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Preparation and characterization of activated carbon using coconut and palm kernel shells

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Abstract

Activated carbons were prepared from coconut and Palm kernel shells. The samples were carbonized and chemically activated using 0.1M H_3PO_4, 0.1M NaOH and 0.1M $ZnCl_2$ as the activating agents. The adsorption capacities of activated carbons prepared were determined using different standard solutions of the Bromophenol Blue and Congo red solutions. Values of solute adsorb ranging from 1.9mg - 0.2mg and 1.98-1.30g were obtained for Bromophenol blue and Congo red respectively. Activated carbon prepared from coconut shell compare favourably well with commercial activated carbon (CAC) than the one prepared from palm kernel shell. The ash content and the fixed carbon were 94 and 60% respectively. Coconut shell has a higher fixed carbon content. In terms of adsorptivity and percentage fixed carbon, H_3PO_4 activated coconut shell carbon (PCA) was found to be superior to other activated carbon where NaOH and $ZnCl_2$ had been used as activating agents and it is also superior to the one prepared from palm kernel shell. The nature of the raw material from which the carbon is prepared, the activating reagent used and the nature of the solute material being adsorbed are considered as the factors favouring the performance of PCA.

Keywords: Activated carbon, Adsorption, Bromophenol Blue and Congo Red

Introduction

Activated carbon is an amorphous form of carbon, microcrystalline, non-graphitic in nature, a product of carbonization and activation of carbonaceous material which has been specially treated so that it possesses a very high internal porosity due to large surface area (Bansal *et al*, 1988; Debussy, 1992). A vast number of materials can be used to produce activated carbon; almost any organic matter with a large percentage of carbon could theoretically be activated to enhance its sorptive characteristics. Two distinct types of activated carbon recognized commercially are: (i) Liquid-phase carbon and (ii) Gas-phase carbon (Doyin, 1988). The three major processes of producing activated carbon are: Carbonization, purification and activation (Bansal *et al*, 1988). According to Bansal, the effectiveness of activated carbon as an adsorbent is attributed to its unique properties, including large surface area, a high degree of surface reactivity, universal adsorption effect, and a favourable pore size (Ogbonaya, 1992). Activated carbons are used for the following: Sugar decolourization, Solvent and solution reclamation, refining of oil and fat, removal of impurities, water purification, metal ions removal, decolourizing, drying and degumming of petroleum fractions, removal of industrial odour, removal of small quantities of radioactive contaminant (Fadil *et al*, 1994; Kardiravela and Namsasivayan, 2003; Francisco *et al*, 2010). Owing to this universal usefulness and large applications, research on the use of activated carbon has attracted the interest of different scientists. Activated carbon prepared from *cocos* and *elaeis* family has been found suitable for the removal of organic and inorganic pollutants (Rahman *et al*, 2006; Olayinka *et al*, 2009; Francisco *et al*, 2010).

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This paper discusses the preparation of activated carbon from coconut shell and palmkernell shell as low cost absorbent using orthophosphoric acid (H_3PO_4), potassium hydroxide (KOH), and Zinc Chloride ($ZnCl_2$) as activating agents. Their adsorption capacities were carried out by the removal of Bromophenol blue and Congo red dyes.

Materials and Methods

Collection samples

Coconut and Palm Kernel shells were collected from Alagbede farm in Osun State. The samples were sun dried, and grounded into pieces in order to enhance carbonization. It was then purified, chemically activated using the methods reported by Adekola and Adegoke in 2005.

Preparation of Activated Carbon

100 g of coconut shells and palm kernel shells were carbonized in a specially constructed chamber, after cooling, the charred products were grounded with the use of mortal and pestle. The samples were screened to obtained samples of different sizes with the use of local sieve 0.112 – 0.125µm, 0.125 – 0.3 µm, and 0.3 – 0.5 µm. The charred products were purified by placing the charred sample in a 500 cm^3 beaker. About 250cm^3 of 0.5M HCl was added to the sample in the beaker. The mixture was stored and heated until evolution of gas occurred and stopped. The content was filtered and carbon residue was rinsed with distilled water until it was neutral to litmus paper. Chemical activation was done using different activating agents such as H_3PO_4, KOH, $ZnCl_2$. 25.0g sample of the purified carbon was put into a beaker containing 500cm^3 of 1.0M H_3PO_4. The content was thoroughly mixed and heated until a paste was formed according to the method described by Odebumi and Okeola, 2001. This was transferred into crucible and placed in a muffle furnace fixed at 500^oC for 2 hours. The activated sample was allowed to cool to room temperature after which it was washed with distilled water until the pH became neutral the sample was dried in an oven at about 80^oC – 100^oC. The same procedure was repeated using 1.0M KOH and saturated solution of $ZnCl_2$ as described by Adekola and Adegoke in 2005.

