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Studies of Effect of Temperature, pH and Particle Sizes on the Removal of Carotene from Palm Oil by Activated Carbon

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ABSTRACT: The effect of some process variables on the removal of carotene from palm oil by activated carbon produced from Enugu coal was investigated. Activated carbon from coconut shell was employed to provide the necessary frame work for the adsorptive assessment. The activated carbon precursors were carbonized prior to activation with the activating agents. The batch method of adsorption was conducted and the extent of adsorption was monitored spectrophotometrically. The bleaching capacity of the adsorbent increased with the adsorbent dosage up to an optimum value. The removal of carotene increased with decrease in pH value and recorded the maximum adsorption at pH 4. The particle size analysis showed that 150μm had the highest adsorption capacity. In the same manner, the uptake of carotene from palm oil increased with the temperature rise. The analysis of these parameters will provide useful guides for the effective design of a vegetable oil plant.

I. INTRODUCTION

Carotene is a class of polytenes, the long chains of conjugated double bonds, the presence of which explains the intense colour of carotenoids ranging from yellow to red and purple. Carotene is present in numerous (or different) vegetable oils including yellow maize (corn) oil, groundnut oil, soy-bean oil, olive oil, palm oil etc.

Palm oil is deep red in colour due to the presence of Carotene. It contains the highest known concentration of agriculturally derived carotenoids, ranging from 500 – 700 ppm of its total composition and 15 to 300 times as many as retinal equivalents as Carrot, leafy vegetables and tomatoes [1]

However, for palm oil to enjoy universal industrial applications, the composition of carotenoid substances should be moderated. The removal of this colouring matter called carotene from palm oil by activated carbon is an adsorption process. Bleaching of vegetable oil reduces chlorophylls, carotenoids and peroxides by adsorbing the pigments onto the adsorbent. After bleaching the oil becomes brighter, less coloured and more stable [2-3].

In this research work, activated carbon produced from Enugu coal was used as an adsorbent for the adsorption of carotene from palm oil. Activated carbon is an amorphous form of carbon that has been specially treated to acquire improved adsorptive properties such as surface area, pore volume, pore size and pore size distribution etc. Activated carbon has long been a substance of great importance in the removal of small concentration of solute from liquids. Its applications range from the treatment of domestic and industrial waters to the production of refined sugar, decolourization of petroleum products, purification of vegetable oils as well as in pharmaceutical industries for drug production. Recent research interest has been shifted from advanced water treatment to its use in direct contact with blood as an artificial kidney, remediation of contaminated soil and as catalyst and catalytic support in process industries for catalytic reaction optimization [4].

Several factors determine the adsorption of solute pigment by adsorbent. These include sorbent structural properties (Pore size, porosity, surface area), particle sizes, density of adsorbent, ionic charge, solubility, stability of complexes, ionization, interaction of multiple solutes, initial dosage (concentration) of the adsorbate, pH and temperature etc [5]. The removal of solute from aqueous solution is also highly dependent on the degree of ionization and speciation of the adsorbate [6]. In a carbon-liquid interface, the liquid size of the interface exerts a positive influence. The intensity, magnitude and direction of this influence will vary according to the specific types of solutes and solvents. The net resultant can serve to augment, modify or nullify the attractive forces exerted by the carbon side on the interface [7].

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The effects of multiple solutes can be quite specific depending on the relations existing among the various solutes. Some solutes diminish the adsorption of others while others may cause a mutual increase. Still others show no measureable change. Compounds that show great adsorbability from pure solutions are often preferentially adsorbed [8]. Again, the quantity adsorbed does not always reflect the strength of the attachment to the surface. Consequently, the amount adsorbed from the pure state should not be used to predict the proportion in which solute components will be adsorbed from a mixture. But when several substances are adsorbed from a mixture, the combined adsorption may equal or exceed or be less than that of any individual ingredient from a pure solution. When the total amount adsorbed from a mixture is approximately equal to the sum of the separate ingredients as measured from the pure solutions, this indicates that each solute is attracted to separate adsorption sites [8]. The research interest in this work borders on the investigative study of the effect of particle sizes of adsorbent, pH and temperature on the removal of Carotene from palm oil by activated carbon (CAC) produced from Enugu coal. Also, an extension of the study was made on coconut based activated carbon (NAC) to serve as a comparative analysis towards ascertaining the efficacy of the activated carbon from Enugu coal.

