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Preparation and Characterization of African Star Apple Seed Shell (*Chrysophyllum Africanum*) For The Removal of Acid Red 9

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Abstract: *In the present work, activated carbon (AC), prepared from the shells of African star apple seed (Chrysophyllum Africanum) (and modified with H_3PO_4) were characterized (with SEM and FTIR) and applied for the adsorption removal of Acid red 9 dye from aqueous solution. The scanning electron micrograph of the adsorbent showed an unsmooth appearance with irregularly arranged molecular units while the FTIR spectrum showed some carbon associated bonds. The application of the Central Composite Design (CCD) for response surface analysis led to a projection that 97% removal of acid dye could be achieved in 60 minutes with an adsorbent dosage of 1.4 g at a pH of 6. The adsorption isotherm parameters were analyzed. The adsorption process followed Freundlich isotherm model. The adsorption capacity of this seed can be utilized for the treatment of strongly polluted industrial wastewaters containing acid dye*

Keywords: *Water contamination, dye, remediation, adsorption, activated carbon African apple seed*

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1.0 Introduction

Some industrial sectors (such as textiles, leather, paint and others) employ various dyes and pigments that have the potential to contaminate the aquatic system through effluent discharge (Eddy and Garg, 2021; Uchechukwu *et al*., 2018). The toxicity of several dyes has been reported including obscurity against the penetration of light to the water (and associated interference with the mechanism of photosynthesis), complex formation with metal ions in the water and others (Odoemelam *et al*., 2018). Some health-related challenges have been confirmed to be associated with the consumption of dye contaminated water, ranging from headache to cancer and death (Qada *et al*., 2017). Given their established toxicity, dye removal technologies such as precipitation, adsorption, oxidation and others have been investigated for their removal efficiencies (Eddy, 2009). However, adsorption is one of the best methods because it can be conducted at a cheaper cost, the process can easily be implemented and proper materials selection can be done to utilize eco-friendly and biodegradable components (Uchechukwu *et al*., 2015). Acid red 9 is one of the commonly used dyes and its toxicity has been established (Fernandez *et al*., 2007). Several studies have been reported on the adsorption removal of some acid dyes using different adsorbents.

Among all the adsorbents reported, activated carbon is known for its outstanding performance. However, the major challenge facing the application of activated carbon for the adsorption removal of contaminants from water is the high cost (Ozer and Dursun, 2007). To overcome this challenge, several alternative routes of synthesis have been investigated and most of them have been found to deliver efficiency that is not significantly different from the conventional charcoal (Krowiak *et al.,* 2014). In the present study, the shell of *Chrysophyllum Africanum* seed is investigated for its potential in synthesizing activated carbon and to apply the synthesized shell for adsorption removal of Acid red dye from aqueous solution. This shell has some inherent adsorption properties. For example, Amuda *et al*. (2007) decontaminated lead polluted water using the shell of *Chrysophyllum Africanum* seed as adsorbent.

2.0 Materials and Methods

2.1 Preparation of Acid red dye solution

Samples of African star apple seed shell was collected from the Yan Goro market in Kaduna state. The samples were thoroughly washed to remove all foreign materials. The samples were sundried to constant weight and then in an oven at 100 ºC for 24 hours. They were crushed to a powdered form and then carbonized using the method described by Yahya *et al* 2015. About 100 g of the sample was carbonized at a temperature of 400 ºC for 2 hours to produce activated carbon, Modification of the adsorbent was carried out by mixing it with H_3PO_4 at a ratio of 1: 2 and the carbonization were repeated under the same nitrogen atmosphere and ther similar conditions. The product was first washed with H_3PO_4 and then with distilled water. It was finally dried in the oven to constant weight at a temperature of 110 °C.

2.2 Proximate analysis

The moisture content was determined by the oven drying method at 105 °C. The difference in weight before and after drying gave the moisture content. The ash content was evaluated as the weight of the samples that remains after ashing the sample in a furnace at 500 °C **(**Ilaboya *et al.,* 2013). The bulk density of the sample was measured as the weight of the sample divided by the volume. The weighing was carried out using a graduated cylinder.

