

DEVELOPMENT AND CHARACTERIZATION OF ADSORBENT FROM COCONUT SHELLS FOR PURIFICATION OF DYE WASTEWATER

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Abstract

Preparation and characterization of adsorbent from coconut shell has been carried out successfully through chemical activation process using KOH, H₃PO₄ and KH₂PO₄ as activating agents. The effects of carbonization temperatures and activating agents on yield (dry weight basis), pore volume, iodine number were studied. The result showed that in all cases, increasing the carbonization temperature from 400 to 700 °C caused a decrease in the percentage yield of carbon and bulk density while the iodine number and pore volume is increased as temperature increased. Proximate analysis showed that the coconut shell is made up of 7.4 % moisture content, 10 % volatile matter, 2.5 % ash content while the fixed carbon is 80.01 %. The developed adsorbent have ash content ranges from 1.98-2.80 %, bulk density from 0.49-0.67 g/ml and pore volume ranging from 0.59-0.79 ml/g. The effectiveness of the developed adsorbent carbon was tested on dye effluent to determine its adsorption capacity. The performances of the adsorbents were observed to vary with the chemical activation agent used as follows KOH > H₃PO₄ > KH₂PO₄ at all the carbonization temperatures.

Keywords: Coconut shell, characterization, adsorbent, purification, wastewater, dye.

Introduction

Coconut shell is used for manufacturing of a variety of products of commercial importance including activated carbons. Activated carbon is the oldest known adsorbent (Gupta and Suhas, 2009; Carrott *et al.*, 2003; Lillo-Rodenas *et al.*, 2007; Phan *et al.*, 2006). Commercially available activated carbon are usually derived from natural materials such as wood, coconut shell, lignite or coal, but almost any carbonaceous materials may be used as precursor for the preparation of carbon adsorbent (Rozada *et al.*, 2003; Crini, 2006). The activated carbon is extensively used in the refining and bleaching of vegetable oils and chemical solutions, water purification, recovery of solvents and other vapours, recovery of gold, in gas masks for protection against toxic gases, in filters for providing adequate protection against war gases/nuclear fall outs, etc (CBD, 2006; Gupta and Suhas, 2009). Activated carbon is a non-graphite form of carbon which could be produced from any carbonaceous material such as coal, lignite, wood, paddy husk, coir pith, coconut shell, etc. (Nwabanne and Igbokwe, 2012; Roque-Marlberbe, 2007; Olafadehan *et al.*, 2012). Studies have shown that activated carbons are good materials for removal of different types of dyes in general, but their use is still sometimes limited due to high cost. Therefore the choice of coconut precursors over others is due to its availability locally in large quantity and its potentials as sorbent due to its physicochemical characteristics and low-cost. Coconut shell is cheap and abundant agricultural by-products in tropical countries like Nigeria and Ivory Coast. Adsorption technique is one of the preferred methods for the removal of dyes from wastewater because of its efficiency and low-cost. In this regard, several types of carbons or adsorbents have been prepared from agricultural by-products such as bagasse (Valix *et al.*, 2004; Juang *et al.*, 2001, 2002a; Tsai *et al.*, 2001; Ahmedna *et al.*, 2000), coir pith (Namasivayam and Kavitha, 2002; Namasivayam *et al.*, 2001a), banana pith (Kadirvelu *et al.*, 2003), date pits (Banat *et al.*,

2003), sago waste (Kadirvelu *et al.*, 2003), silk cotton hull (Kadirvelu *et al.*, 2003), corn cob (Juang *et al.*, 2002a), maize cob (Kadirvelu *et al.*, 2003), straw (Kannan and Sundaram, 2001), rice husk (Mohamed, 2004; Malik, 2003; Guo *et al.*, 2003; Kannan and Sundaram, 2001), rice hulls (Ahmedna *et al.*, 2000), fruit stones (Aygu'n *et al.*, 2003), nutshells (Aygu'n *et al.*, 2003; Ahmedna *et al.*, 2000), pinewood (Tseng *et al.*, 2003), sawdust (Malik, 2003), coconut tree sawdust (Kadirvelu *et al.*, 2000, 2003), bamboo (Wu *et al.*, 1999) and cassava peel (Rajeshwarisivaraj *et al.*, 2001a), have been used for the removal of dye from wastewater. Generally the public perception of water quality is greatly influenced by the colour. In wastewater the first contaminant to be noticed is usually colour (Banat *et al.*, 1996). Any amount of dyes present in water is highly visible and undesirable (Robinson *et al.*, 2001; Banat *et al.*, 1996). Today dye is still one of the most important class of pollutants, and once they enter the water it is no longer good and sometimes difficult to treat as dye have synthetic origin and complex molecular structure which makes them more stable and difficult to be biodegradable (Gupta and Suhas, 2009; Forgacs *et al.*, 2004; Rai *et al.*, 2005). Industrial wastewaters are often contaminated with various compounds like phenol, chromium, suspended solids, dissolved organic compounds, etc, and it is recommended that these wastewaters should be treated to an environmentally acceptable limit. The current problems in wastewater treatment stem primarily from the increasing pollution of wastewaters by organic compounds that are difficult to decompose biologically. These substances resist the self-purification capabilities of rivers as well as their inability to decompose in conventional wastewater treatment plants (Olafadehan and Aribike, 2000). The aim of this study is to prepare adsorbent from coconut shell at different temperatures using different activating agents; and to compare their performance for the removal of dye from wastewater.