II. MATERIALS AND METHODS

A. Enugu Coal and Coconut Shell

Enugu Coal was obtained from Onyeama Coal mine at Enugu while the Coconut Shell and palm oil were sourced at Eke market in Ede-Oballa town, Nsukka L.G. of Enugu State.

B. PRE-ACTIVATION TREATMENT

Enugu Coal: 50g of the sample was ground with a shock crusher and a grinder. The ground coal was dried in an oven at 90^0C for 24 hours and later sieved to mesh size of 500µm.

Coconut Shell: 30g of the sample was measured out and heated to 300^0C in a muffle furnace for 1 hour. The product was dried in an oven for 12 hours.

C. CARBONIZED OF COAL

20g of the pretreated coal sample was further crushed to a size of 200 – 400µm and placed in a retort vessel and put in a heating furnace. It was heated in absence of air at a temperature of 600^0C for 1½ hours. The sample was allowed to remain in the furnace for 4 days where it was cooled to room temperature [5-9].

D. ACTIVATION OF COAL AND COCONUT SHELL SAMPLES

The carbonized coal sample was re-crushed to 150 – 200µm particle sizes and placed in a galvanized vessel. 1.2M H_3PO_4 was added to the coal sample and to the mixture was added 10ml of water in similar ratio. The mixture was then heated to 600^0C under high pressure in a furnace for a period of 5 hours. The sample was allowed to cool to room temperature and washed with distilled clean water to remove excess acid. The pH was adjusted between 6 and 8 using 0.5M NaOH. The product sample was dried in an oven at a temperature of 105^0C for 1½ hours and stored in an air-tight container for usage. Similarly, the same method of activation was carried out using activating agents of $ZnCl_2$ and H_2SO_4. Also, the same procedure was repeated using carbonized coconut shell sample.
Finally, the adsorptive properties of both samples of raw and activated carbon were evaluated in accordance with the procedures described by [7-9]

III. BLEACHING

In this adsorption study, the activated carbon from activating agent of H_3PO_4 was used based on its assessment of adsorptive characteristics [5]. The bleaching was carried out by weighing out 40ml of palm oil into a conical flask placed in a thermostatic bath. The initial colour absorbance of palm oil was measured with Unicam 500 series UV spectrophotometer at a maximum wavelength of 400nm.
1.0g of adsorbent (repressing 2.5 weight percent of adsorbent) was measured out and mixed with 40ml of palm oil. The resulting mixture was heated and thoroughly stirred until a temperature of 80^0C was attained. The temperature was

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maintained for 40 minutes accompanied by constant stirring. The spent adsorbent was subsequently filtered out using a filter paper and the filtrate, the bleached oil collected in a beaker. The final colour of the bleached oil was measured with spectrophotometer and the degree of bleaching of the oil evaluated.

The experimental procedure was repeated by using 2.0g, 4.0g, 6.5g, 9.0g, 12.0g, 15g and 20g of adsorbent (representing weight percent of 5, 10, 15, 20, 25, 30 and 35 of adsorbent). For each adsorbent dosage, the test was conducted at temperatures of 90^0, 100^0, 110^0 and 120^0C at constant particle size of 150µm and time interval of 40 minutes.
Also, the effect of the particle size of adsorbent was studied by conducting the same bleaching procedure with various particle sizes of 200, 180, 150 and 75µm at constant temperature of 110^0C and the same time interval of 40 minutes. Similarly, the bleaching procedure was carried out at various pH values of 4, 6, and 8 at same operating parameter of temperature of 110^0C and time of 40 minutes.

IV. RESULTS AND DISCUSSION

Table 1: Properties of Coal and Coal-based Activated Carbon

Parameter	**Raw Coal**	**Activated Carbon from H_3PO_4**	**Activated Carbon from H_2SO_4**	**Activated Carbon from $ZnCl_2$**
Moisture content %	9.88	3.53	4.40	5.16
Ash (% d.b)	16.44	8.37	11.92	10.88
Vol. Matter (% d.a.b)	34.41	20.24	20.24	22..10
Fixed Carbon (% d.b)	41.27	67.86	63.44	61.84
Surface area, m^2/g	506	745	683	729
Porosity	0.441	0.536	0.503	0.511
Density g/cm^3	0.944	0.905	0.851	0.911

