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Kinetics and Adsorption Studies of Methyl Red on Sheanut Shell Activated Carbon

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Theproduction of activated carbon from sheanut shell was studied and investigation of its effective adsorptive power in the removal of methyl Red azo dye from aqueous solution was carried out. The bio-material was activated using zinc Chloride, through a one step process. Some parameters were measured, which includes the effect of pH, amount of adsorbent, contact time, concentration of the adsorbate and effect of temperature. The iodine adsorption number (IAN), also indicates the high adsorption capacity of the produced activated carbon. The XRD result for the powered particles shows an amorphous structure. The results of the equilibrium data obtained from the experiments were fitted to the Langmuir and Freundlich isotherm models. The results obtain shows that the adsorption process follows the Freundlich model, which suggest a heterogeneous layer adsorption process with a R^2 value of 0.8. Specific rate constants were tested using two kinetic models; the pseudo-first and the pseudo-second order kinetics. The results obtained showed that pseudo-second order adsorption kinetics was obtained for all cases. The Arrhenius and Van't Hoff plots show the reaction is spontaneous, endothermic and it's a physical reaction process.

Keywords: Adsorption isotherm, Activated charcoal, Thermodynamics

I. INTRODUCTION

The presence of dyes in the aquatic environment as a result of production and dying processes causes advance effect on the ecosystem and human health (Inthom,D.etal 2004). Many of the dyes have mutagenic, carcinogenic and teratogenic effects when in contact with skin or inhaled. The dye effluents also hampered the growth and photosynthetic activity of bacteria in the water bodies.

Physical adsorption is the primary means by which adsorbent works to remove contaminants from water. The most widely used adsorbent is activated carbon (Baker,I.Setal 1992), because of Carbon's highly porous nature which provides a large surface area for contaminants (adsorbates). Activated carbon can be prepared either by physical or chemical means, using a variety of starting materials. Most starting materials reported in the preparation of activated carbon includes coconut shells, shell hull palm tree, apricot stones, almond shells and animal waste, with the most popular being Wood charcoal or coal (Itodo, I.A etal 2008). The most effective pollutant removal process universally accepted is adsorption, due to ease in handling, low consumption of reagents, ease for recovery of value added components through desorption and regeneration of adsorbent, and low cost (Rengarag, S. etal 2002 and Maryam, K. etal 2008).To use the adsorption isotherms, in predicting the expected adsorption in the process of adsorption, the feasibility of the activated carbon, and the design of the most effective way to remove the contaminant are required (Hameed, B.H and Hakimi, H.2008).The adsorption studies of methyl red on lemon grass activated charcoal at 30^{o}Cwas reported to be endothermic and spontaneous in nature, with optimum removal at pH 2 (MohdAzmierAhmadetal 2019)

Methyl red (MR) is an hazardous anionic dye, commonly used as mono azo dye in textiles, laboratory assays, and other products. The shea tree (vilellariaparadoxa) is a leafy vegetable, and one of the economic tree crops prevalent in the northern Nigeria.

We report here the adsorption performance of sheanut shell activated carbon of methyl red in aqueous solution. The functions of pH, time, and temperature on the adsorption of methyl red were studied also. The kinetics, isotherms and thermodynamic parameters were determined.

Experimental Procedure

The reagents used in this study are all of analytical grades from Sigma or Aldrich ltd, and used without further purifications.De-seeded

Sheanut shells were obtained from a local mill in Abuja F.C.T, Nigeria. It was pre-treated and activated using thermo-chemical method described by Turoti, M.etal (2007).Thepre-treated Sheanut shell was carbonized and activated using 1 M $ZnCl_2$(Khalili N.R, 2000)˙ The percentage yield, burn-off and moisture content were assessed using known relations(Odebunmi, E. and Okeola, F. 2001)

DETERMINATION OF POROSITY

The porosity was assessed based on swelling and Iodine Adsorption Number (IAN)using a known relation

$IAN = C_s(V_b - V_s)/2M_a$------------- 1

Where: C_s is the molarity of thiosulphate solution (mol/dm^3), V_s is the volume of thiosulphate (cm^3) used for titration of the AC aliquot, V_b is the volume of thiosulphate (cm^3) used for blank titration and M_a is the mass of AC (g).

