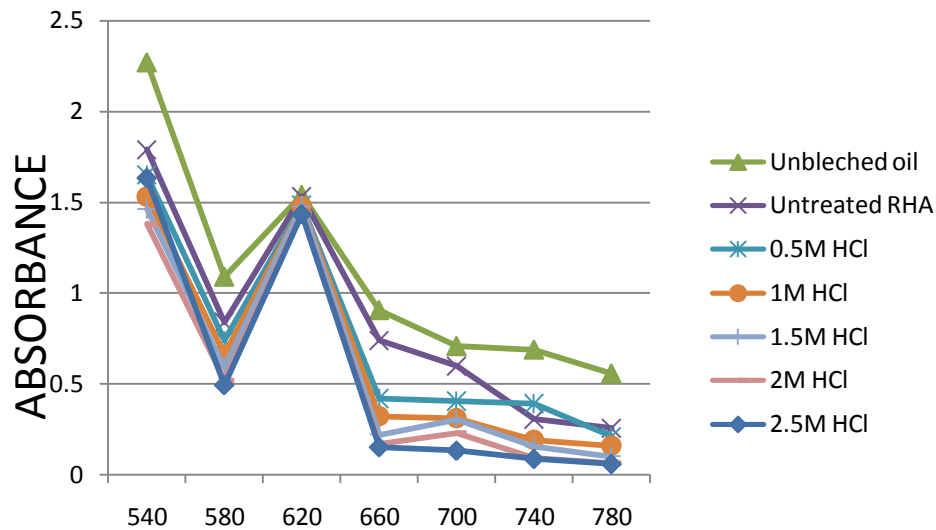


Figure 3: The Effect of Varying Concentration of HCl Acid Pre-treated RHA for Bleaching of Palm Oil.

#### IV. Discussion Of Results

The physical appearance of untreated rice husk was seen to be brownish before pre-treatment. After pre-treatment, it was seen to be light yellowish in colour due to the concentration of acid added to the husk. The light yellowish colour indicates that the pre-treatment of the rice husk has increased the surface area of the rice husk after treatment.

Table 1 represents the effect of carbonization temperatures at constant carbonization time of 1 hour on the analytics of carbonized rice husk. It can be observed from table 1 that as carbonization increases from 500-700°C, the ash content of the rice husk decreased from 6.78-5.72. The reduction in ash content with increase in temperature can be attributed to the effect of an increase in the temperature of the muffle furnace. The shrinkage of the ash present in the furnace increases the dielectric loss factor due to an increase in temperature of the rice husk burned inside the furnace. Also presented in table 1, is the effect of temperature on the porosity of the carbonized rice husk. The result shows that an increase in temperature resulted into increase in porosity of the carbonized rice husk.

Table 2 represents the effect of carbonization temperatures at constant carbonization time of 2 hours on the analytics of carbonizing rice husk ash. It can be observed from this table that as carbonization increases in temperature from 500-600 °C, the ash content of the rice husk increases from 5.451-5.740. The increase in ash content with an increase in temperature can be attributed to the effect of shrinkage of the burning husk due to reactivity and its pozzolanic properties. Also the carbon content present in the ash also decreases with an increase in the ash content. In other words some carbon present in the ash turn into ash in order to increase the silica content present in the ash. At 600-700 °C, the ash content of the rice husk decreases from 5.74-5.54. The reduction in the ash content with an increase in temperature can be attributed to the effect of shrinkage of the ash present in the muffle furnace and also the silica content is transferred from amorphous stage to crystalline stage. It also increases the dielectric loss factor due to an increase in temperature of the rice husk. Also presented in table 2, is the effect on the porosity of the carbonized rice husk. An increase in temperature leads to an increase in the porosity of the carbonized rice husk at a constant time interval.

Table 3 represents the effect of carbonization temperatures at constant carbonization time of 3 hours on the analytics of carbonizing rice husk ash. It can be observed from this table that as carbonization increases in temperature from 500-600 °C, the ash content of the rice husk increases from 5.420-5.999. The increase in ash content with an increase in temperature can be attributed to the effect of shrinkage of the burning husk due to reactivity and pozzolanic properties. Also the carbon content present in the ash also decreases with an increase in the ash content i.e. some of the carbon content was converted into ash content which enables the ash to increase with a decrease in the carbon content. At 600-700 °C, the ash content of the rice husk decreases from 5.999-5.131. The reduction in the ash content with an increase in temperature can be attributed to the effect of shrinking of the ash present in the furnace and also the transformation from amorphous to crystalline silica. Also, an increase in porosity of the carbonized rice husk resulted on the increase in temperature at constant time interval.