The yield of activated carbon (Fixed Carbon Content Determination) was defined as the ratio of the weight of the activated carbon product to that of the original raw material (both weights were on a dry basis).

$$Y_{ch} = (\frac{W_{ch}}{W_o}) \times 100 \qquad (1)$$

where W_{ch} is the weight of carbon retrieved from the furnace and W_o is the dried weight of the sample.

2.3 Characterization of the activated carbon

A FTIR spectrometer (Agilent Technologies Cary 630 USA) was used to detect the functional groups in the sample. The scanning was done within the range, 400 and 4000 cm^{-1}. And the resulting information was recorded as a spectrum.

2.3.1 Scanning electron microscopy (SEM)

The morphology of the activated carbon and its surface pores were ascertained using a scanning electron microscope (FEI Quanta 250 USA).

2.3.2 FTIR analysis

The FTIR analysis was carried out between the frequency range of 400 and 4000 cm^{-1} using Agilent Technologies Cary 630 USA FTIR instrument

2.4 Adsorption studies

The maximum wavelength of absorption of Acid dye was evaluated by plotting values of absorbance that correspond to the various wavelengths. The observed λ_{max} was used as the reference wavelength for all measurements. A calibration curve was prepared by plotting values of absorbance against the respective concentrations of serially diluted solution. The UV spectrophotometer (Agilent Technologies

Cary 300 Series) was used for all spectroscopic analyses. All other concentrations were evaluated through extrapolation from the calibration curve.

Batch adsorption experiments were carried out as reported elsewhere (Odoemelam *et al*., 2018). . The percentage removal of acid red 9 was calculated using the following equation;

$$Adsorption\ Efficiency = \frac{C_i - C_f}{C_i} \times 100 \quad (2)$$

where: C_i and C_f are the initial and final concentrations of the dye in the solution.

3.0 Results and Discussion

The average proximate information of the seeds sample was moisture (2.90%), ash (1.3%), C (61.17%) and the yield was 62.23 % while the bulk density was 0.543 %.

The high fixed carbon makes this material a good precursor for the production of activated carbon (De *et al.,* 2013). The bulk density of activated carbon usually suggests that if the activated carbon is added to water it will sink and this will give better contact with the adsorbate and thereby leading to an effective adsorption process. The bulk densities obtained in this study revealed that the African star apple seed shell could be of great potential for wastewater treatment as the values are higher than 0.25 g/cm^3, the minimum required for commercial adsorbents as reported by Denver 1991.

