KINETIC AND THERMODYNAMIC MODEL FOR THE REMOVAL OF ULTRAMARINE BLUE FROM AQUEOUS SOLUTION USING MODIFIED CASSAVA PEELS

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ABSTRACT

This work presents the kinetic and thermodynamic model for the sorption of ultramarine blue on modified cassava peels. The effects of various experimental parameters such as adsorbent dosage, contact time, pH, concentration and temperature were investigated. The extent of ultramarine removal increased with increase in adsorbent dosage, contact time, concentration and temperature. The data obtained fitted to the Langmuir isotherm. The Sf value, which is the separation parameter was obtained as 0.633, indicating that the biomass is an excellent adsorbent for ultramarine blue. The Freundlich Model estimated the adsorption intensity (R^2) as 0.993. The R^2 value and the Linear plot obtained showed that the Freundlich isotherms favours the adsorption process. Pseudo-first and pseudo-second order kinetics were used to analyse experimental data and it was found that both models described the data very well. Thermodynamic parameters like free energy (ΔG), enthalpy (ΔH) and entropy (ΔS) of the system were calculated by Langmuir constant (k) and it was found that the adsorption process was endothermic in nature.

Keywords: Adsorption kinetics, dye removal, absorption isotherms and models

INTRODUCTION

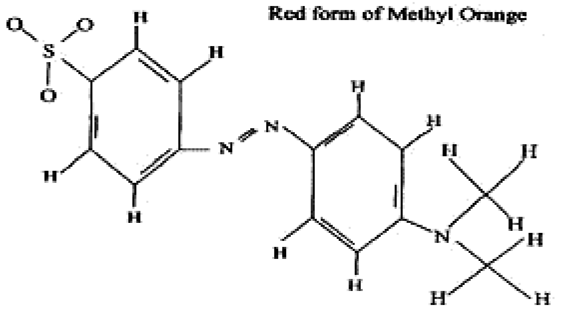
Dyes production industries and many other industries which utilize dyes and pigments are increasing globally everyday as far as technologies advancement is concern. Presently, it was estimated that about 10,000 of different commercial dyes and pigments exist and over $7x10^5$ tones are produced annually world wide (Grag *et al.,* 2004). These industries include textile, paper, plastic, food cosmetic etc, in which textile rank first and their wastes being discharged consist majorly of these dyes and pigments. Consequently, this constitute environmental hazard due to the fact that many of theses dyes and pigments are difficult to degrade. They are generally stable to light, oxidizing agents and are resistant to aerobic digestion (McKay and Sweeney, 1980). Hence, contaminations due to dyes pose not only environmental hazard, but also severe public health concern because of their persistence in nature and non biodegradable characteristics.

However, the removal of dyes from the wastes discharged by the aforementioned industries, especially textile industries now call for urgent attention in order to safeguard our environment and public health. In this regard, a number of conventional methods are available for the removal of dyes from industrial effluents, including ion exchange, adsorption, membrane technology and coagulation (Nigam, 2000). But in all, adsorption, which is a process that occurs when a gas or liquid solute accumulate on the surface of a solid or a liquid (adsorbent), forming a film of molecules or atoms (the adsorbate), has been one of the effective methods adopted so far. The process of adoption has an edge over the other methods due to its sludge free clean operation and completely removed dyes, even from the diluted solution.

Meanwhile, in adopting adsorption method in the removal of dyes, activated carbon (powdered or granular) has been one of the most widely used adsorbents. This is due to the fact that it has excellent adsorption efficiency for organic compound, through its use is limited in industries of developing countries due to its high cost. Several studies have shown that numerous low cost materials have been successfully applied in the removal of dyes from aqueous solution, some of which are coal, fly ash, wood silica, agricultural waste etc. however, only few of them could be employed effectively to remove dyes from the waste stream.