Analysis of the Activated Carbon Samples

The yield of carbonized product was done by weighing100g of each of the raw materials. These were carbonized separately. The carbonized product was cooled to room temperature and weighed again. The process was repeated twice (Adekola and Adegoke in 2005).

The ash content and percentage fixed carbon were carried out on each sample of the activated carbon. The ash content of an activated carbon is the residue which remains when the carbonaceous portion is burnt off and ash determination is important only for organic adsorbents like activated carbon. From the previously dried sample of the activated carbon, the carbon content was determined by igniting a carefully weighed 2.0g sample of activated carbon in a hot bunsen burner flame and cooled in a dessicator. The crucible containing the ignited carbon was placed in a muffle furnace at 900^0C for three hours. After heating, the crucible was allowed to cool to room temperature. The cycle of heating in the furnace, cooling and weighing was repeated three times for each sample. The percentage ash content was first determined and the percentage fixed carbon was deduced from it (Odebunmi and Okeola, 2001; Adekola and Adegoke in 2005). From the weight loss, the percentage ash content was calculated. The percentage fixed carbon was also calculated (Ogbonaya, 1992).

Samples of the prepared carbon were activated with orthophosphoric acid. 0.1g of each of this particle size were weighed and put in conical flask containing 100 cm^3 of bromophenol blue solution of 20 mg/L covered with rubber stoppers. The mixture was shaken vigorously and continuously on a mechanical shaker for 3 hour. The mixture was then filtered and the absorbance of the filtrate measured at the predetermined wavelength for bromophenol blue using UV – Vis spectrophotometer. Absorbance and Concentration were determined from the calibration graph. The same procedure was followed for the remaining samples of the activated carbon treated with solution of 1M H_3PO_4 (Odebunmi and Okeola, 2001).

0.1g of the prepared activated carbon sample with particle size 0.112-0.125 µm was weighed into 250 cm^3 conical flask. 100cm^3 of solution containing 20 mg/l and 25 mg/l concentration of bromophenol blue and congo red were chosen for the determination respectively. The mixture was corked using rubber stopper, it was then shaken vigorously and continuously on a mechanical shaker for about three hour. The resulting incomplete decolourised filtrate was measured at predetermined wavelength of 10 nm and 5 nm intervals using UV – Vis spectrophotometer. The residual concentration of the filtrate was then determined. The extent of adsorption and the amount of substance adsorbed were obtained and this was repeated for other samples of activated carbon prepared (Odebunmi and Okeola, 2001).

Results and discussion

Percentage yield of carbonized product

Different samples of activated carbon prepared from coconut and palm kernel shells. The percentage yields of carbonized products are given in Table1. Six different activated carbons were prepared from coconut and palm kernel shells using H_3PO_4, $ZnCl_2$ and KOH as activating agents and they were identified as follows; H_3PO_4 activated coconut shell carbon (PCA), $ZnCl_2$ activated coconut shell carbon (ZCA), KOH activated coconut shell carbon (KCA), H_3PO_4 activated palm kernel shell carbon (PKA), $ZnCl_2$ activated palm kernel shell carbon (ZKA), KOH activated palm kernel shell carbon (KKA). Commercial activated charcoal (CAC) was used as control.

Table 1: Percentage yield of carbonized products.

Raw materials	Weight before Carbonized (g)	Weight after Carbonization (g)	Percentage yield (%)	Average (%)
Coconut shell	100 100	30.80 28.90	30.80 28.90	29.50
Palm kernel shell	100 100	28.90 26.80	28.90 26.80	27.50

The result of the yield of carbonized product is stated in Table 1 above. The yield for each sample was estimated. Coconut shell was found to have a higher yield and the values obtained are in the range of those reported by other researcher (Silverman, 1981; Adediran and Nwosu, 1996; Odebunmi and Okeola, 2001).