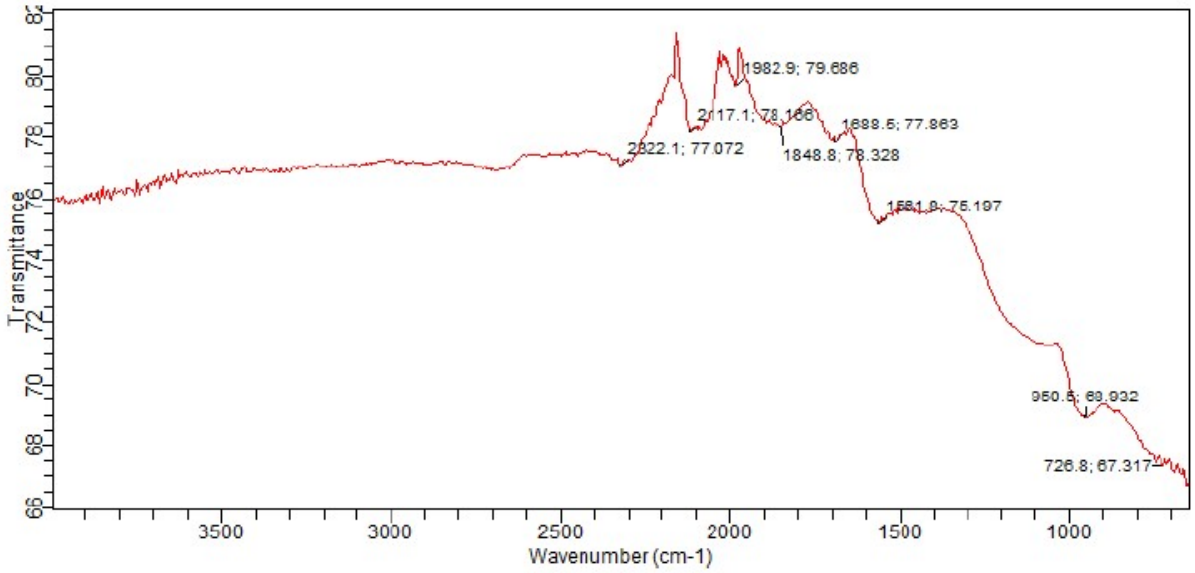

Fig 1: FT-IR Spectroscopy of African star apple seed shells activated carbon

The FTIR spectrum of the prepared activated carbon is presented in Fig. 1. The band observed at 2822.1 cm^{-1} is due to O – H stretching vibrations, whiles the bands at 2117.1 cm^{-1}, 1982.9 cm^{-1}, 1848.4cm^{-1}, 1688.5 cm^{-1}, 1561.8 cm^{-1}, were assigned to the stretching vibration of C≡C, C═C, C-CH$_j$ and C−O−H respectively. Other bands at 950.5 cm^{-1} and 726.8 cm^{-1} may be ascribed to twisting vibration of the C=C−H bond. This showed that the prepared activated carbon has some functional groups that may be useful for the adsorption of the dye onto its surface (Adib *et al.,* 2016)

Fig. 2 shows the surface morphology of the adsorbent, as revealed by a scanning electron micrograph. Examination of the micrograph reveals that the system consists of irregular molecular arrangements with observed surface heterogeneity and pores for trapping adsorbate. Similar patterns have been reported by Buvaneswar and Singanan (2020) for some activated carbon materials. Arroyo-Gomez *et al.* (2018) have also attributed some of the

observed features in the micrograph to good textural properties that are also relevant towards enhanced adsorption. This indicates the possibility of good textural properties (Arroyo-Gomez *et al.,* 2018). However, the observed micrograph is slightly at variance with the one presented by Khaniabad, *et al*. (2015) for activated carbon produced from aloe vera waste. The difference may partly be due to modification if out adsorbent with H_3PO_4.