In this study, the potential of modified cassava peel in the removal of ultramarine blue dye from aqueous solution has been investigated. It is a low cost agro-industrial waste that is common in the southern part of Nigeria. Experimental parameters affecting the adsorption process such as pH, concentration, adsorbent dosage, contact time and temperature were studied. The equilibrium adsorption data were fitted in Langmuir and Freundlich isotherm, pseudo-first order and pseudo-second order were considered. Thermodynamic parameters were also estimated in order to study the nature of the system.

The Structure of Ultramarine Blue



Materials and Methods

Sample Collection and Preparation of the Adsorbent

About 1.5kg of cassava (*manihot esculenta cranz*) peels were collected from a cassava mill in Abraka in Ethiope East Local government Area of Delta State. These were extensively washed with tap water to remove soil and dust, sprayed with distilled water and then sun dried for five days. Then the dried cassava peels were burnt in the absence of air in order to get the charcoal. The charcoal obtained was further ground and sieved using 450um sieve. The sieved adsorbent obtained was preserved in a plastic container for further studies.

Preparation of Dye Solution

The dye (ultramarine blue) used in this study was obtained from a commercial market without further purification. The dye stock solution was prepared by dissolving accurately weighed dye in distilled water to the required concentration for each of the experimental parameters been considered.

MATERIALS AND METHODS

(a) *Effect of Contact Time on Adsorption*

The experiment on the effect of contact time on the adsorption of the dye ion by the cassava peel adsorbent was performed according to the previous works of Sumajit *et al* (2007). 2 g of the modified adsorbents was weighed into five different conical flasks. Concentration of 10 mg/L of the dye was prepared using distilled water and 50mL of the dye solution was measured into the five flasks. The flasks were then labeled for time intervals of 20, 40, 50, 60, 80 and 100 min. The flasks were tightly covered and agitated at the appropriate time intervals. At the end of each time intervals, the suspensions were filtered using Whatman No. 45 filter paper and then centrifuged for 30 min at 180 per min. The dye ion concentration was determined using DR 2010 spectrophotometer.

(b) *Effect of Adsorbent Dosage on Adsorption*

The experiment on the effect of adsorbent dosage on the adsorption of dye ion by the cassava peel adsorbent was performed according to the previous works of Sumanjit *et al.,* (2007). 2, 3,4, 5 and 6 g of the modified adsorbent were weighed into five different conical flasks. 50mL of the dye solution was measured into the five flasks. The flasks were then labeled for dosage differences of 2, 3, 4, 5 and 6g. The flasks were tightly covered and agitated for 20 minutes, and thereafter the suspensions were filtered using Whatman No. 45 filter paper, then centrifuged. The dye ion concentration was determined using DR 2010 spectrophotometer

(c) *Effect of Dye Ion Concentration on Adsorption*

The experiment on the effect of dye ion concentration on adsorption was performed according to the previous works of Sumanjit *et al.* (2007). Several standard dye solutions of 10, 20, 30, 40 and 50 mg/L were prepared. 50mL of each of the dye solution was added to accurately weighed 2±0.01 g modified adsorbent in five different flasks and agitated for 20 minutes. At the end of the time, the suspension was filtered using Whatman No. 45 filter paper and centrifuge. The dyes ion concentration was determined using Dr 2010 spectrophotometer (British Equipments)

(d) *Effect of Temperature on Adsorption*

The experiment on the effect of temperature on adsorption was performed according to the previous work of Mishra *et al.* (2009). 2g of the modified adsorbent was weighed into five different conical flasks and 50mL of the dye solution (l0 mg/L) was measured into the five flasks. The flasks were labeled for temperature differences of 30, 40, 50, 60 and 70°C. The flasks were tightly covered and heated at the appropriate temperature using thermostatic water bath, at 20 mins each. At the end of the time, each of the flasks were brought out and agitated for about 5 min. Then the suspensions were filtered using Whatman No. 45 filter paper and centrifuged. The dye ion concentration was determined using DR 2010 spectrophotometer.