SURFACE MORPHOLOGY AND SPECTROSCOPIC STUDY

The X-ray Diffraction (XRD) analysis were carried out with PANalytical X-ray, Philips Analytical, and the spectra were analysed using PC-APD diffraction software. The Spectroscopic studies were carried out using Fourier Transform Infra-Red (FTIR) spectroscopy was obtained with FTIR-84005, SHIMADZU corporation, on a KBr disc.

Batch adsorption studies.

The solutions of pH 2.0-7.0 were prepared by addition of 2 ml of the stock solution of the dye, and diluted to 100 ml of the buffer solutions of the respective pH. The pH was monitored pH meter Model HM-7E, the pH was adjusted by adding appropriate amount of either 1 M NaOH or 1 M HCl solution before each experiment. The absorption spectra were recorded on a UV-Visible SP 250 spectrophotometer. The concentration of the remaining amount of dye was calculated from their absorbance.(Gimba, C.E 2001)

a- Effect of PH and contact time

The efficiency of sorption is dependent on the pH of the solution, because variation in pH leads to the variation in the degree of ionization and the surface properties of the sorbent (Langmuir, I. 1918). In view of this, comparative experiments were performed over a pH range 2.0-7.0, and contact time 0-180 minutes, to obtain the optimum pH for dye adsorption and also the optimal wavelength at variable pH values. The optimal wavelength occurs at pH 3, with a wavelength of 490 nm. Therefore, all subsequent studies werecarried out at pH 3 and wavelength of 490 nm.

b- Effect of adsorbent concentrations

The dye solutions of 50, 100, 150, 200, 250 and 300 mg/L with fixed adsorbate of 0.2 g in a250 ml Erlenmeyer flask, at PH 3, at ambient temperature for 120 min. was use for this study.

c- Effect of temperature

The adsorption experiments were carried out to study the effect of temperature variation, at 298K, 300K, 303K and 313 K in a water bath incubator, at optimal pH value of 3 and adsorbent dose of 0.1 g. The equilibrium contact time for adsorption was varied between 10 - 120 min at agitation speed of 120rpm

II. RESULTS AND DISCUSSION

Proximate analysis of the Sheanut Shell (SS), Char Sheanut Shell (SS char) and Activated Carbon Sheanut Shell (ACSS) (< 2mm aperture) samples are shown in Table 1. SS was shown to have high moisture content, pH value and bulk density than both the char and activated carbon. Activated carbon has high porosity based on swelling than the char and SS. The SS has high moisture content, which is asaresult of the presence of organic content and liquid. The result shows that the moisture and volatile content decreased significantly during the activation process, which is largely due to the loss of the organic substancesduring thermal heating.

Table 1: Some physical properties of SS, Char and activated carbon

Sample	% yield	% Burn off	Bulk density (g/cm^{-1})	pH	Porosity	% Moisture c	% Ash	Colour
S.S	_	_	**0.524**	**9.20**	**0.718**	**3**	__	Dark Brown
Char	**35.6**	_	**0.377**	**7.10**	**0.401**	**2.08**	**26.05**	Black
AC S.S	**30.5**	**14.33**	**0.345**	**7.57**	**0.952**	__	__	black

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IODINE ADSORPTION NUMBER (IAN)

The carbon particles adsorb some amount of iodine molecules (IAN) when added to 0.02N iodine solution. From the result obtained, the IAN for AC S.S estimated in mg iodine per gram of adsorbent is fairly high (0.550), as compared to that reported by Itodo, I.A etal (2008) for shea nut shells (0.1338 - 0.1505) and groundnut shells (0.1115 - 0.1394). It thus implies that the degree activation and affinity of small particle contaminants to AC S.S will be high.