Table 2: Properties of Raw Coconut Shell and Coconut-based Activated Carbon

Parameter	**Raw Coconut Shell**	**Activated Carbon from H_3PO_4**	**Activated Carbon from H_2SO_4**	**Activated Carbon from $ZnCl_2$**
Surface area, m^2/g	611	967	910	948
Porosity	0.493	0.567	0.614	0.553
Density g/cm^3	0.919	0.861	0.889	0.869

The results of the analysis of Enugu Coal and Coconut Shell (raw and activated) are given in Tables 1 and 2. The results show that the physical properties of the adsorbents changed significantly after the activation process. Moisture content, ash content and volatile matter were reduced whereas specific surface area, fixed carbon, and porosity increased. For instance, the surface area and porosity increased from 506 to $745m^2/g$ and 0.441 to 0.536 respectively for activated carbon produced with phosphoric acid. The same trend was obtained from the other activating agents investigated. The results from coconut shell sample recorded relatively improved values of these properties aforementioned, thus lending credence to its adsorbent rating. Similar results were reported by earlier workers such as [9-11]
The figures i – vi, depict the results of absorbance of unbleached and bleached oils at various adsorbent dosage. The results show that there is a relatively rapid adsorption of carotene from palm oil by the adsorbent earlier in the run follow by a decreasing rate of adsorption and finally a slow approach to equilibrium for the various parameters of particle size, pH, adsorbent dosage and temperature investigated. This trend concurred with the natural phenomenon observed in adsorption [12].
The effect of adsorbent dosage was studied by keeping all other operating conditions constant while varying the mass of adsorbent. The results show that the degree of removal of carotene from palm oil increases with the quantity of adsorbent. But the amount of adsorbate carotene adsorbed per unit mass of adsorbent decreases considerably. The

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decrease in unit adsorption with the increasing quantity of adsorbent is basically due to adsorption sites remaining available and unsaturated as adsorption progresses. The sharp steepness or reduction in percentage colour reduction between zero and 20 percent weight of adsorbent dosage indicated the occurrence of maximum capacity of adsorption. But at a range of 20 to 25% of adsorbent dosage, the reduction in colour pigment progressively decreased to a minimum. This means that the adsorbent dosage above 25% will produce insignificant change in carotene removal and that any further increase in adsorbent dosage will result in un-economical process as equilibrium is approached. The analysis of the experimental data viz-a-vis the required standard colour values accepted in vegetable industries shows that the adsorbent dosage of 15 to 20% is very satisfactory as optimum adsorption capacity [6]. Remarkably, in vegetable oil plants, the bleaching of oils requires considerable less adsorbent than the bleaching of the same oil under similar conditions. This is mainly due to effective design of the equipment and process conditions of plant which create the necessary conditions for the optimum utilization of all contending variables [7].

In figures i and ii, the effect of pH was studied by keeping all other process variables constant. At varying pH values of the adsorbent, the coal-based activated carbon exhibited consistently high adsorption measurements at the range of pH 4 – 8, recording the highest percentage colour reduction of carotene pigment at the pH of 4. But the value of carotene removed decreases with the increase in pH level. As observed, the least values were recorded at pH of 8. The enhanced removal of carotene at low pH levels may be attributed to the larger number of H^+ ions present which in turn neutralize the negatively charged surface, thereby reducing hindrance to the diffusion of carotenoid substances. At higher pH values, the reduction in adsorption capacity of the adsorbent may be due to the abundance of OH^- ions which constitute one of the barriers to the diffusion process. Some corroborative results have been reported by many workers including Gueu et al (2006) and Teker et al (1999). According to these researchers, the pH of the adsorbate (carotene) solution is one of the factors that influence adsorption as it affects the surface charge of the adsorbent, level of dissociation of functional groups on the adsorbent surface, degree of ionization and speciation of the adsorbate, solubility of metal ions and concentration of the counter ions in solution.

The theory of the smaller the particle size, the larger the surface area and subsequently the higher the removal of carotene from palm oil by the adsorbents was observed in this work. This effect is depicted in figures iii – iv. In general, intra-particle mass transfer effect increased with decreasing particle size. From the results, the highest adsorption capacity of the adsorbent was recorded at the range of particle size of 150µm. However, the value decreased at 75µm particle size, a result that may be attributed to the effect of agglomeration of the particles which is mostly prevalent at very minute particle sizes [2 -15].