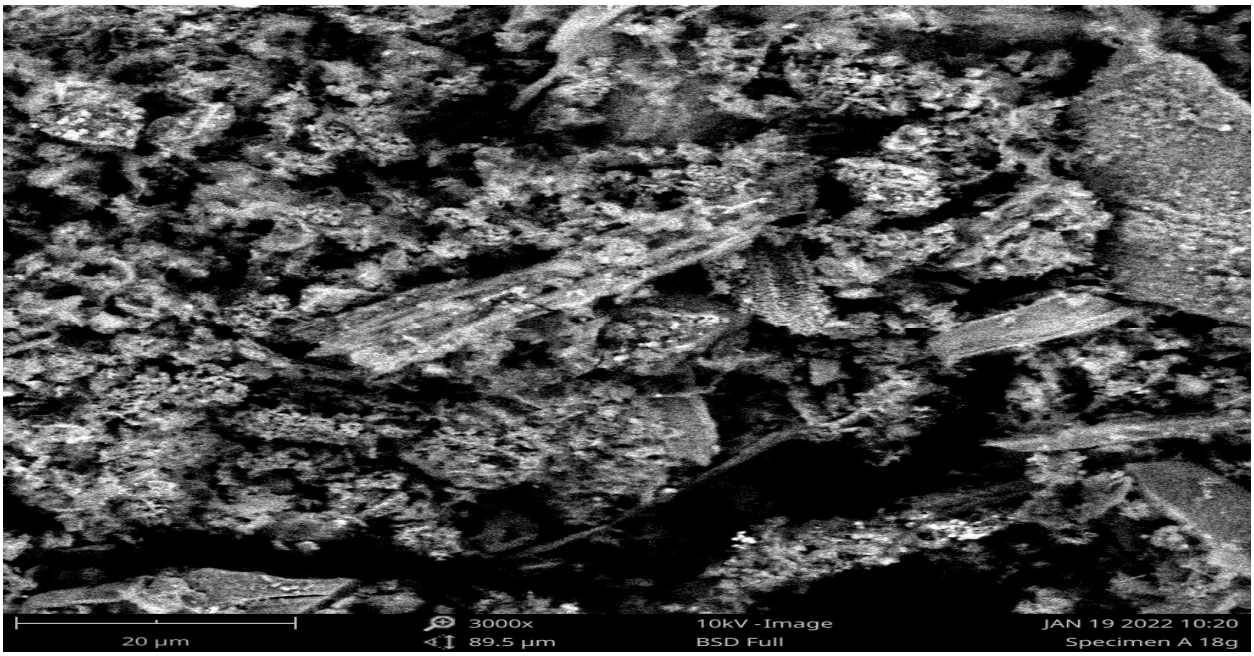

Fig. 2 Scanning electron micrograph of H_3PH_4 modified activated carbon

Ziezio *et al*. (2020) had reported the mechanism of involving in the modification of activated carbon with H_3PO_4 at a temperature range of 373 to 773 K as follows,

$$2H_3PO_4 \rightarrow H_4P_2O_7 + H_2O \quad (3)$$

$$2H_3PO_4 \rightarrow H_5P_3O_{10} + 2H_2O \quad (4)$$

$$nH_3PO_4 \rightarrow H_{n+2}P_nO_{3n+1} + (n-1)H_2O \quad (5)$$

The above mechanism entails that as long as the number of n (equation 5) increases, the ease of adsorption increases. It is generally accepted that H_3PO_4 act as an acid catalyst by enhancing bond cleavage reactions. It can also act as a reactant to create crosslinks through cyclisation and condensation. In the presence of organic matter, the acid can combine to form a phosphate bridge.

Table 1 below showed the design matrix consisting of each factor combination and their respective response which is the percentage removal of acid red 9 from the simulated solutions. The data presented in the table showed various adsorption efficiencies for the respective experimental runs, with percentage removal ranging from 25% down to 97%. A quadratic model suggested for the adsorption as shown in Table 2 had a coefficient of determination (R^2) = 0.8627 and a p-value of 0.001.

Table 1: Experimental runs and responses

S/No	C (mg/L)	pH	Dosage (g)	Time (mins)	% Removal
1	10	3.5	1.25	120	89
2	25	3.5	2.00	5.0	91
3	25	3.5	1.25	62.5	93
4	25	6.0	1.25	5.0	87

5	40	1.0	1.25	62.5	41
6	25	3.5	1.25	62.5	78
7	40	3.5	1.25	120	61
8	40	6.0	1.25	62.5	88
9	40	3.5	1.25	5.0	39
10	10	3.5	2.00	62.5	69
11	40	3.5	0.50	62.5	34
12	25	3.5	0.50	120.0	52
13	25	1.0	1.25	5.0	25
14	25	3.5	2.00	120	43
15	40	3.5	2.00	62.5	57
16	10	3.5	1.25	5.0	69
17	25	3.5	1.25	62.5	63
18	25	6.0	2.00	62.5	97
19	25	3.5	1.25	62.5	83
20	25	3.5	0.50	5.0	41
21	25	1.0	0.50	62.5	39
22	10	3.5	0.50	62.5	62
23	10	1.0	1.25	62.5	58
24	25	6.0	1.25	120.0	79
25	25	1.0	1.25	120.0	46
26	25	6.0	0.50	62.5	68
27	25	1.0	2.00	62.5	40
28	10	6.0	1.25	62.5	96
29	25	3.5	1.25	62.5	68

Table 4: Model Fit Summary

Parameter	Value
Standard Deviation	11.11
R^2	86.27%
R^2_{adj}	72.54%
R^2_{pred}	59.61%

The ANOVA for the full quadratic model below shows an F-value of 6.28 implies the model is significant (Table 3). There is only a 0.07% chance that an F-value this large could occur due to noise. P-values less than 0.0500 indicate model terms are significant. In this case, A, B, C, CD, C^2, D^2 are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model.

The Lack of Fit F-value of 0.81 implies the Lack of Fit is not significant relative to the pure error, non-significant lack of fit is good.