FT-IR analysis

The FT-IR spectroscopic study of theactivated sample showed three major absorption bands at 3406cm^{-1}, 2354.2cm^{-1}and 1680cm^{-1}. The medium band at 3450cm^{-1} might be due to the adsorption of water molecules as a result of an O-H stretching or an NH- band(Vijaya, P.P and Sandhya, S. 2003).The band at 2354.2cm $^{-1}$ is likely due to CH stretching and the band at 1680cm $^{-1}$ due to a C=O group.

XRDANALYSIS

The X-ray diffractogram for the activated carbon is shown below (fig.1). The XRD spectra illustrated, shows no specific spectrum for the presence of a crystal structure. The peaks are in clusters and are of indefinite form. It therefore implies that the activated carbon is amorphous.

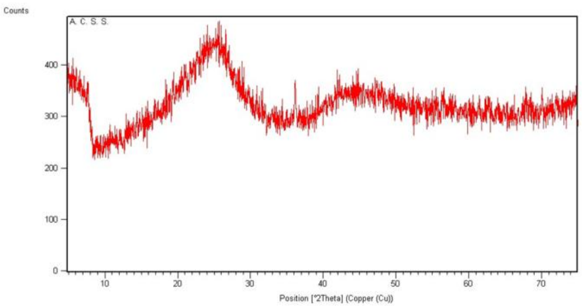

Fig. 1: X-RAY DIFFRACTION GRAPH OF ACSS

BATCH ADSORPTION STUDIES

EFFECT OF PH ON ADSORPTION CAPACITY

Adsorption of adsorbate on the surfaces of adsorbent is a function of pH, the hydrogen ion concentration affects the degree of ionization of the dye and the surface properties of the adsorbent. The effect of solution pH on the adsorption of MR onto ACS.S. is show in fig 2, the adsorption process is influenced significantly by the pH variation. The optimum absorption occurs at pH 3, that is the sorption of methyl red increases from pH 2 to 3.0, and decreases over pH 4.0 to 7.0. In this study, it was found that the bio-sorption was slightly unfavorable at pH lower than pH 3. This results in the formation of aqua complexes thereby retarding the dye due to concentration of H^+ increase. Low pH value leads to lowering of the negative charge on the surface of the surface of the charcoal, and increase in pH values leads to the hydroxyl ion concentration increases on the negatively charged surfaces of the charcoal leading to the decrease of the anionic dye adsorption. The positive hydrogen ions provide an electrostatic attraction between the activated carbon surface and the dye molecules leading to maximum adsorption (Muhammad, J.I and Muhammad, N.A 2007))

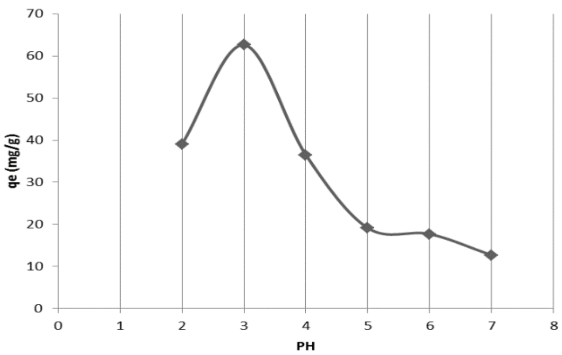

Fig.2: Effect of pH on the adsorption

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EFFECT OF TEMPERATURE

It was observed that the amount of adsorbate adsorbed per gram of adsorbent, $q_e$decreases along with an increase in temperature since the adsorption process is exothermic. This may be due to the increase in solubility of the dye at higher temperature, and decrease in adsorbtion as a result of decrease in adsorbate –adsorbent interactions.This also indicated desorption steps increase at higher temperature than the adsorption (Ilesanmi, O. etal 2013).This is due to increase in average kinetic energy of the molecules of the adsorbate in solutions containing the adsorbent which increases the number of effective collisions of the adsorbate molecules interacting with the surface of the adsorbent, thus increasing the adsorption capacities. The adsorption isotherms(Fig.3), are of L- type, implying that they have high affinity for activated carbon. The initial sharp rise in the extent of adsorption with increase in temperature shows that there is difficulty of finding vacant sites for adsorption on the adsorbent by the adsorbate molecules.

