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Removal of Chlorine from Portable Water using Activated Carbon of Tiger Nut Chaff

OHIMOR, E. O. [1,*], *OSHIBAMOWO, O. R.* [2]

[1,2] *Federal University of Petroleum Resources, Delta State, Nigeria*

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ABSTRACT

Activated carbon is an economical and effective adsorbent for the purification of contaminated water in water treatment processes amongst other wide range applications in industrial practices. The aim of this research work is to produce, characterize and determine the adsorptive efficiency of activated carbon from tiger nut chaff as a good adsorbent for the removal of chlorine from potable water. The tiger nut chaff was carbonized and activated with tetraoxosulphate (vi) acid to widen the pores of the tiger nut chaff. The carbonized tiger nut chaff was examined by Scanning Electron Microscope (SEM) and X-ray diffraction spectroscopy (XRD) before and after adsorption. The activated carbon was used as adsorbent for the removal of chlorine from portable water via batch adsorption process. The experiment utilized the Central Composite Design of the Design Expert software in the analysis of experimental data from the adsorption process. The variables affecting the adsorption process are contact time and adsorbent dosage. Results obtained from the research shows that an increase in contact time and adsorbent dosage resulted in an increase in the removal efficiency. Experimental data were subjected to optimization with the aim of improving the adsorptive removal of chlorine by targeting the process parameters to the desired levels and it was described by 3D plot. Results from SEM graphs of the activated carbon shows that the adsorbent has rough texture with heterogeneous surface and a variety of randomly distributed pore sizes. Results from XRD graphs shows that sharp peaks were observed at 0KeV and 4.00KeV confirming the presence of carbon and oxygen by interacting with other functional groups. Conclusively, high percentage of chlorine was adsorbed on the microspores of the carbon structure which shows that tiger nut chaff activated carbon is a very good source of AC for adsorption of residual chlorine.

1. INTRODUCTION

The delivery of safe and quality drinking water is of high priority for human health. But the detection of off-taste and odour in drinking water is one of the major causes of criticisms from consumers to water companies. The appearance, taste and odour of drinking water is the only tangible means for consumers to judge water quality and this will build the trust of consumers and possibly avoid using water from unsafe source (Rogers, 2001). Chemical formed during water treatment, natural products in water, nature of the source, environmental factors and many others are various factors that can bring about odour and taste in portable water. The World Health Organization, (WHO) defines potable water as water that is clear

*Corresponding author, e-mail:Ohimor.evuensiri@fupre.edu.ng
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and transparent, odorless, tasteless, and microorganism-free or free from chemical substance in concentrations that can pose a risk to healthy living (Jaguaribe, 2005). Water fit for human consumption without going through any form of treatment will be known as natural potable water, which has no changes in its original chemical composition and microbial purity but often time conventional treatment are carried out to improve water quality for industrial and human consumption (Kebs, 2015).

The study of more eco-friendly techniques of water purification in Nigeria has prompted several researches on the production and characterization of activated carbons from agricultural wastes. Thus, due to the increasing demand of activated carbon, there is a strong need for the sorting out new precursors for the preparation of activated carbon which should be cost effective. Although, a variety of raw materials were explored for the preparation of activated carbon in earlier studies, agricultural wastes such as coconut shells (Azevedo *et al.*, 2007; Hu and Srinivasan, 2001), coconut fibres (Ohimor *et al.,* 2021a), wood (Gomez-Serrano *et al.,* 2005; Klijanienko *et al.,* 2008), cotton stalk (Deng *et al.,* 2010), almond shells (Bansode *et al.,* 2003), rice husk (Fierro *et al.,* 2010; Guo and Rockstraw, 2007), date pits (Girgis and EIHendawy, 2002) were used for the production of activated carbon. Scientists are still trying to explore new materials depending on their availability and suitability for activated carbon production. Nevertheless, the utilization of agricultural wastes as raw material for the preparation of activated carbon has increased notably in recent years. Activated carbons depend on the physical and chemical features of the raw materials as well as the activation method (Lua and Gua, 2001). Usually, the raw materials for the production of activated carbon are those with high carbon but low inorganic contents such as wood, lignite, peat and coal (Lua and Guo, 2001). The high adsorptive capacities of activated carbons are highly related to porous features such as surface area, pore volume, and pore size distribution (Das *et al.,* 2015; Ohimor *et al.,* 2021b).