Materials and Methods

The materials and equipment employed in this work include among others, phosphoric acid, sodium thiosulphate, potassium hydroxide, di-hydrogen tetraoxophosphate acid, dye effluent, dessicator, hotspot furnace, electric oven, heating mantle and turbidometer. All the chemicals used are of analytical grade. In this work, coconut shell was collected from Bosso Market in Minna, Niger state. The dye effluent on the other hand was collected from a local dye industry in F-Layout Mobile Minna, Nigeria, using a plastic can of ten litres capacity. The coconut shells were separated and cleaned from other materials such as coconut fiber and dirt by hand picking. The soil on coconut shell was removed by washing with clean water. The washed sample was spread under the sun to remove the liquid content from its solid phase in order to reduce the moisture content and make it ready for carbonization. The crushing process was carried out to first crush the coconut shell to smaller sizes. After carbonization the sample was crushed again to powdered form using mortar and pestle. All the experimental analysis in this research was conducted in triplicate and the values reported are the average limits within deviation of 0.002%.

Proximate Analysis of Raw Coconut shell

Determination of moisture content

A known weight (2g) of raw coconut shell sample was dried in an oven at 105°C until a constant weight was attained. The moisture content was determined as the difference between the initial and final weight (ASTM, 1987).

Determination of volatile matter

The standard crucible was preheated in the muffle furnace at 400 °C for 5 minutes. The crucible was cooled in the desiccators and 2 g coconut sample was placed in it. The crucible was now placed in the muffle furnace at 900 °C for 5 minutes. The loss in weight accounts for the volatile content of the coconut shell.

Determination of ash content

The standard crucible was preheated in the muffle furnace. It was then cooled in the desiccators. 2 g of coconut shell sample was placed in the crucible and heated at 750 °C for 1 hour. The incombustible residue gives the ash content of the coconut shell.

Determination of fixed carbon

This is gotten by deducting the sum of the percentages of ash, volatile, and moisture content from 100. It is expressed as

Fixed carbon %= 100-moisture %- ash %- volatile matter %

Carbonization and Chemical Activation

The coconut shell was divided into four samples of 300 g each and labelled M₁, M₂, M₃, and M₄. The carbonization process was monitored at the temperature of 400 °C, 500 °C, 600 °C and 700 °C respectively for 2 hours in a furnace for each sample. The carbonized samples were divided into four (4) parts according to the carbonization temperatures. Chemical activation involves the impregnation of each part of the carbonized sample with 1M phosphoric acid (H₃PO₄), 1 M potassium hydroxide (KOH) and 1 M di-hydrogen teraoxophosphate acid (KH₂PO₄) for 24 hours to open the pore, after which it was dried in oven at 110 °C for 3 hours (Okafor and Aneke, 2005). The next step was washing of the activated carbon with distilled water. The washed water was tested with litmus paper until it became neutral. The activated carbon was then drained and dried at room temperature (Okafor and Aneke, 2005). After drying at room temperature; the activated carbon was dried in an oven at a temperature of 110 °C for 3 hours. This process was followed by packing the activated carbon into plastic containers. Finally, characteristic properties of produced activated carbon such as ash content, pore volume, iodine number, and yield and bulk density were determined. The activated carbons were used to treat dye effluent using standard procedures as described by Dias *et al* (2007); Yusuf and Sonibare (2005); Madukasi *et al* (2001). The dye effluent was characterized before and after treatment with the developed adsorbent carbon samples.