Ash content and percentage fixed carbon

The result of ash content and fixed carbon analyses are presented in Table 2.

Table 2: Percentage ash and fixed carbon contents of different activated carbon prepared

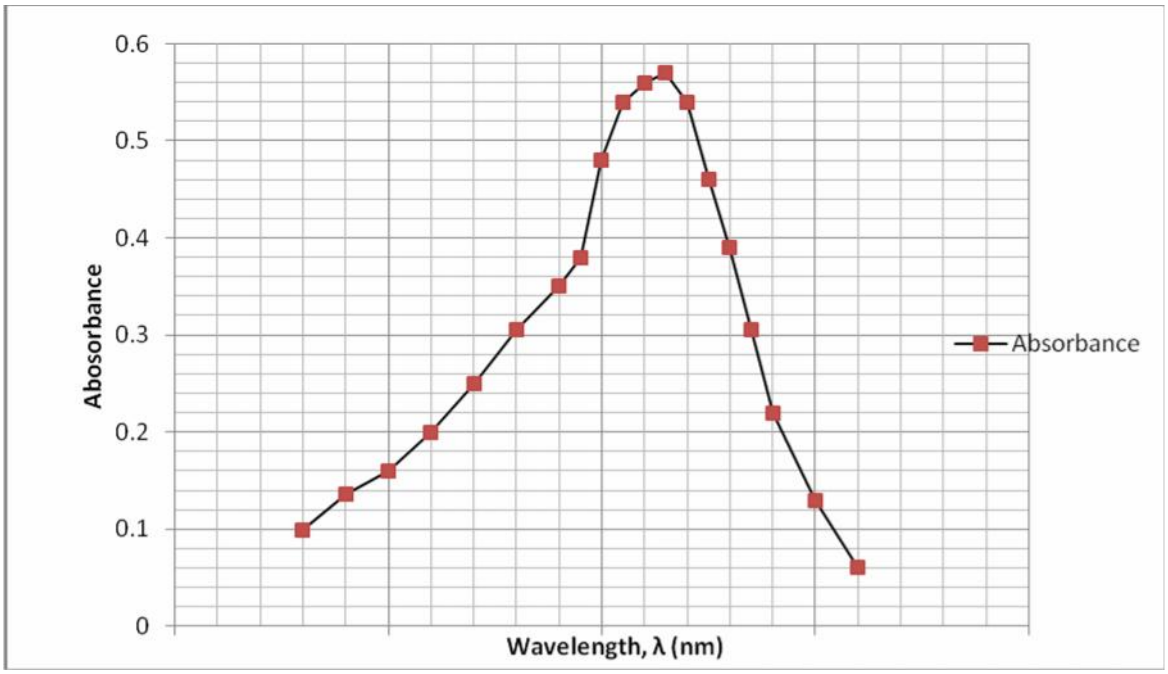
Sample	Percentage ash (%)	Percentage fixed carbon %
PCA	6.0	94.00
KCA	11.6	88.40
ZCA	16.8	83.20
PKA	40.0	60
KKA	8.8	91.2
ZKA	30.8	69.2

From the data, samples from palm kernel shell have the lowest percentage fixed carbon except KKA, (activated carbon produced from palm kernel shell using KOH solution as the activating agent). It was observed that samples with the lowest percentage fixed carbon have the lowest adsorption capacity and this finding agreed with that of Okeola (1999). Samples of activated carbon produced from coconut shell have the highest percentage fixed carbon and high adsorption capacity in the adsorption test.

Standard calibration graph for the coloured solutions

The spectra of bromophenol blue and congo red solutions

The plot of absorbance against the wavelength is made for bromophenol blue and congo red and are shown in figures 1 and 2 respectively. The wavelength corresponding to the maximum absorbance for different coloured solutions was found to be 615nm and 525nm for Bromophenol blue and Congo red respectively.