(e) *Effect of pH on Adsorption*

The experiment on the effect of pH on adsorption was carried out based on the previous works of Sumanjit *et al.,* (2007). Modified adsorbent (2± 0.0 lg) was weighed into five different flasks. 50mL of the dye solution (l0mg/L) was measured and added into the five flasks. The solutions were adjusted to pH 2.0, 4.0, 6.0, 8.0, and 10.0 by adding a solution of HCl (0.1M) or NaOH (0.1M) and the pH readings were confirmed by the use of pH (21R English model) meters. The flasks were then tightly covered and agitated for 20min. At the end, the suspensions were filtered using Whatman No. 45 filter paper and centrifuged. The dye contents were determined using DR 2010 spectrophotometer.

Data Evaluation

(a) *Calculation of the Degree of Dye Removal*

The amounts of dye removal by the adsorbent during the series of the batch experiments were determined using a mass balance equation expressed as shown below.

$$Qe = (Co - Ce)\ \frac{V}{M}$$

Where qe – dye concentration on the biomass (mg/g) at equilibrium
Ce = dye concentration in solution (mg/L) at equilibrium
Co = initial dye concentration in solution (mg/L)
V = volume of dye solution used (mL)
M = mass of adsorbent used (g)

(b) *Kinetic Treatment of Experimental Data*

In order to comprehensively investigate the mechanism of adsorption, the equations of pseudo-first order and pseudo-second order mechanisms are applied to experimental data.

The linear form of pseudo-first-order model which has been described previously is given as: -

$$In(qe-qt) = lnqe-kt$$

Where
qe = mass of dye adsorbed at equilibrium (mg/g)
qt = mass of dye adsorbed at time t (mg/g)
k = equilibrium constant

The linear plot of ln(qeqt) versus t confirms the model. The linear form of pseudo-second-order model which has been also descried previously is given as:-

$$\frac{I}{qt} = \frac{I}{ho} + \frac{It}{qe}$$

Where
qt = amount of dye ions on the adsorbent surface (mg/g) at anytime t.
qe = the amount of dye ions adsorbed at equilibrium (mg/g)
ho = the initial adsorption capacity (mg/g min).

The initial adsorption rate, ho is defined as:

$$ho = K_2qe^2$$

Where K_2 is the pseudo-second order rate constant (g/mg min).

RESULTS AND DISCUSSION

(a) *Effects of Contact Time on Dye Removal*

The effect of stay time for the adsorption of the dye was studied between the intervals of 20 min and was varied from 20 to 100 min. Figure 1 illustrate the adsorption of dye at different time duration. As the contact time was increased from 20 to 100 mins, the amount of dye ions increased from 0.l05 mg/g to 0.245 mg/g.

This may be due to the fact that as the dye solution-adsorbent, system is being agitated at longer time, more of the atoms of the dye tend to accumulate on the surface of the adsorbate until equilibrium is reached.
However, similar trends have been observed by some other researchers (Hajira *et al.,* 2008; Mishra *et al.,* 2009; Sumanjit *et al.,* 2007).

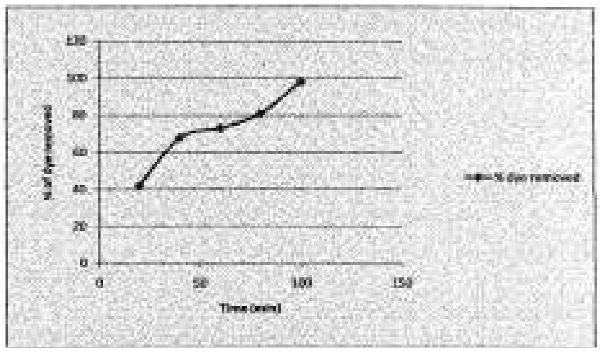


Figure. I: *Effect of contact time on the adsorption of the dye by cassava peels absorbent.*

(a) *Effect of Adsorbent Dosage on Dye Removal*

The effect of amount of adsorbent (cassava peel) on the adsorption of ultramarine blue system was studied, in which the amount of adsorbent was varied from 2 to 6 g. Figure II show the effect of adsorbent dosage on the removal of the dye in which the amount of dye removed increased from 0.095-0.228 mg/g as the adsorbent dosage was increased from 2-6 g. From Figure II, it is observed that the optimized adsorbent dose is 6 g in which 0.228 mg/g of dye was adsorbed. Moreso, the maximum percentage (91%) of dye adsorbed is obtained at this dosage (6 g) was also shown in Figure II.