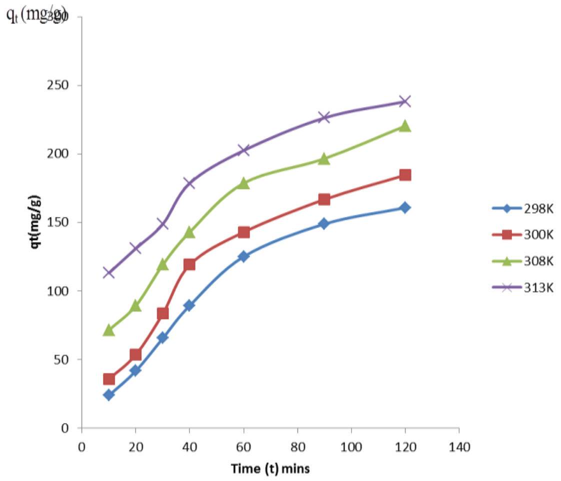

Fig.3: Adsorption isotherms at different temperatures.

CONCENTRATION DEPENDENT STUDY

The results in Fig. 4 show that an increase in initial concentration enhances the interaction between the dye molecules and the surface of the Activated carbon. The saturation point of the time profile of the dye uptake by adsorbent is a single, smooth and continuous curve. For the dye to diffuse into the porous structure of the adsorbent, it has to overcome the boundary layer effect, before diffusing from the film layer, which takes a relatively longer time. In addition, increasing the initial dye concentration increases the number of collisions between dye molecules and the adsorbent, which enhances the adsorption process.

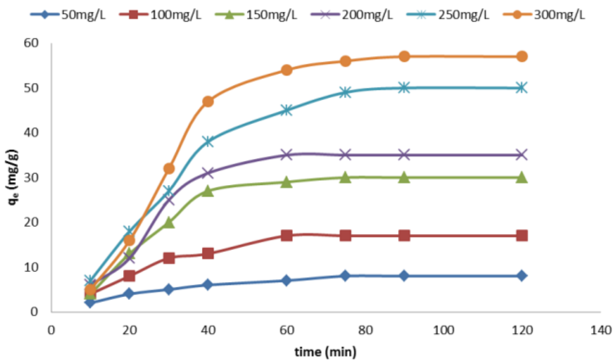

Fig.4: Effect of concentration on the adsorption

EFFECT OF CONTACT TIME

The efficiency of the bath adsorption process is affected by the contact time. The effect of contact time on the amount of MR adsorbed on the fibers was investigated at initial concentration of 200 mg L^{-1}. Fig 5 shows a rapid adsorption of

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the dye at the initial stages of the adsorption and equilibrium was attained within 45 min,Such uptake indicates a high degree of affinity towards the MR molecules via chemisorption (Weber W.J. and Morris, J.C 1963). The rate of the adsorption process increased rapidly in the first 45 min. and then grew more slowly as the agitation increases. Above the 100 min of agitation the rate of absorption becomes insignificant.

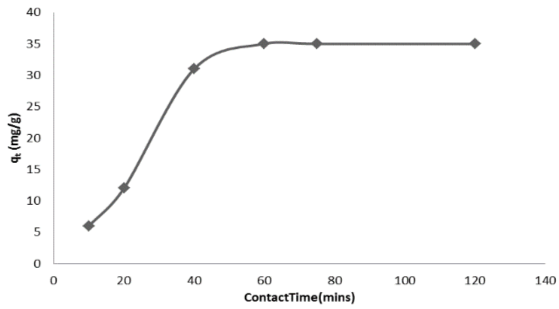

Fig.5:Effect of contact time on adsorption

ADSORPTION ISOTHERMS STUDIES

The adsorption isotherm study was carried out on two isotherm models: Langmuir which assumes a monolayer adsorption adsorbates on a homogeneous surface without any interaction between the adsorbed molecules, and model takes the linear form(Langmuir I. 1918)

$$C_e/q_e = 1/K_L q_m + C_e/q_m$$

Where qe(mgg-1) is the maximum adsorption capacity, $K_L(mg^{-1})$ bonding energy. TheFreundlich.is based on adsorption on a heterogeneous surface, can be expressed linearly as (Freundlich, HMF 1906)

$$\text{Log } q_e = \log K_f + 1/n \log C_e$$

Where K_f and n are adsorption capacity and intensity respectively.