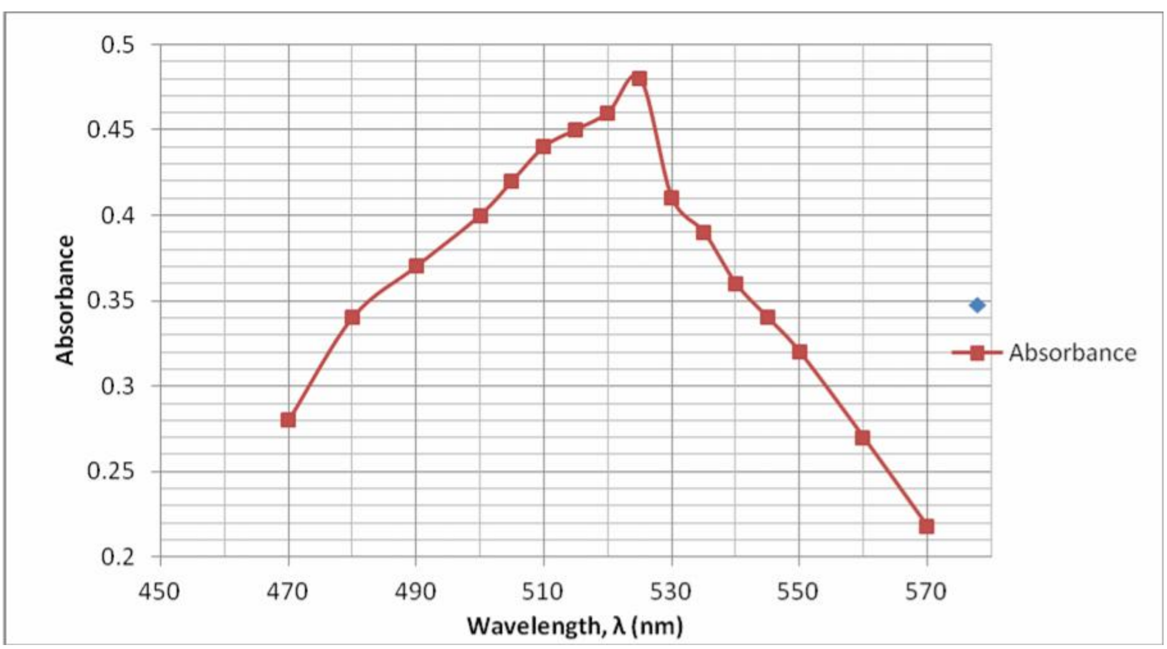


Fig 2: Spectrum of Congo Red solution

Standard calibration curve

Standard calibration curve were plotted for both Bromophenol blue and Congo red respectively, the figure is as shown below. From the graph, the molar absorptivity ($\mathcal{E}_o$) was found to be 5.6x10^5 l $cm^{-1}g^{-1}$ and 3.68x10^4 l$cm^{-1}g^{-1}$ for Bromophenol blue and Congo red solutions respectively.