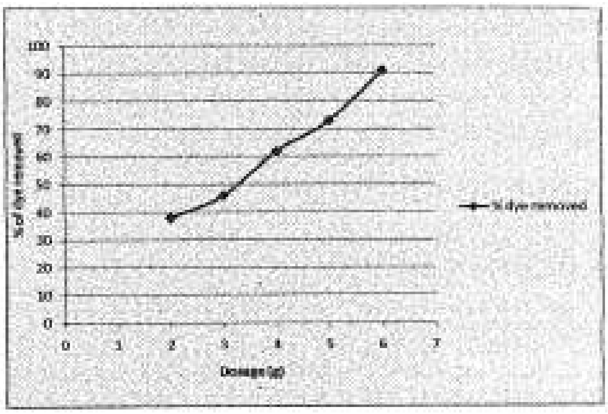


Figure II: *Effect of adsorbent dosage on the adsorption of UB.*

However, the increase in dye uptake could be attributed to increase in the binding sites for adsorption. Meanwhile similar behavior has been reported by other workers (Alok, 2006; Sumanjit *et al.,* 2007; Yamin *et al.,* 2007).

(b) *Effect of Concentration on dye Removal*

The experimental results of the removal of ultramarine blue dye by cassava peel at various initial dye ion concentrations are as shown in figure III. The adsorption capacity increase from 0.185 to 1.027 mg/g with an increase in the concentration of dye from 10 to 50 mg/L, having maximum adsorption of 1.027 mg at 50mg/L

However, the actual percent removal of the dye was found to increase with increase in initial dye concentration fig III. This may be due to the fact that as the dye concentration is increasing, more dye is available for adsorption on the absorbent. This is due to the effect of concentration gradient which is the main driving force for the adsorption process, Mishra *et al.* (2009). Similarly, this trend has been observed by other workers (Hajira *et al.,* 2008) Mishra *et al.,* 2009).

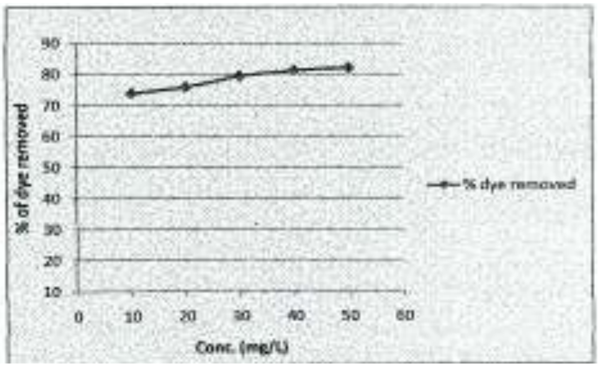


Figure. III: *Effect of concentration on dye removal.*

(c) *Effect of Temperature on Dye Removal*

The dependence of dye adsorption on temperature was studied within the range of temperatures 30-70°C at the interval of 10^0C. The effect of temperature on the adsorption of dye is shown in Figure IV, in which the amount of dye adsorbed increased from 0.115 to 0.123 mg/g with increases in temperature from 30 to 70°C.

Though, the increase in the amount of dye removed as the temperature increases is not much significant, according to Hiroyuki *et al.* (1994), the higher removal due to increasing temperature may be attributed to chemical reaction taking place between the functional groups of the adsorbate/adsorbent and the dye. Moreso, at high temperature, there would be increase in the mobility of the larger? dye ion and also producing swelling effect within the internal structure of the adsorbent, thus enabling the large dye molecule to penetrate further (Mckay, 1982).