Results and Discussion

The results of proximate analysis of moisture content, ash content, volatile matter and fixed carbon are presented on Table 1.0 and these values that were obtained are in agreement with standard precursors (Su *et al*, 2006; Jambulingam *et al*, 2007). Table 2.0 presents the effect of carbonization temperature on char yield while Table 3.0 shows the results of chemical activation of coconut shell. Table 4.0 shows the physical properties of activated coconut shell using different activating agents and at various temperatures while Table 5.0 is the characteristic properties of dye effluent before and after treatment with the developed coconut shell-based active carbons.

Table 1: Proximate analysis of raw coconut shell

| Parameter | % by weight dry basis |
|------------------|-----------------------|
| Moisture content | 7.4 |
| Volatile matter | 10 |
| Ash content | 2.5 |
| Fixed carbon | 80.1 |

As shown in Table 1, the coconut shell is made up of 7.4 % moisture content, 10 % volatile matter, 2.5 % ash content and 80.1 % fixed carbon on percentage by weight dry basis which agrees with literature values as reported by Su *et al*, (2006); Jigisha *et al*, (2004); McDoughall, (1991). Therefore coconut shell is very suitable for the production of active carbons in the treatment of dye wastewaters.

Table 2: Effect of carbonization temperature on yield

| Temp(°C) | Time(hr) | Final Weight (g) | Char Yield (%) |
|----------|----------|------------------|----------------|
| 400 | 2 | 95.62 | 31.87 |
| 500 | 2 | 89.27 | 29.70 |
| 600 | 2 | 78.58 | 26.19 |
| 700 | 2 | 71.42 | 23.80 |

Initial Weight =300g

From Table 2, the corresponding weight of each sample, before and after carbonization for two hours at 400 °C, 500 °C, 600 °C and 700 °C are shown. From the table, it can be seen that char yield decreased as the carbonization temperature is increased from 400 to 700 °C. The maximum solid yield was observed at temperature of 400 °C (31.87 %) while the least was observed at 700 °C (23.80 %). The effect of carbonization temperature on the char yield is due to differences in the amount of the volatile components released during carbonization. As the temperature of carbonization increases, more volatile components are exhausted from the coconut shell and it is in order of 700 °C > 600 °C > 500 °C > 400 °C. This result corresponds to previous studies of Su *et al*, (2006) and Jambulingam *et al*, (2007).

Table 3: Chemical activation of coconut shell

| Activating agent | Carbonization Temp (°C) | Initial Wt of Sample (g) | Final Wt of Sample (g) |
|---|-------------------------|--------------------------|------------------------|
| M ₁ ,KOH | 400 | 31.87 | 29.12 |
| M ₂ ,KOH | 500 | 29.70 | 26.89 |
| M ₃ ,KOH | 600 | 26.19 | 24.03 |
| M ₄ ,KOH | 700 | 23.80 | 21.10 |
| M ₁ ,H ₃ PO ₄ | 400 | 31.87 | 30.00 |
| M ₂ ,H ₃ PO ₄ | 500 | 29.70 | 27.90 |
| M ₃ ,H ₃ PO ₄ | 600 | 26.19 | 24.98 |
| M ₄ ,H ₃ PO ₄ | 700 | 23.80 | 22.96 |
| M ₁ ,KH ₂ PO ₄ | 400 | 31.87 | 30.93 |
| M ₂ ,KH ₂ PO ₄ | 500 | 29.70 | 28.11 |
| M ₃ ,KH ₂ PO ₄ | 600 | 26.19 | 25.15 |
| M ₄ ,KH ₂ PO ₄ | 700 | 23.80 | 23.00 |

M₁,_{KOH} = Sample 1 activated with KOH at 400°C carbonization temperature, M₂,_{KOH} = Sample 2 activated with KOH at 500°C carbonization temperature, M₁,_{H₃PO₄} = Sample 1 activated with H₃PO₄ at 400°C carbonization temperature, M₂,_{H₃PO₄} = Sample 2 activated with H₃PO₄ at 500°C

carbonization temperature, $M_1, \text{KH}_2\text{PO}_4$ = Sample 1 activated with KH_2PO_4 at 400°C carbonization temperature, $M_2, \text{KH}_2\text{PO}_4$ = Sample 2 activated with KH_2PO_4 at 400°C carbonization temperature.

Chemical Activation

After the carbonization process, the four (4) samples M_1, M_2, M_3 and M_4 were divided into three equal parts each, to make twelve (12) samples as shown in Table 3. These samples were then activated with 1 M H_3PO_4 , 1 M KOH and 1 M KH_2PO_4 solution. Table 3 shows the result of the activation, with activation time of 1 hour and activation temperatures for $400\text{-}500^\circ\text{C}$. These results show the loss in weight of each sample which is due to the dehydrating effect of the activation agent and rising with distilled water which removed the remaining ash and chemicals from the samples. From the result, it was noticed that yield is always less for samples activated with KOH followed by those activated with H_3PO_4 and KH_2PO_4 and increases with carbonization temperature in the order $400 > 500 > 600 > 700^\circ\text{C}$.