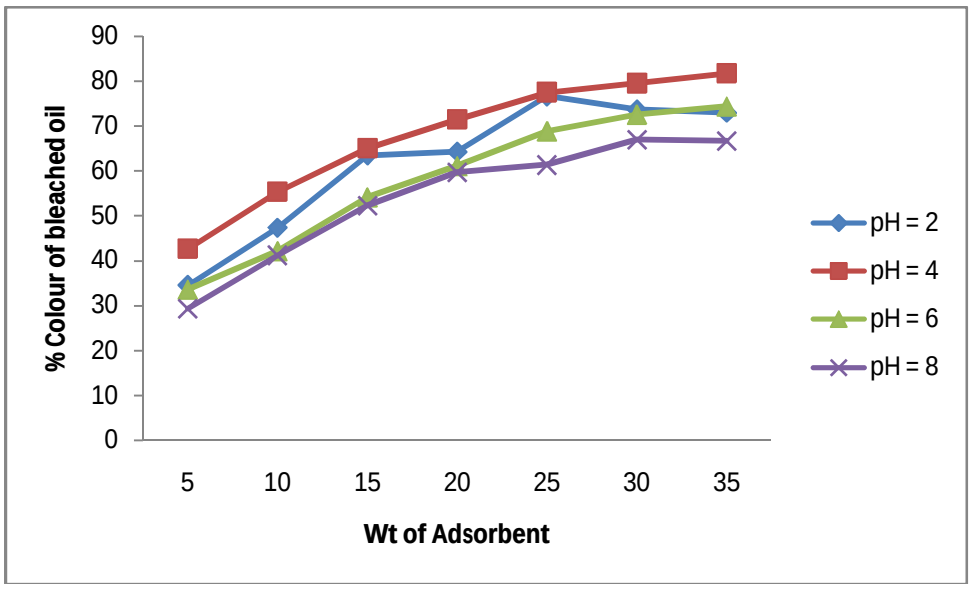

Fig. i: CAC and PO at varying pH values (at constant temperature and particle size)

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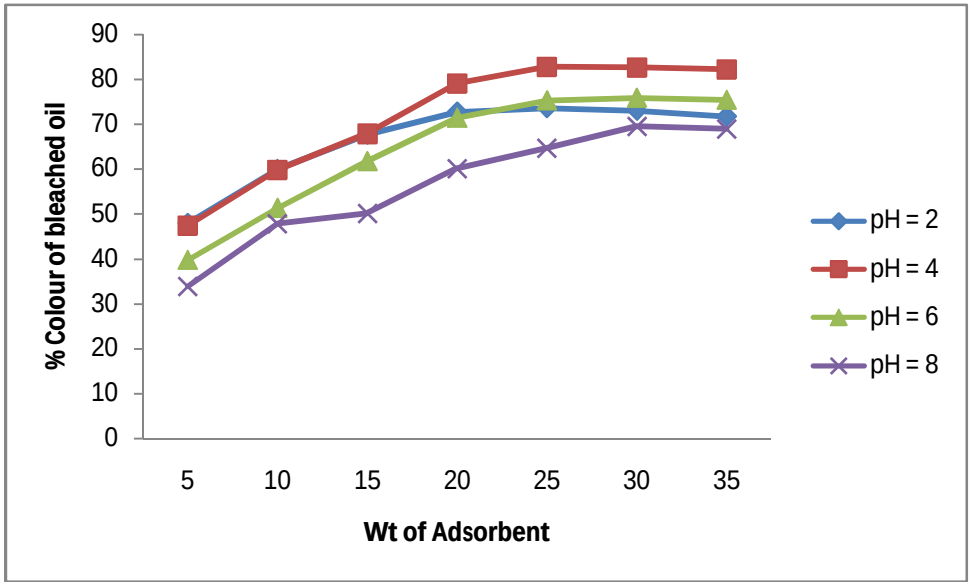

Fig ii: NAC and PO at varying pH values (constant temperature and particle size)