The paper aims to produce, characterize and determine the adsorptive efficiency of activated carbon (AC) from tiger nut chaff as a good adsorbent for the removal of chlorine from potable water.

2. MATERIALS AND METHOD

2.1 Sample Collection

For this work, tiger nut was collected from Mami market in Uvwie Local Government Area of Delta State, State. The tiger nut was washed gently with water to remove mud and other impurities present on the surface and then sundried. It was then shredded into smaller particles. The tiger nut was washed again with water and squeezed to remove excess water. To further reduce the moisture content of the precursor, it was dried by atmospheric drying for 24 hours to obtain tiger nut chaff.

2.2 Preparation of the Activated Carbon

In this research, the tiger nut was put in a furnace at 500^0C for 1hour for it to carbonize. The carbon gotten is then crushed and sieved with a 300 micrometer sieve. The carbon was activated chemically by dissolving and impregnating it in 1litre of tetraoxosulphate VI (H_2SO_4) acid of 0.5M for 12hours and then sieved. The activated carbon was the neutralized by a continuous washing with distilled water till the pH is 7. The activated carbon is then thermal dried in a furnace at 400^0C

2.3 Chlorination of Potable Water

A 100ppm solution of chlorinated water was produced with 100mg of chlorine pellets added to 1litre of distilled water to produce 100mg/l solution of chlorinated water.

2.4 Adsorption Process

100ml of chlorinated water was poured into a conical flask with a specified amount of activated carbon added into it. It was then put in a water bath at constant temperature of

40^0C for a specified time. This process is a batch process. After the specified time has elapsed, the mixture of chlorinated water and activated carbon that was put in the water bath was filtered into a beaker using a filter paper and then the final concentration of the adsorbate was measured using a spectrophotometer with DPD1 as reagent. The process was repeated for as many runs required.

2.5 Experimental Design and Process Optimisation using RSM

Design Expert Software was used to create a Response Surface Methodology (RSM) model for the designing and analyzing of the experimental matrix, in order to measure the effect of various factors on the removal of chlorine from potable water. The levels of the studied factors are presented in Table 1 below;

Table 1: Design of Experiment Analysis of experiment factors

Factor	Low Factor Level (-1)	Mid-point Factor Level (0)	High Factor Level (+1)	Standard Deviation
Time (Min)	17.32	35.00	52.68	14.43
Adsorbent Quantity (g/ml)	3.17	6.00	8.83	2.31

3. RESULTS AND DISCUSSION

3.1 Scanning Electron Microscopy (SEM)

The prepared activated carbons were examined by Scanning Electron Microscope (SEM) to analyze the surface of the adsorbents. SEM micrographs of the chemically activated carbons by tetraoxosulphate (VI) acid (H_2SO_4) were presented in Figure 1. In all six cases, well-developed porous surface was observed at higher magnification. The pores observed from SEM images are having diameter in micrometer (μm) range. These pores are considered as channels to the microporous network. From the Figure, it can be observed that all the adsorbents have rough texture with heterogeneous surface and a variety of randomly distributed pore size.

The surface features of the prepared adsorbent (activated carbon produced from tiger nut chaff) before it was used for the adsorption shows a wide variety of pores, some shiny surface as spotted in the SEM Figure 1 with X10000 magnifications. This may be due to the reaction of the impregnating agent (H_2SO_4). Activated carbon with chemical activation has more advantage over physical activation (Lua and Gua, 2001), as a result of higher yields, more surface area and better porosity of carbon configurations. The particle sizes are irregular, containing high surface area available for adsorption of the adsorbate, which shows the adsorbent to be a good material for adsorption study (Das *et al.*, 2015; Ohimor *et al.*, 2021b).

observed 0KeV and 4.00KeV in case of the carbon before activation confirmed the presence of Carbon element and Oxygen by interacting with other functional groups. The sharp peaks observed 0.00KeV, 1.00KeV and 3.00KeV in the case of the carbon after activation may be due to the presence of hydrogen sulphate and also Carbon and Oxygen present.