Table 4 represents the effect of carbonization time at constant temperature of 600 °C, on the analytics of carbonizing rice husk ash. It is observed that the optimum burning temperature of the carbonized rice husk was given to be 600 °C, because it yields the best ash content, carbon content and its porosity during the analysis at different time interval ranging from 1-3 hrs. The best optimum time was also observed to be 3 hrs based on the ash content, carbon content and porosity. At 1 hr, the carbon content was higher than that of 2 hrs and that of 2 hrs was higher than that of 3hrs. It simple means that at 3 hrs, the carbon content present in the ash at 1hr and 2 hrs were converted into ash which enables the ash content to increase from 5.88-5.99 g. The result also point out that high amorphous silica was produced (Nair *et al.*, 2006).

Table 5 represents the characterization of the developed Adsorbent using HCl as Activating Agent. It is observed that at constant temperature of 600 °C and constant time of 3hrs, 1.5M HCl of the pre-treated sample exhibit the highest ash content compared to the various pre-treated concentrations and yield a porosity of 0.937. The carbon content of the sample decreases with an increase in the ash content due to the temperature and burning time effect. 2.5M concentration also exhibit higher properties after the concentration of 1.5M. 0.5M exhibit the lowest properties compared to the other concentrations of the pre-treated samples.

Results obtained on the effect of varying concentration of HCl acid pre-treated RHA for bleaching of vegetable oil are presented graphically.

Figure1 represents the effect of varying concentration of HCl acid pre-treated RHA for bleaching of groundnut oil. It is observed that there is a large difference between the bleached oil and the unbleached oil. An increase in absorbance leads to a decrease in the wavelength of the oil. The increase in absorbance resulted into an increase in concentration. This is due to the pigment and chromophones present in the oil. The optimum bleaching concentration that yields the best bleaching potential among the bleaching agent from 0.5M-2.5M is observed to be 2M. 2M bleached the oil effective than the other concentration of the acid with a decrease in absorbance. Absorbance is the different between bleached oil and unbleached oil.

Figure 2 represents the effect of varying concentration of HCl acid pre-treated RHA for the bleaching of palm kernel oil. It is observed that the absorbance increases with a decrease in the wavelength. Also, the absorbance increase with increase in the concentration of an acid due to pigment and chromophones. The absorption is not proportional to the wavelength therefore; increase in wavelength does not increase the rate of absorption. The optimum bleaching concentration that yields the best bleaching potential among the bleaching agent is observed to be 2.5M. 2.5M of the sample bleached the oil faster than the other concentration of the acid at a constant temperature and time interval.

Figure 3 represents the varying concentration of HCl acid pre-treated RHA for the bleaching of palm oil. It is observed that the wavelength increases from 540-800 nm and the rate of absorbance decreases 2.3-1.4 for all acid concentration. The reduction in absorbance with increase in wavelength can be attributed to the concentration of the acid due to pigment and chromophones (due to the electrons in the chemical bonds only absorb certain wavelength of light). At 620 nm, the rate of absorbance is observed to be 1.5 that is to say the absorbance of 620 nm, is greater than that of 580 nm. It was observed from the graph that untreated sample exhibit the highest absorbance as the wavelength increases. In general, 2.5M HCl pre-treated sample exhibit the optimum bleaching potential at constant temperature and time interval compared to the other concentrations from 0.5-2M HCl samples.

The mean bulk density of the untreated RHA was determined to be 2088.9 kg/m<sup>3</sup> which confirm the literature review value ranging from 2000-2300 kg/m<sup>3</sup> (Velupillai *et al.*, 1997).

## V. Conclusion

The research aimed at the development and characterization of adsorbent from rice husk ash to bleach vegetable oils was successfully carried out. The following conclusion can be drawn out from the research work.

- The preparation of the rice husk ash by acid leaching of the rice husk (using 0.5M to 2.5M HCl with mixing ratio of 1 gram to 10 ml of rice husk) followed by calcinations at 600 °C, the best acid concentration for the bleaching of the oil was found to be 2M for groundnut oil and 2.5M for both palm oil and palm kernel oil.
- Acid treatment improved the bleaching power of the rice husk ash.
- The bulk density of the RHA was found to be 2088.9 kg/m<sup>3</sup>
- The best temperature for calcinations of the rice husk was given as 600 °C for 3 hours to produce the ash in an amorphous phase and not crystalline phase based on literature and ash content.
- It was also noted that an increase in the absorbance lead to a decrease in the wavelength in nm and the unbleached rice husk ash exhibit the best amount of absorbance followed by untreated rice husk ash.
- The results presented graphically indicate that the untreated and pre-treated samples (from 0.5M-2.5M HCl) falls close to each other and the gap between them is slightly small and also below the untreated oils.

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