Table 4: Characterization of coconut-based activated carbon

| Activation Agent | Activation Temperature ($^\circ\text{C}$) | Ash content (%) | Bulk density (g/ml) | Pore volume (ml/g) | Iodine number (mg/g) |
|--|---|-----------------|---------------------|--------------------|----------------------|
| KOH | | | | | |
| M_1, KOH | 400 | 1.98 | 0.63 | 0.66 | 882.73 |
| M_2, KOH | 500 | 2.00 | 0.59 | 0.70 | 962.01 |
| M_3, KOH | 600 | 2.16 | 0.51 | 0.75 | 1120.92 |
| M_4, KOH | 700 | 2.25 | 0.49 | 0.79 | 1150.01 |
| H_3PO_4 | | | | | |
| $M_1, \text{H}_3\text{PO}_4$ | 400 | 2.04 | 0.64 | 0.62 | 859.62 |
| $M_2, \text{H}_3\text{PO}_4$ | 500 | 2.21 | 0.61 | 0.68 | 958.22 |
| $M_3, \text{H}_3\text{PO}_4$ | 600 | 2.27 | 0.55 | 0.71 | 1072.13 |
| $M_4, \text{H}_3\text{PO}_4$ | 700 | 2.38 | 0.53 | 0.76 | 1131.08 |
| KH_2PO_4 | | | | | |
| $M_1, \text{KH}_2\text{PO}_4$ | 400 | 2.23 | 0.67 | 0.59 | 848.11 |
| $M_2, \text{KH}_2\text{PO}_4$ | 500 | 2.31 | 0.65 | 0.64 | 950.98 |
| $M_3, \text{KH}_2\text{PO}_4$ | 600 | 2.37 | 0.58 | 0.69 | 1032.19 |
| $M_4, \text{KH}_2\text{PO}_4$ | 700 | 2.42 | 0.56 | 0.71 | 1129.11 |

The important physical and chemical properties tested for in the activated carbon were ash content, bulk density, and pore volume and iodine number as shown on Table 4. These values are seen to increase with increase in carbonization temperature and also vary slightly with the activation agent used.

Results as presented indicate that samples activated with KOH have least percentage of ash content compared with H_3PO_4 and KH_2PO_4 . Compared with the commercially available activated carbon, the coconut shell based activated carbon has significantly an acceptable amount of ash, an average of approximately 2.27 %. Only 2-4 % ash was indicated in the commercially available powdered activated carbon (CCI, 2006). The bulk density as shown in Table 4 is seen to decrease with increase in carbonization temperature and as well as with the activation agent used for all samples. This is because as the temperature increases, the amounts of carbon contained in the sample reduces as more ash is formed. The variation of bulk density with respect to activation agent is in the order $\text{KH}_2\text{PO}_4 > \text{H}_3\text{PO}_4 > \text{KOH}$ (Hutchins, 1988). Table 4 also shows the variation of pore volume with carbonization temperature and activation agent.

As carbonization temperature increases the pore volume is observed to increase. The pore volume of the coconut shell activated carbon based on activation agent is as follows $\text{KOH} > \text{H}_3\text{PO}_4 > \text{KH}_2\text{PO}_4$. This shows that pore volume is a function of activation agent and temperature as well as the precursor used. This was collaborated in the study by Gregg and Singh (1982), where it was shown that pore volume is a function of activating agent and temperature. The

iodine number, which is defined as the milligram (mg) of iodine adsorbed per gram (g) of activated carbon, was used as a measure of adsorption capacity of activated carbon produced. According to Kim (2004), iodine value expresses the actual adsorptive power of the adsorbent. Thus it is a more practical parameter to use than the specific surface area of the adsorbent to compare its adsorptive capacity. For the iodine number determination of the activated carbon, Table 4 shows that as the carbonization temperature is increased from 400 to 700 °C, the iodine number also increased from 882.73- 1150.01 mg/g for samples activated with KOH, 859.62- 1131.08 mg/g for samples activated with H₃PO₄ and 848.11- 1129.11 mg/g for samples activated with KH₂PO₄. In comparison with standard values, the iodine number should range between 900-1200 mg/g (ASTM, 2002). Therefore, it can be said of the developed adsorbent as having met the standard because all their values are within the prescribed range. Generally, carbonization and activation agent have a significant effect on the formation of adsorbent porous structures.