Table 3: ANOVA for the full quadratic model

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	10861.58	14	775.83	6.28	0.0007	significant
A-concentration	1260.75	1	1260.75	10.21	0.0065	
B-pH	5896.33	1	5896.33	47.76	< 0.0001	
C-dosage	850.08	1	850.08	6.89	0.0200	
D-time	27.00	1	27.00	0.2187	0.6472	
AB	20.25	1	20.25	0.1640	0.6916	

AC	64.00	1	64.00	0.5184	0.4834	
AD	1.0000	1	1.0000	0.0081	0.9296	
BC	196.00	1	196.00	1.59	0.2283	
BD	210.25	1	210.25	1.70	0.2129	
CD	870.25	1	870.25	7.05	0.0188	
A^2	126.53	1	126.53	1.02	0.3285	
B^2	119.47	1	119.47	0.9677	0.3420	
C^2	1124.50	1	1124.50	9.11	0.0092	
D^2	590.55	1	590.55	4.78	0.0462	
Residual	1728.42	14	123.46			
Lack of Fit	1158.42	10	115.84	0.8129	0.6418	not significant
Pure Error	570.00	4	142.50			
Cor Total	12590.00	28				

The regression equation observed from modelling of the adsorption parameters is expressed in equation 6

$$Removal\ (\%) = 77.00 - 10.25A + 22.17B + 8.42C + 1.50D + 2.25AD + 4.00AC + 0.50AD + 7.00BC - 7.25BD - 14.75CD - 4.42A^2 - 4.29B^2 - 13.17C^2 - 9.54D^2 \quad (6)$$

where A is the concentration of the dye, B is the pH of the bulk solution, C is the adsorbent dosage and D is the period of contact. A quadratic model suggested for the adsorption as shown in Table 3 had a coefficient of determination (R^2) = 0.8627 and a p-value of 0.007, from Table 4. The p-value less than 0.05 indicates that it is significant. This was further confirmed by the high R^2 of the model showing its high significance. The lack of fit had an F-value and p-value of (0.8129 and 0.6418,) respectively as shown in table 3, pointing to its insignificancy. With all these conditions met, the model is said to be a good one (Garba *et al.,* 2016).

3.1 Some interactive effects of variables on adsorption

3D plots of interactive effects of the factors are presented in Figs. 3 to 6 respectively. The 3D plot of the interactive effect of pH and concentration of Acid red 9 in the samples in Fig. 3. From the plot, it can be deduced that the concentration is inversely proportional to pH. Thus, as the pH of the system increases (i.e from a high acidic medium to a lower acidic medium 2 to 6 on the pH scale), the final concentration of acid red 9 decreases. Likewise, it can be said that acid red 9 is adsorbed more efficiently at a low acidic medium (pH of 6) by African star apple-activated carbon.

From the 3D plot of the effect of pH and adsorbent dosage on the final concentration of acid red 9 is presented in Fig.4, it was observed that the adsorbent dosage did not have much of a significant impact on the final concentration of acid red 9. This finding implies that the amount of African star apple seed shell activated carbon used did not significantly affect the adsorption of acid red 9 as initially predicted. Again, it was observed that at high pH (lower acidic medium), the percentage adsorption increased. This aligned with findings in Fig. 5.

Figure 6 presents the effect of pH and contact time on the adsorption of acid red 9. From the plot, it was observed that the pH of acid red 9 is directly proportional to the contact time. It was however observed that the relationship between pH and the concentration was stronger than that with the contact time. This finding implies that more acid red 9 is removed at higher pH (pH of 6) and longer contact time.

This finding is similar to that reported in the literature. The longer the contact time, the more of a compound is adsorbed (Dong *et al.,* 2010) where acid red 9 removals were greatly influenced by low acidic pH conditions enabling the deprotonation of acid red 9 to form organic anions.

Figure 5 present the effect of contact time and adsorbent dosage of African star apple seed shells. From the plot, it was deduced that contact time is directly proportional to adsorbent dosage with a very strong relationship. This implies that a high adsorbent dose creates more pore spaces that will accommodate more acid red 9 molecules within a longer time.