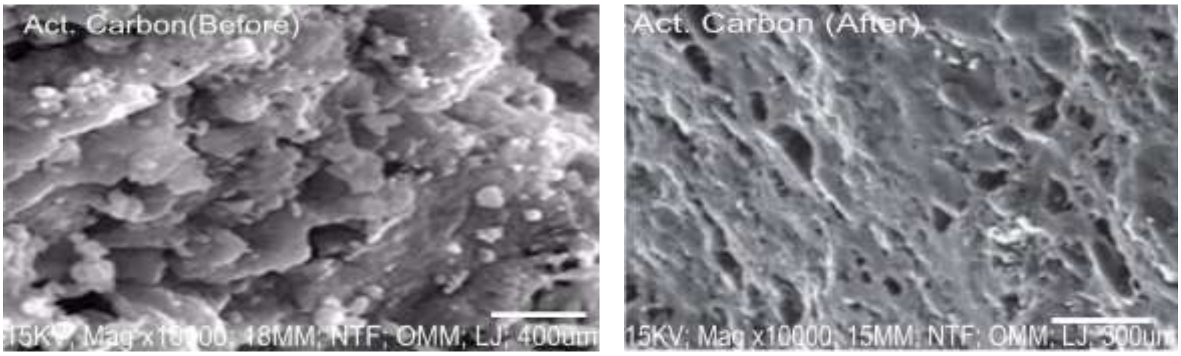

Fig 1: SEM images of the prepared Activated Carbon for before and after activation

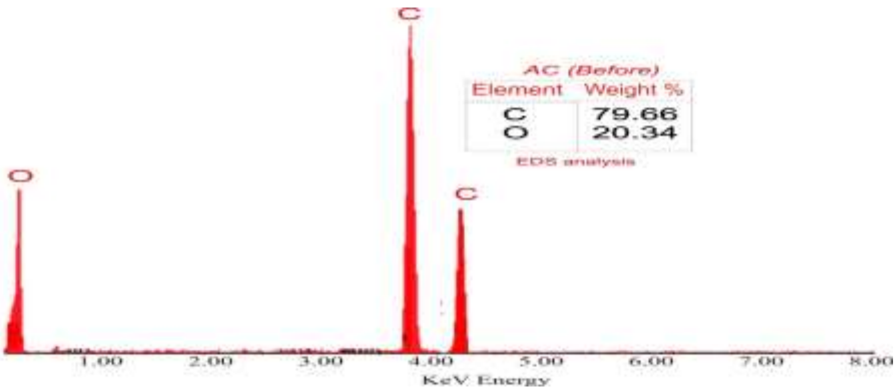

Fig. 2: XRD pattern of the carbon before activation

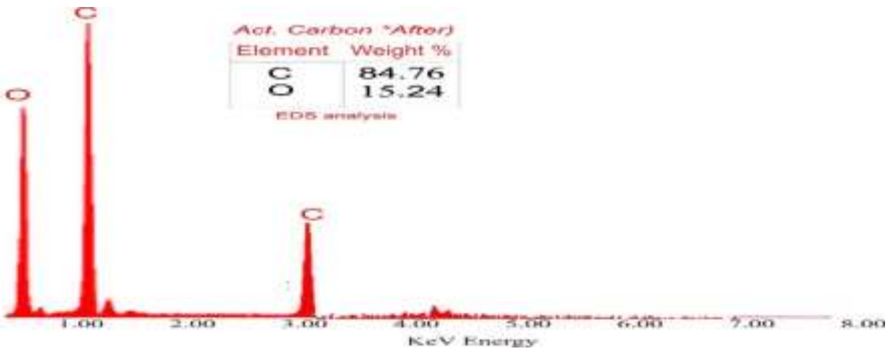

Fig 3: XRD pattern of the carbon after activation

3.3 Analysis of Response Surface Design (RSD)

Constant initial concentration and constant temperature of 40^0C was taken into consideration as well as different process parameters such as contact time and adsorbent dosage were taken as variables for the design of the experimental matrix. In analyzing the factorial design, the original measurement units for the experimental factors (actual units) were transformed into coded units. The factor levels are coded as –1 (low) and +1 (high). The response was expressed as the final concentration percentage (% R) of Chlorinated water.