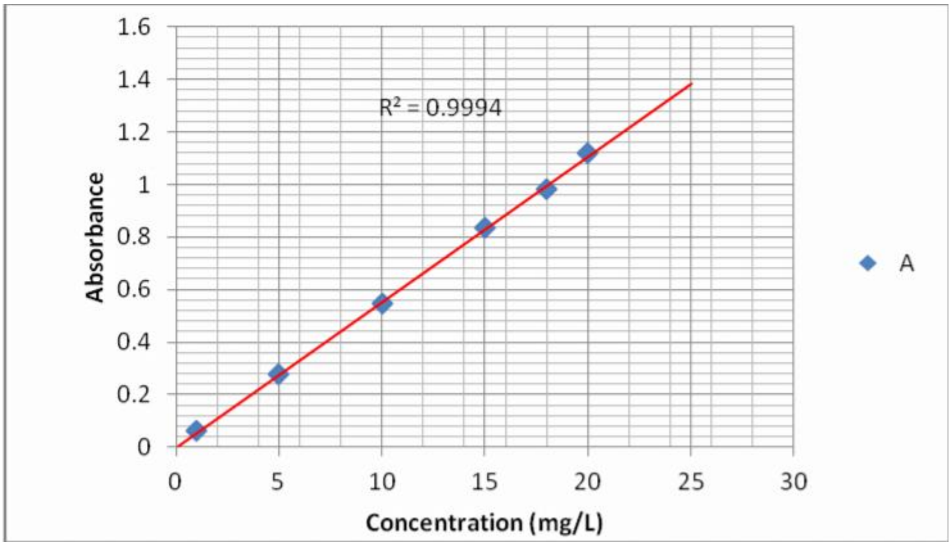


Fig 3: Standard calibration graph for Bromophenol Blue solution

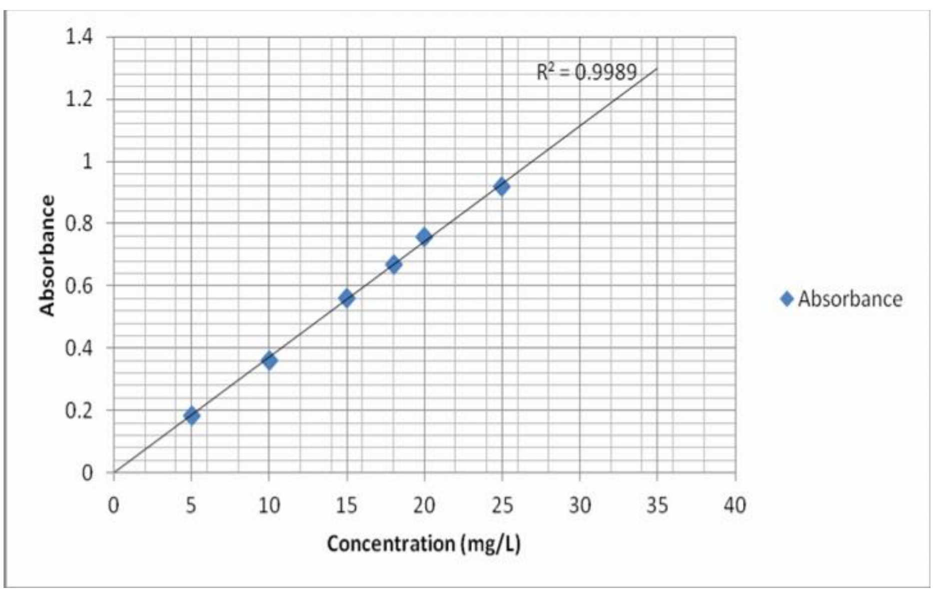


Fig 4: Standard calibration graph for Congo red solution

Effect of particle size on the adsorption capacity of activated carbon

The particle sizes used for this adsorption are (0.112-0.125)µm, (0.125-0.30) µm and (0.3-0.5) µm respectively. From Table 3, it was observed that the bigger the particle size, the higher the adsorbance and the lower the quantity of solute adsorbed. This observation correlates with that of Duff and Ross (1968) and Okeola (1999) in a related work on the effect of adsorbent particle size on the adsorption capacity of activated carbon. Result obtained revealed that adsorption capacity of the activated carbon decreases with increase in the particle size as follows (0.112-0.125) > (0.125-0.3) > (0.3-0.5).

Table 3: The effect of particle sizes on the ability of activated carbon to adsorb Bromophenol blue solution

Particle Size (µm)	Adsorbance of decolourized solution ± 0.001	Concentration of decolourized solution ± 0.01mg/l	Quantity of solute adsorbed ± 0.1mg
PCA			
0.5-0.3	0.380	6.09	1.40
0.3-0.125	0.130	2.03	1.79
0.125-0.112	0.088	1.05	1.90
PKA			
0.5-0.3	1.050	18.09	0.19
0.3-0.125	1.000	18.0	0.20
0.125-0.112	0.758	13.08	0.69

Adsorption of bromophenol blue and congo red

The result of adsorption characteristics of activated carbon are shown in the Table 4 and 5 below. Due to the adsorption ability of each activated carbon samples, decolourisation was carried out on all the coloured solution prepared at different concentrations. The concentrations used for the analysis are 20 mg/l and 25 mg/l for Bromophenol Blue and Congo red solutions respectively. Samples of activated carbon with particle sizes (0.125-0.112)µm were chosen for this adsorption characterization due to higher fixed carbon. The results are presented in Fig 6 and 7 respectively.

The results of adsorption characteristics of activated in Bromophenol blue solution are reported in Table 4 and it is presented in figure 6. The quantity of solute adsorbed was obtained using

$$Q = \frac{V(cm^3)(C_i - C_f)mg/cm^3}{1000}$$ …………………………..(Vanderborght and Van, 1977)

C_i is the initial concentration before the adsorption and C_f is the final concentration after adsorption.

V = 100 cm^3 (Volume of the solution prepared from the stock solution).