These models describe the relationship between the dye adsorbed on the adsorbent, and its equilibrium concentration at 298, 300, 308, and 313K temperatures. The applicability of the isotherm models to the adsorption study was compared by judging the correlation coefficients R^2 values, and was used in understanding the adsorption behavior and the heterogeneity of the adsorbent surface.

Adsorption data for MR on ACSS show a linear form of Langmuir isotherm with scatter points as given in fig, 6. The R_L values for the Langmuir as given in Table 2 follows a favorable path with the range given at $0 < R_L < 1$.The correlation coefficient of Langmuir isotherm R^2 is 0.7375, shows that the adsorption mechanism does not follow the Langmuir wholly.

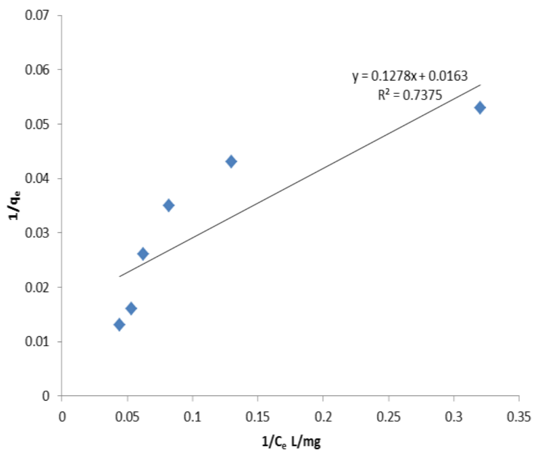

Fig. 6: Langmuir isotherm for MR sorption onto ACSS at 27°C

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Table2: theLangmuir R_L values at various initial concentrations, C_o.

$C_{o\ mg/L}$	R_L
50	0.137
100	0.074
150	0.050
200	0.038
250	0.031
300	0.026

TheFreundlich isotherm, Fig 7 gave a better fitted straight line, implying that this model is better obeyed, and a range of correlation coefficient of 0.8878. Thus, it is reasonable to conclude that the adsorption of MR on the Activated carbon consist of heterogeneous adsorption sites, in respect of adsorption phenomenon. This indicates that MR is chemisorbed on the surfaces of ACSS

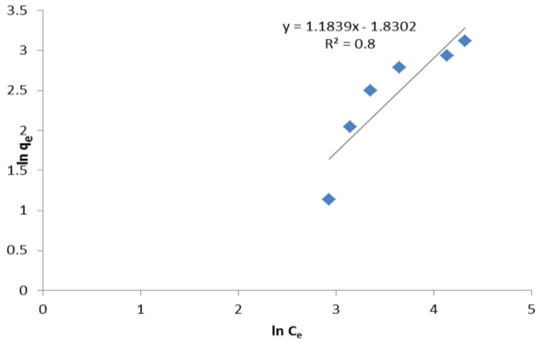

Fig. 7: Freundlich isotherm for MR sorption onto ACSS at 27°C

ADSORPTION KINETICS STUDIES

Two simplified kinetic models were adopted to examine the mechanism of the adsorption process. The pseudo – first-order equation is given as

$$\log (q_e-q_t) = \log q_e - k_1t/2.303$$

where k_1 is rate constant (min^{-1}),q_e is the amount of dye adsorbed at equilibrium (mgg^{-1}), q_t (mgg^{-1}) is the amount adsorbed at time t

The linear form of pseudo-second order rate law is represented by

$$t/q_t = 1/k_2q_e^2 + (1/q_e)t$$

where k2 is the second order rate constant (min^{-1})

Experimental results were fitted to Pseudo-First and Pseudo-second order kinetic model. The integrated and linearized Pseudo-first order kinetic model expression was given by Lagergren equation (Ho YS, and McKay G. 1998).