The design matrix of all the factors in actual values for all the experimental runs is shown in Table 2 below;

*Corresponding author, e-mail:Ohimor.evuensiri@fupre.edu.ng
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Table 2: Design Matrix

Run Order	A	B	Response Values
	Time (min)	**Adsorbent quantity (g/ml)**	**Chlorine Concentration (%)**
1	35	2	93.42
2	17.3223	8.82843	84.87
3	35	6	89.47
4	52.6777	8.82843	78.95
5	35	6	89.47
6	35	6	89.47
7	35	6	89.47
8	52.6777	3.17157	86.18
9	35	10	75.66
10	60	6	84.21
11	35	6	89.47
12	17.3223	3.17157	87.5
13	10	6	86.18

After estimating the main effects, the effect of interactions were determined by performing the analysis of variance (ANOVA). Sum of squares (SS) of each factor quantifies its importance in the process and as the value of SS increases, the significance of the corresponding factor in the process also increases. The ANOVA results for the Cl_2 removal by AC were presented in Table 3 below;

*Table 1**:** ANOVA for Quadratic model*

Source	Sum of Squares	Df	Mean Square	F-value	p-value	
Model	243.54	5	48.71	10.64	0.0036	Significant
A-Time	12.57	1	12.57	2.74	0.1416	
B-Adsorbent Quantity	152.92	1	152.92	33.39	0.0007	
AB	5.29	1	5.29	1.16	0.3181	
A^2	35.55	1	35.55	7.76	0.0271	
B^2	46.60	1	46.60	10.17	0.0153	
Residual	32.06	7	4.58			
Lack of Fit	32.06	3	10.69			
Pure Error	0.0000	4	0.0000			
Cor Total	275.60	12				

*df = degree of freedom, Factor coding is **Coded**. Sum of squares is **Type III - Partial***

From the result of analysis of variance obtained in Table 3, it was seen that the *F*-value of the model is 10.64 which implies that the model is significant and the *P*-value of the model is 0.0036 which means that the probability of this *F*-value occurring due uncontrollable factors is only 0.36%.

A model reduction is considered in order to improve the quadratic model and this was done on the basis that any model term with *P*–value less than 0.05 is a significant model term, while model terms with *P*–value greater than 0.10 are insignificant model terms. Hence, the model terms in Table 3 that are significant are B, A^2, B^2. Thus the quadratic model is to be reduced to only these terms;

Concentration = 75.8876 + 0.573485 * A + 3.14143 * B + -0.023 * AB + -0.007234 * A^2 + -0.323516 * B^2