Treatment of Wastewater

The dye effluent was analyzed for pH, turbidity, colour; odour, density, total solid, and biochemical oxygen demand (BOD) before and after treatment with the active carbons. The results are presented in Table 5. After the initial measurement of these parameters, the wastewater was treated with the developed activated carbon for 24 hours and it was observed that the pH was from 12.59 to 8.9-10.11. These values fall within the admissible limit of 6-9.5 according to environmental protection act (EPA, 2004).

Table 5: Effluent analysis before and after treatment

| Activating Reagent | Temp (°C) | Treatment | pH | Turbidity (NTU) | Density (g/ml) | Total Solid (mg/dm ³) | BOD (mg/dm ³) |
|---------------------------------|-----------|-----------|-------|-----------------|----------------|-----------------------------------|---------------------------|
| | | Before | 12.59 | 90.00 | 1.16 | 160 | 370 |
| KOH | 400 | After | 9.67 | 83.10 | 1.10 | 59.02 | 68 |
| KOH | 500 | After | 9.50 | 81.08 | 1.08 | 54.03 | 62 |
| KOH | 600 | After | 9.08 | 78.11 | 1.07 | 48.05 | 57 |
| KOH | 700 | After | 8.90 | 76.03 | 1.07 | 40.03 | 54 |
| H ₃ PO ₄ | 400 | After | 9.86 | 84.06 | 1.11 | 64.04 | 70 |
| H ₃ PO ₄ | 500 | After | 9.71 | 82.86 | 1.09 | 61.04 | 65 |
| H ₃ PO ₄ | 600 | After | 9.32 | 80.04 | 1.09 | 55.09 | 61 |
| H ₃ PO ₄ | 700 | After | 9.05 | 77.91 | 1.08 | 47.05 | 57 |
| KH ₂ PO ₄ | 400 | After | 10.11 | 86.98 | 1.14 | 70.02 | 73 |
| KH ₂ PO ₄ | 500 | After | 9.91 | 85.06 | 1.12 | 68.08 | 68 |
| KH ₂ PO ₄ | 600 | After | 9.83 | 83.12 | 1.10 | 65.03 | 64 |
| KH ₂ PO ₄ | 700 | After | 9.71 | 81.22 | 1.09 | 60.01 | 60 |

Results obtained also show that the turbidity of wastewater reduced from 90 (NTU) to between 76 and 86 after treatment with developed adsorbent. The reduction in turbidity was highest in the dye effluent treated with KOH-based active carbons than in those treated with H₃PO₄ and KH₂PO₄ based ones at the same carbonization temperature. The treated effluent appearance was noticed to have become clearer, i.e. not as dark as it was before treatment. Also the offensive odour was also totally removed by the activated carbon for the twelve tests carried out. The same table also shows how the total solid in the wastewater was reduced from 160 to less than 75. This also agrees with those quoted by environmental protection act (FEPA, 2001). From the tables, the decrease in density from the initial value was observed; the value is \ reduced more in samples treated with KOH activated carbon than with those activated with H₃PO₄ and KH₂PO₄ at the same carbonization temperature. The biochemical oxygen demand (BOD) was also measured by using the activated carbon carbonized at 600 and 700 °C only. The results vary as shown in Table 5, and values were seen to decrease with increase in carbonization temperature and also decrease with activation agent according to this trend KH₂PO₄ > H₃PO₄ > KOH. According to federal environmental protection agency (FEPA, 2001) the acceptable limit of BOD in dye effluent is between 30 and 100 mg/dm³, all the values as stated in Table 5 fall within this limit.

Conclusions

This research work has revealed some latent facts about the activation agents, carbonization temperature, usefulness and effectiveness of adsorbent produced from coconut shell. From these studies, coconut shell based adsorbent was found to effectively adsorb pollutant indicators pH, total solids, odour and BOD. Chemical activation agents (KOH, H₃PO₄, and KH₂PO₄) were found to affect the adsorptive capacity of the activated coconut shell based upon variations in the characteristics of the carbons such as ash content, bulk density, pore volume and iodine

number. Coconut shell-based KOH adsorbent had higher adsorption capacities than coconut shell-based H_3PO_4 and KH_2PO_4 adsorbents. This may be as a result of high surface area of the KOH activated carbon compared to H_3PO_4 activated carbon and KH_2PO_4 activated carbon. Using coconut shell to develop adsorbent have potentially provide a less expensive and more environmentally friendly raw material than the commercial coal, as well as producing an active carbon processed from a renewable material instead of a non-renewable one.

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