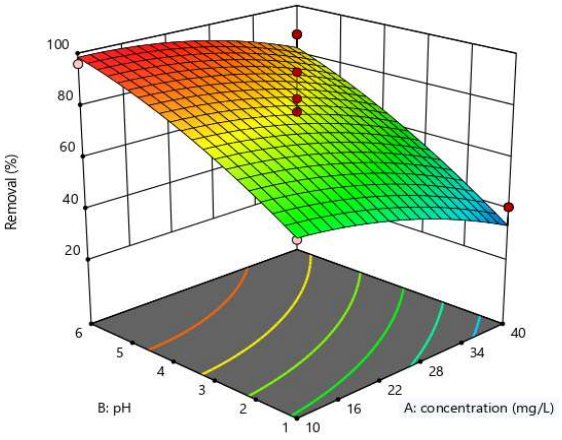

Fig. 3: Interactive effect between pH and concentration

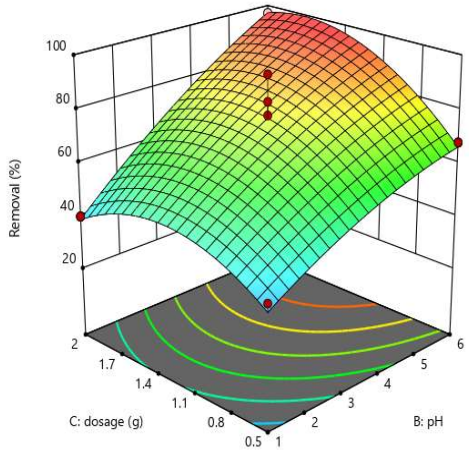

Fig. 4: Interactive effect between pH and adsorbent dosage

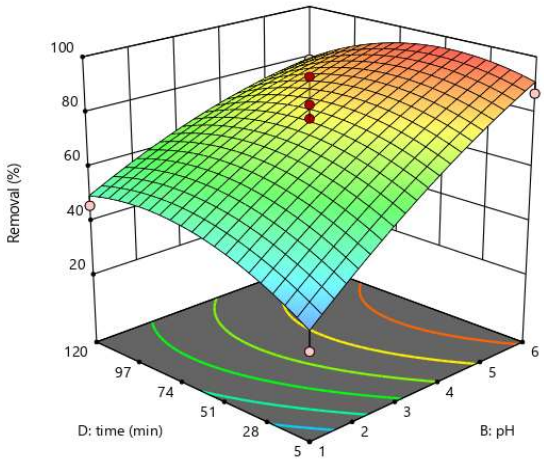

Fig. 5: Interactive effect between pH and contact time

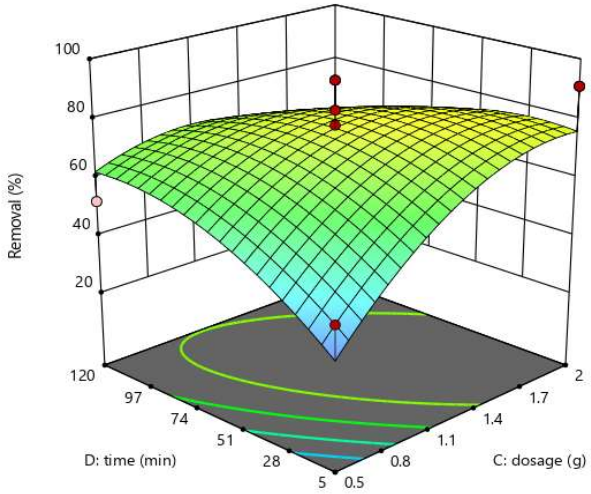

Fig. 6: Interactive effect between dosage and contact time

3.2 Freundlich Isotherm

Freundlich isotherm model describes the sorption on a heterogeneous surface which means, adsorption of solutes from a liquid to a solid surface.it also assumes that different sites have different adsorption energy due to surface heterogeneity (Meroufel *et al.,* 2013)

The empirical equation proposed by Freundlich is given as equation 7

$$q_e = K_f C_e^{1/n} \quad (7)$$

The equation when rearranged to obtain a linear form by taking logarithms of q_e becomes:

$$\log q_e = \log K_f + \frac{1}{n}\log C_e \quad (8)$$

where C_e is the equilibrium concentration of the adsorbate, K_F and n are Freundlich constants.