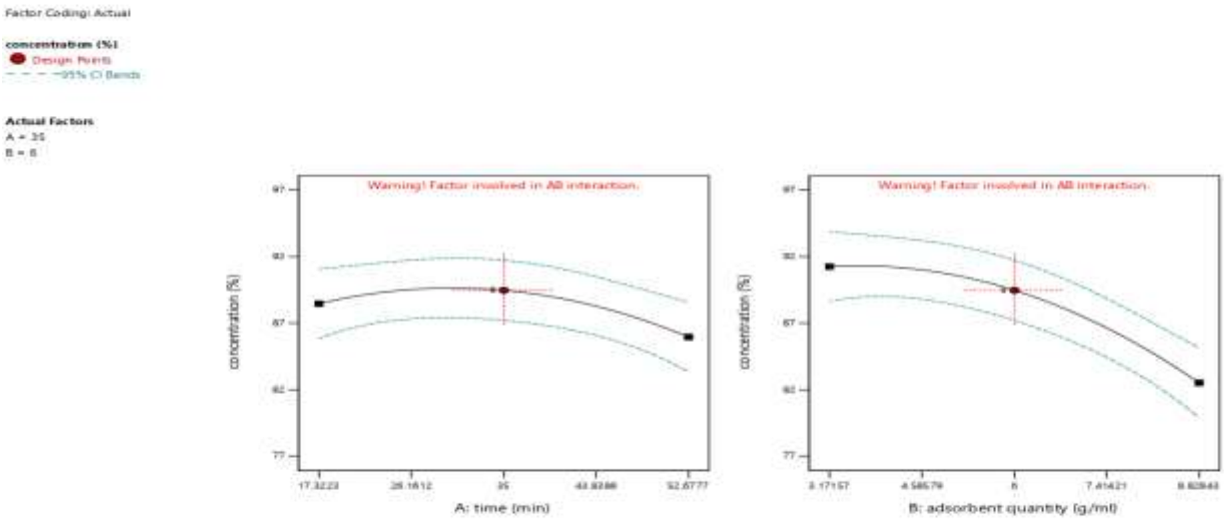

Fig. 4: Effect of main factors on *Cl_2 removal for activated carbon* (A) Time (B) Adsorbent quantity

3.4.1 Effects of main factors

The effects of main factors on the Cl_2 removal were shown in Figure 4 below. The main factors are contact time and adsorbent dosage i.e. with the increase of contact time and adsorbent dosage, the Cl_2 removal percentage increased to a great extent.

3.4.2 Interaction Effects of main factors

The contour plots of interaction of factors were shown in Figure 5. Compared to the effects of main factors, the effect of interaction of factors on the Cl_2 removal is very less.

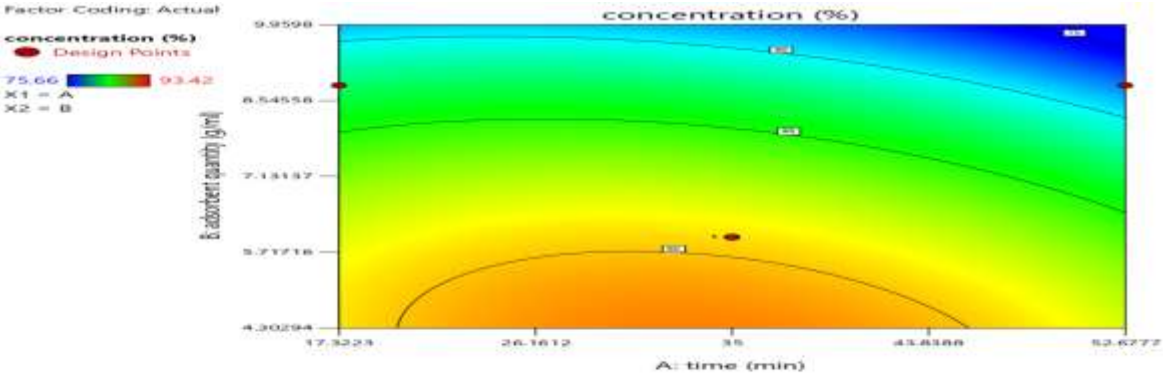

Fig. 5: Contour plots of interactions of Time and Adsorbent quantity for Activated Carbon

3.5 Optimization

Optimization of Cl_2 removal was carried out by a multiple response method called desirability (D) function to optimize the process parameters such as adsorbent dose and contact time. The goal of optimization was to improve adsorptive removal of Cl_2 by targeting the process parameters to desired levels. The 3D surface plot of the Cl_2 removal by AC was shown in Figure 6.