Fig 8 and 9 shows the fitted kinetic data on pseudo first and second order equations of MR on ACSS. The results gave a better linear plots for all the temperature as seen in fig 8, for a pseudo first order rate law, while the pseudo second order rate plot in fig 9 are not well fitted, especially at higher temperatures.

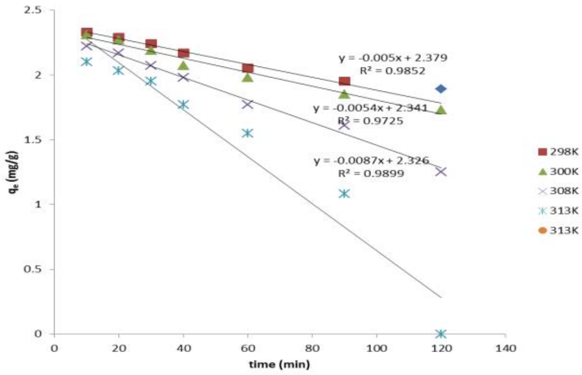

Fig.8: Pseudo first order kinetics of Methyl-red dye uptake on ACSS

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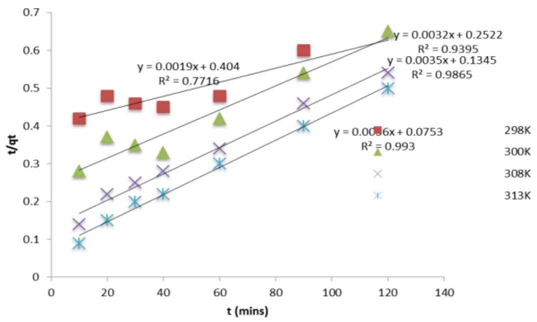

fig.9: Pseudo second order kinetics of Methyl-red dye uptake on ACSS

The correlation co-efficient R^2, the rate constants, k_1 and k_2, and the amount adsorbed at equilibrium, q_e were calculated as shown in table 3 and 4. It can be clearly seen from the results that the R^2 values are relatively high and the experimental and calculated q_e are in close agreement for the pseudo-first order kinetics(0.973 - 0.999) than the pseudo-second order kinetics (0.7716 - 0.9930), which shows that the adsorption process follows a pseudo-first order kinetics.

Table 3: pseudo first order experimental results

Temperature	Equation	R^2 value	K_1	q_e(exp.)	q_e(cal.)
298K	y=-0.005x+2.379	0.9852	0.0115	238.1	239.3
300K	y=-0.0054x+2.341	0.9725	0.0124	238.1	219.3
308K	y=-0.0081x+2.305	0.9847	0.0187	238.1	202.1
313K	y=-0.0181x+3.454	0.9359	0.0417	238.1	284.8

Table 4: pseudo second order experimental results

Temp. (K)	Equation	R^2	K_2	q_e (exp.)	q_e(cal)
298K	y=0.0019x +0.404	0.7716	0.0000089	238.1	526.32
300K	y=0.0032x +0.2522	0.9395	0.00041	238.1	312.5
308K	y=0.0035 +0.1345	0.9865	0.000091	238.1	285.71
313K	y=0.0036x +0.0753	0.993	0.00017	238.1	277.78

The Equilibrium Adsorption Constant and the Thermodynamics Parameters

The equilibrium studies at different temperature to determine thermodynamic parameters. The values for the equilibrium adsorption constant, K_c was calculated and shown in Table 5. The K_c value for the adsorption of MR dye on the Activated carbon shows a marked increase, with increase in initial concentration. The K_c values were then used to calculate the Thermodynamic parameters.

Table 5: Data values for the thermodynamic equilibruim constant, K_c at different initial concentrations.