Table 4: Adsorption of bromophenol blue solution using 20 mg/l concentration

Particle Size (µm)	Adsorbance of decolourized solution ± 0.001	Concentration of decolourized solution ± 0.01mg/l	Quantity of solute adsorbed ± 0.1mg
PCA	0.089	1.0375	1.90
KCA	0.915	16.0375	0.40
ZCA	1.080	17.0875	0.30
PKA	0.766	13.075	0.70
KKA	0.494	8.0875	1.20
ZKA	0.630	11.025	0.90
CAC	0.085	1.0333	1.90

From the table above, PCA has the highest quantity of solute adsorbed which compete favourably well with commercial activated carbon, CAC. Fig 5 shows the representation on the bar chart. In this research, it was observed that samples produced with orthophosphoric acid reagent showed a distinct trend and has the highest peak especially in coconut shell. From fig 5 coconut shell activated carbon with orthophosphoric acid as the reagent gave the highest decolourising capacity in Bromophenol blue which this shows that orthophosphoric is the best activating agent suitable for Bromophenol blue decolourisation. The increasing order of the adsorptivity of bromophenol blue is: PCA > KCA > ZCA > PKA > KKA > ZKA (Adediran and Nwosu, 1996; Odebunmi and Okeola, 2001).

Adsorption of congo red solution on activated carbon samples

The concentration of Congo red used for this test analysis is 25mg/l and the calculation for the quantity of solute adsorbed is thus:

Also in Congo red: for PCA
Initial concentration, C_i= is 25 mg/l
Final concentration, C_f = is 5.125 mg/l
Volume = 100cm^3

$$Q = \frac{100(25-5.125)\text{mg}/\text{cm}^3}{1000} = 1.98\text{mg}$$

Q = 1.98 mg

Table 5: Adsorption of congo red solution on activated carbon samples

Sample analyzed	Adsorbance of Decolourized Solution (±0.001)	Concentration of Decolourized Solution ±0.01mg/l	Quantity of Solute Adsorbed ± 0.1mg
PCA	0.194	5.125	1.98
ZCA	0.266	7.063	1.79
KCA	0.230	6.0125	1.89
PKA	0.314	8.5	1.65
ZKA	0.208	5.33	1.96
KKA	0.319	8.25	1.67
CAC	0.218	5.375	1.96

From the table above, the adsorption capacity of PCA was found to be higher than that of CAC. In this research, it was observed that samples produced using orthophosphoric acid as the activating agent showed a distinct trend and has the highest peak especially in coconut shell. Figures 6 and 7 below shows the comparison in adsorption of Congo red and Bromophenol blue onto coconut shell activated carbon (CSAC), palm kernel shell activated carbon (PSAC) and the commercial activated carbon (CAC). Coconut shell carbon activated with orthophosphoric acid (P) was observed to be the best. Orthophosphoric acid activated carbons were observed to have the highest decolourising capacity in Congo red solution and this shows that orthophosphoric is the best activating agent suitable for Congo red decolourisation. The increasing order of the adsorptivity of Congo red is: PCA > KCA > ZCA > PKA > KKA > ZKA.

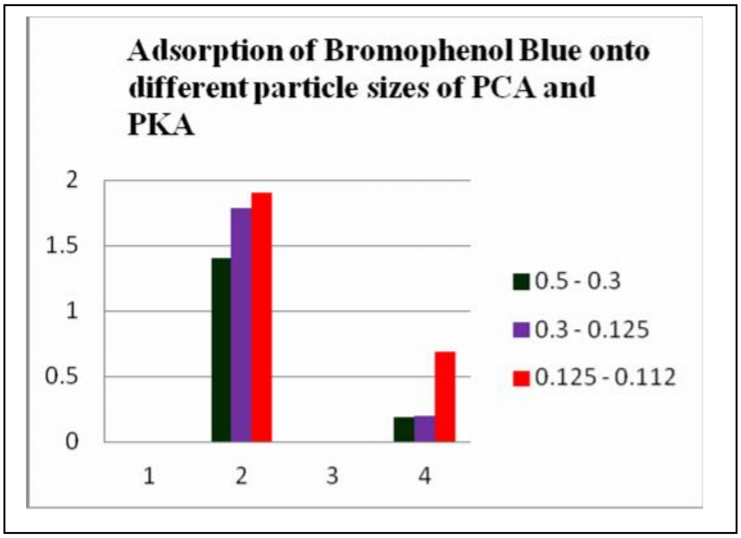


Figure 5:

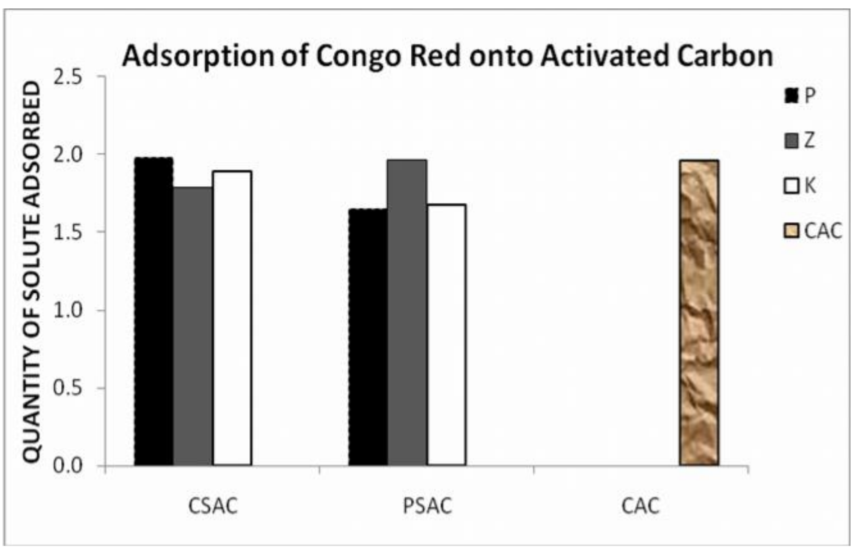


Figure 6:

KEY: P,Z and K represents H_3PO_4, $ZnCl_2$ and KOH respectively.

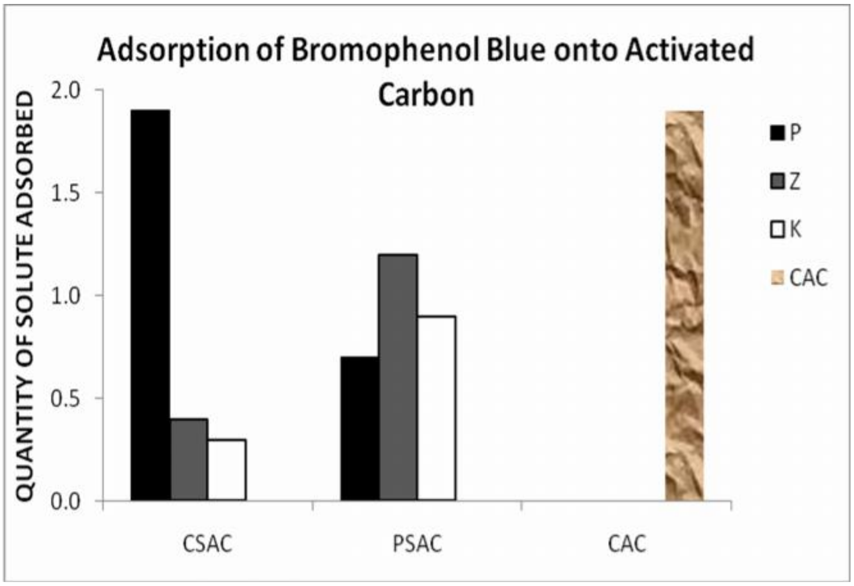


Figure 7:

KEY: P,Z and K represents H_3PO_4, $ZnCl_2$ and KOH respectively.

Conclusion

The activated carbon derived from coconut shell activated with orthophosphoric acid (PCA) showed the greatest adsorption capacity and its adsorption capacity is comparable with that of commercial activated charcoal. However, in bromophenol blue solution, ZCA showed the lowest adsorption capacity while in Congo red solution KKA showed the lowest adsorption capacity. The results of this study show that the magnitude of adsorption capacity of activated carbon can be influenced by various factors such as raw material from which the carbon is prepared, activating reagents used and nature of the solute material being adsorbed (i.e. the adsorbate like bromophenol blue solution).The percentage ash and the percentage fixed carbon were estimated and activated carbon produced form coconut shell, PCA was observed to have the highest percentage fixed carbon. In this research, it can be concluded that PCA was the best of all the samples, this report agreed with report of (Odebunmi and Okeola, 2001).

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