A straight line plot for $\log Q_e$ against $\log C_e$ was obtained.The slope and the intercept correspond to $\frac{1}{n}$ and respectively. From the plot (figure 6), the high correlation coefficient values (R^2) for the removal of acid red 9 using African star apple seed shells 0.7180 obtained from the adsorption study showed that the isotherms fitted well with the Freundlich model which is an indication of cooperative adsorption in sites with different binding energies. This supports the findings of Abechi *et al*. (2013).

Table 5 has the correlation parameter of Freundlich isotherm for acid red 9 removals. K_f (3.963774 Lmg^{-1}) value depicts the maximum amount of acid red 9 on the adsorbent with adsorption intensity, n value of 1.4541 suggesting a favorable acid red 9 sorption hence 1/n >1.

The Freundlich adsorption constant was used to estimate the standard free energy of adsorption of the dye according to equation 7 (Eddy *et al*., 2010),

$$\Delta G^0_{ads} = -RTlog(k_F) \quad (7)$$

The result indicated the free energy of adsorption to be negatively less than the threshold value required for the mechanism of chemisorption. Therefore the adsorption of the dye followed physisorption mechanism (Eddy, 2010)

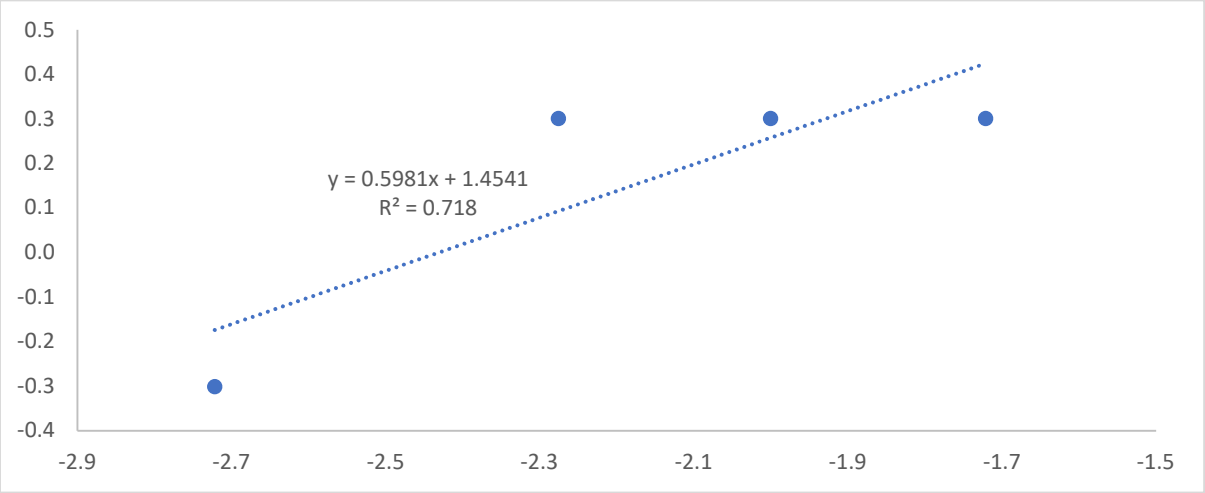

Fig. 6: Freundlich isotherm for adsorption of acid red 9 on the surface of activated carbon

Table 5: Freundlich parameter for the adsorption of Acid Red 9

Parameter	Value
Intercept	0.5981
N	1.4540
K_f	3.9638
R^2	0.7180
ΔG^0_{ads} (J/mol)	-11.4531

4.0 Conclusion

The results and findings of the study reveal that the shells of the African star apple can be modified to serve as an adsorbent for the removal of toxic dyes such as Acid red 9. The adsorption process is spontaneous and could be explained by the Freundlich adsorption isotherm. Also, the adsorption mechanism is physisorption and is dependent on the initial concentration of the dye, time, pH and adsorbent dosage. Therefore, further study and the application of this plant shell for the removal of water contaminants is recommended.

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