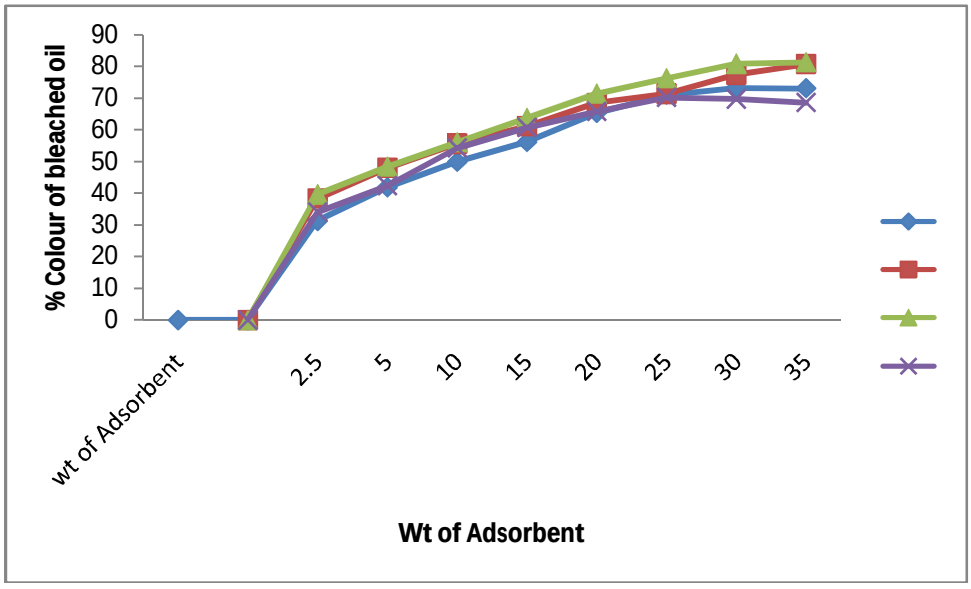

Fig. iii: CAC and PO at varying particle sizes (constant temperature and time)

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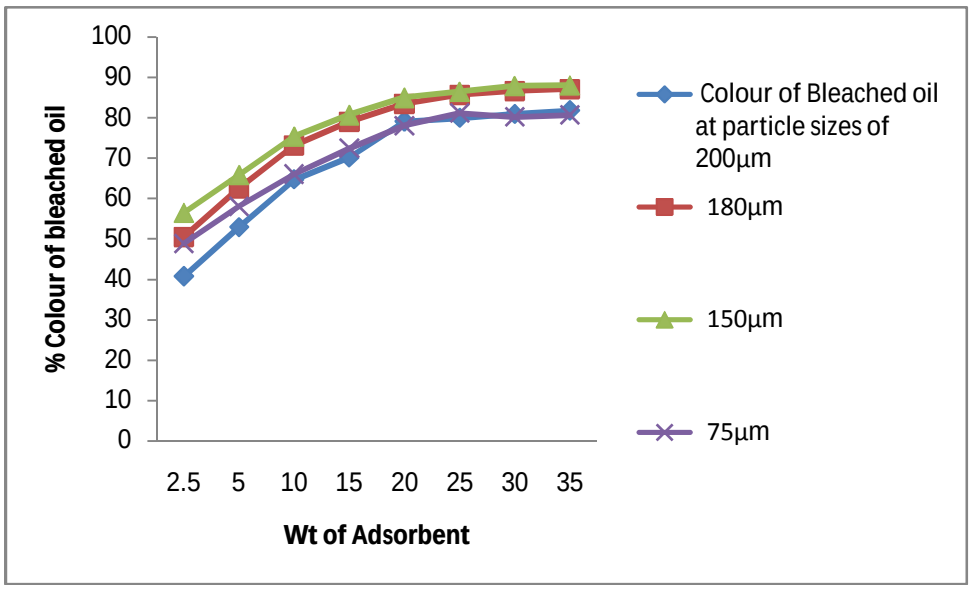

Fig iv: NAC and PO at varying particle sizes (constant temperature and time)