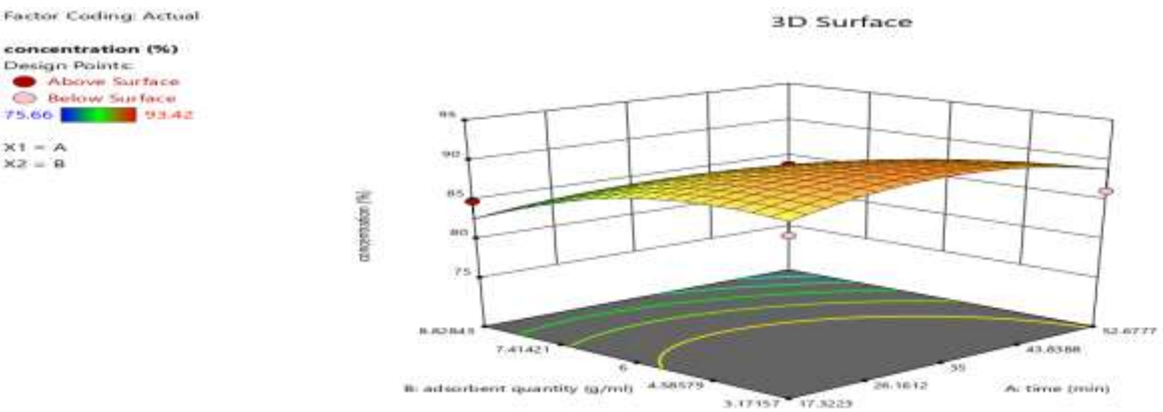

Fig. 6: 3D surface plot for Cl_2 removal by Activated Carbon

3.5.1 Normal Probability Plot

The normal probability plot indicates whether the residuals follow a normal distribution, in which case the points will follow a straight line. The normality of the data can be checked by plotting a normal probability plot of the residuals. If the data points fall fairly close to the straight line, then the data are normally distributed. The statistical analysis of the data in terms of the standardized residual was conducted to verify the normality of the data. The normal probability plot of the residuals of AC for Cl_2 adsorption was shown in Figure 7. The data points fairly close to the straight line indicate that the experiments came from a normally distributed population.

*Corresponding author, e-mail:Ohimor.evuensiri@fupre.edu.ng
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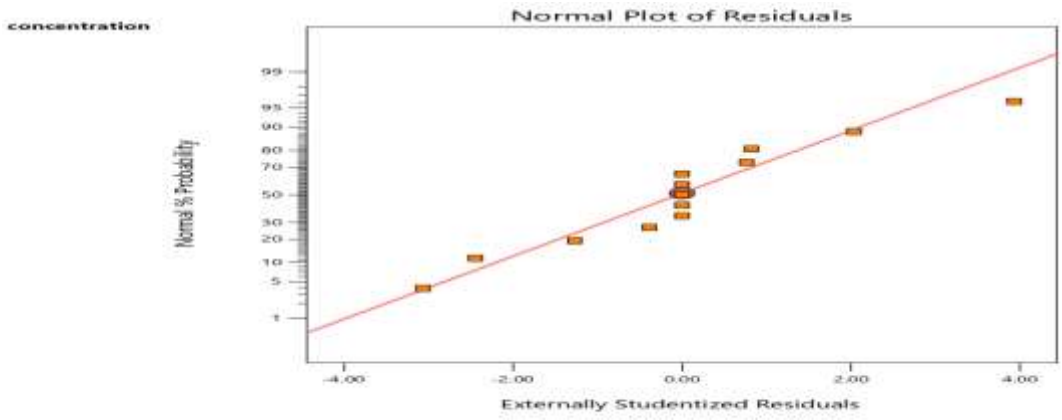

Fig. 7: *Normal probability plot of residuals for Cl_2 removal by AC*

4. CONCLUSION

The objective of the experimental study was to evaluate the potential of tiger nut chaff as a precursor for activated carbon production. Activated carbon with high microporosity was developed for this purpose. High percentage of chlorine (Cl_2) was adsorbed on the micropores of the carbon structure. Central Composite Design (CCD) was employed for the modeling of adsorption process by studying the effect of main factors and their interactions on removal of Cl_2 from potable water. The longer the contact time, the more the absorption and the higher the adsorbent dosage, the more the adsorption of chlorine. Based on detailed experimental investigation, we observed that tigernut chaff activated carbon is a very good source of AC for adsorption of residual chlorine.

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