C_o (mg/L)	C_e (mg/L)	q_e (mg/g)	K_c (ml/g)	In K_c
100	18.75	8.125	433.3	6.07
150	18.75	13.125	700.0	6.55
200	18.75	18.125	966.7	6.87
250	18.75	23.125	1233.3	7.12
300	18.75	28.125	1500.0	7.31

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The Arrhenius plot and the Van't Hoff equation were used to find the thermodynamic parameters (SalaudeenAbulwasiuOlawaleetal 2018)which include the ΔH_O, ΔS_O,ΔG_O and the activation Energy, E_a as shown in figure 10, and 11

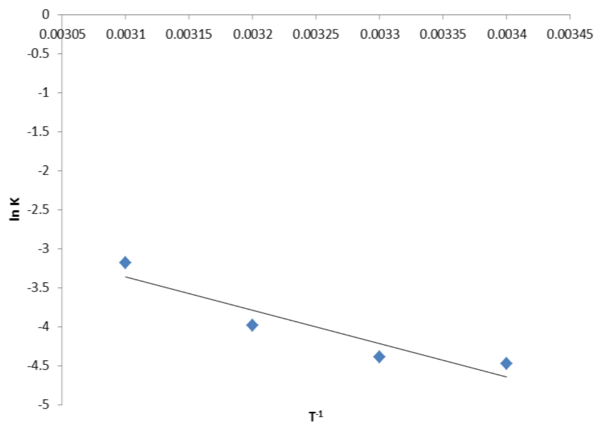

Fig. 10: The Arrhenius plot of specific rate constant at various Temperatures.

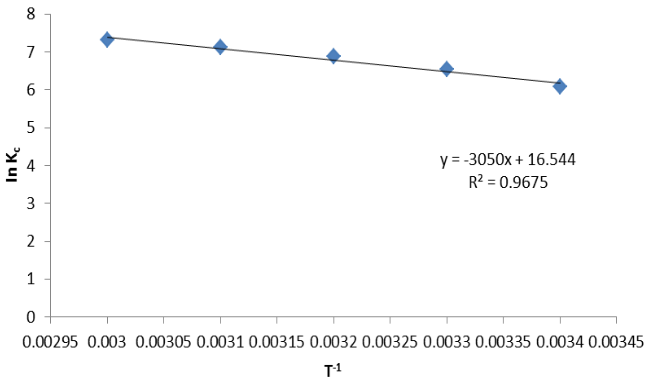

Fig.11: A plot of the Van't Hoff equation of the Equilibrium constant, ln K_c against T^{-1}

the experimental data K from the Arrhenius equation is present in Table 6 below.

Table 6: Experimental data for the Arrhenius equation plot at various temperatures.

Temp. (K)	T^{-1}	K	ln k
298	0.0034	0.0115	- 4.47
300	0.0033	0.0124	- 4.39
308	0.0032	0.0187	- 3.98
313	0.0031	0.0417	- 3.18

The Gibbs free energy, ΔG_O gave negative values which indicate a spontaneous reaction as shown in Table 7. The positive value of the ΔH_O (+30.85KJ/mol) shows an endothermic reaction and the ΔS_Oshows a positive value (155.72KJ/mol), indicating that an increase in temperature will favour the reaction process. From the Arrhenius plot, it is shown that the activation energy, E_a has a value of 35.38KJ/mol which is less than the least energy for a chemical reaction process of 40KJ/mol

Table 7: Thermodynamic parameters for the adsorption of MR on ACSS

ΔS° (J/mol K)	ΔH° (KJ/mol)	E_a (KJ/mol)	A (L/mol sec)	ΔG° (KJ/mol)			
				298K	300K	308K	313K
155.72	+30.85	35.58	2.0 x 10^4	- 15.04	- 16.34	- 17.59	- 18.53

III. CONCLUSION

In this study, the use of grounded activated carbon from Sheanut shell of the size <2mm sieve for the removal of Methyl-Red from aqueous solutions was investigated.The effect of various parameters such as pH, temperature, contact- time and dye concentration were studied.The surface morphology shows a porous, large surface area. The adsorption decreases with increase in temperature and pH of medium and the adsorption isotherms study showed a high correlation for the Freundlich model indicating that heterogeneous adsorption process took placeand the adsorption kinetics results show a high agreement between the experimental and calculated q_e and high R^2 values for the pseudo-first order rate kinetics. The positive value of ΔH implies that the process is endothermic, while the negative values of ΔG indicates that the adsorption is spontaneous

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