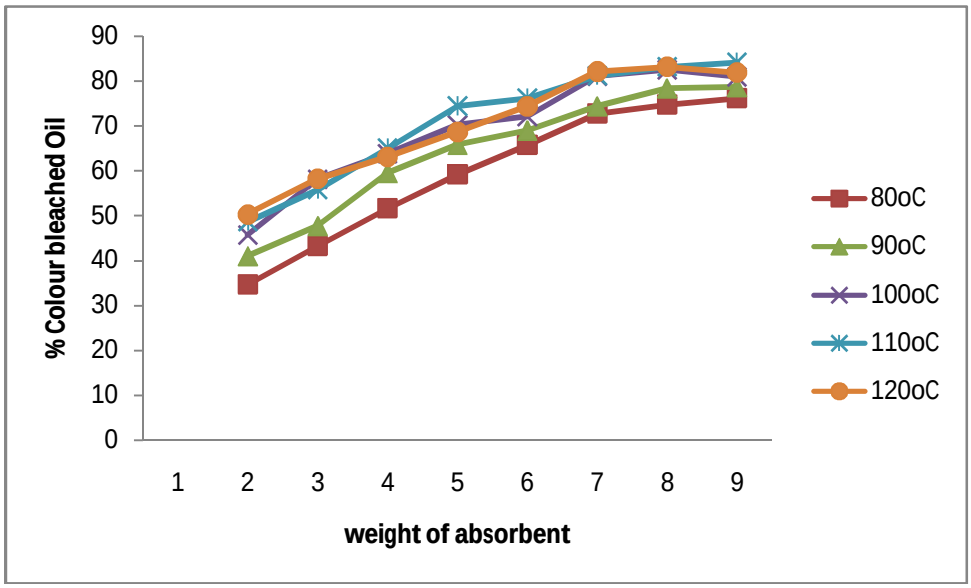

Fig v: CAC and PO at varying temperatures (time = 40 mins & particle size = 150µm).

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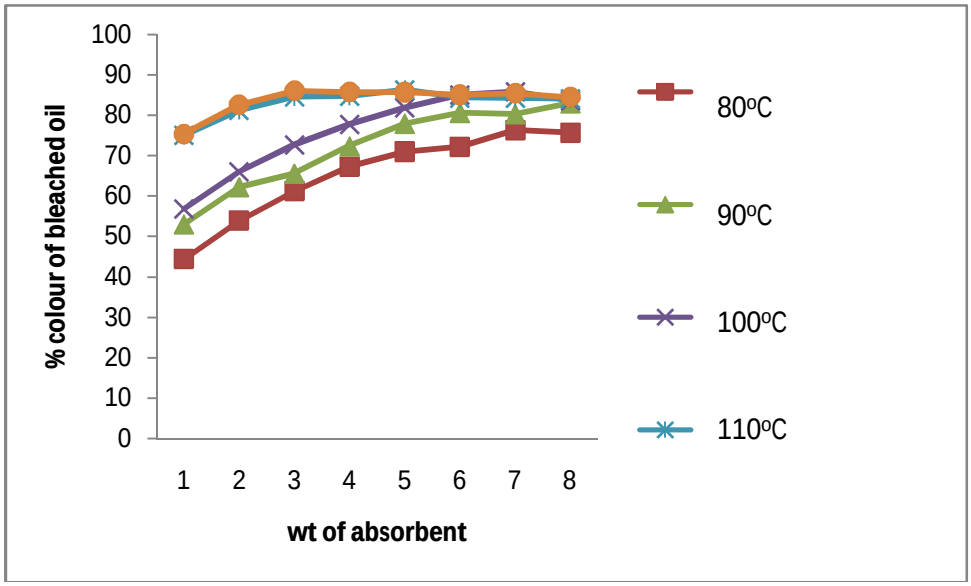

Fig. vi: NAC and PO at varying temperatures (particle size P_s = 150µm and time t = 40 mins)

The effect of temperature on these adsorbents shows that the adsorption capacity of both adsorbents CAC and NAC on the removal of carotene from palm oil varies with temperature. In figures v- vi, the adsorption capacity increases to an optimum value within the temperature range of 80^0 -110^0C. At higher temperature, the carotenoid molecules are activated and the energy imparted to these adsorbates increases their mobility and thereby providing quicker access to the carbon –liquid interface. Secondly, the external mechanical agitation applied might have contributed in an increased permeation rate of the solute particles (carotene) leading to increment in entropy of the system. However, at above 150^0C, the irreversibility of adsorption process sets in, leading to total degradation of the molecular constituents of the oil and subsequent distortion in the trend of carotene uptake by the adsorbents [2-3]. In addition, some substances after being adsorbed undergo a change so that they are no longer soluble in solution and such changes may be accelerated by elevation in temperature.

V. CONCLUSION

This study reveals that the uptake of carotene from palm oil by activated carbon is factor driven. The effect of each of these parameters investigated depicts a specific characteristic trend with a record of high significant reduction of carotene. Although, particle size analysis displaced a slight edge in adsorption efficiency over others. Remarkably, the degree of colour reduction achieved provides important information for the effective utilization of Enugu coal as adsorbent.

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