Despite the aforementioned reasons accompanying increased adsorption with increased temperature, it has been reported, according to Panday *et al.* (1968), increase adsorption at higher temperature is difficult to explain. However, the trend of increase adsorption with increased temperature has been reported by other workers (Gurusamy, 2002; Hajita *et al.,* 2008).

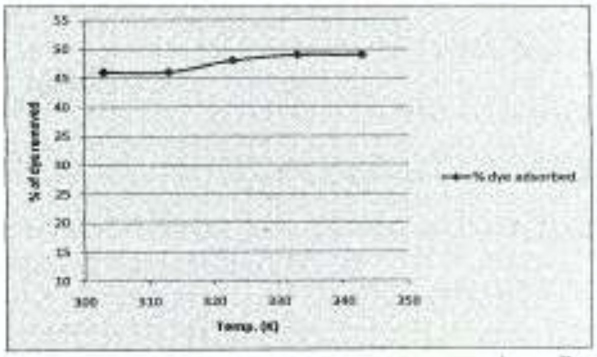


Figure. IV: *Effect of temperature on dye removal.*

(d) *Effect of pH on Dye Removal*

The pH of an aqueous medium is an important factor that may affect the uptake of the adsorbate. The chemical characteristics of both adsorbate and adsorbent vary with pH. According to Horsfall and Spiff, (2005d), most plant materials are made up of complex organic residues such as lignin and cellulose that contain, several type of polar functional groups. These groups can be involved in chemical bonding and may be responsible for the typical cation-exchange characteristics of most biomaterials.

The pH dependence data for adsorption of the dye being investigated are presented in figure V. It is observed that as the pH of dye solution increased from 2-8, the amount of dye adsorbed decreased from 0.135 to 0.087 mg/g, though at pH 10 there was a rapid increase in the amount of dye adsorbed. The initial decrease in the amount of dye adsorbed as the pH increases may be due to the fact that ultramarine blue exists in anionic form at basic pH and in cationic form at acidic pH.

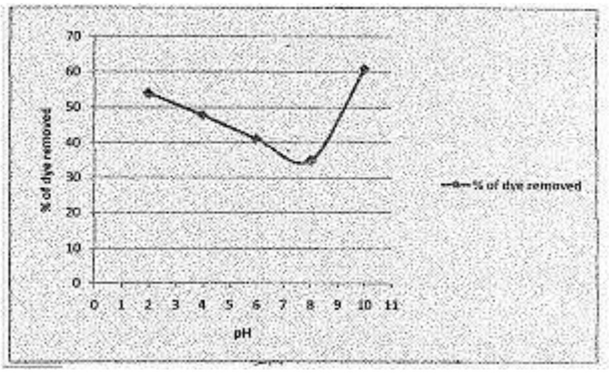


***Figure V:** Effect of pH on dye removal.*

However, according to Yamin *et al.* (2007), in the study of removal of methylene blue by treated activated carbon, in which removal of methylene blue increased with the increased pH. However, adsorption of methylene blue at low pH is probably due to the presence of hydrogen ions, competing with the cations groups on the dye for adsorption sites. So on the contrary, the initial higher adsorption of ultramarine blue at low pH may be probably due to the absence of hydrogen ions which cannot complete with the cations group on the dye for adsorption. Meanwhile similar trend has been reported in the literature for the adsorption of Congo red dye onto Mahua oil cake (Mishra. et al, 2007).

Data Evaluation

(a) *Langmuir Isotherm*

The Langmuir isotherm model was chosen for the estimation of maximum adsorption capacity corresponding to complete monolayer coverage on the biomass surface. The plots of specific adsorption (ce/qe) against the equilibrium concentration (ce) are shown in figure VI and the linear isotherm parameters. q_m, k_L and the coefficient of determinations are presented in Table 1.

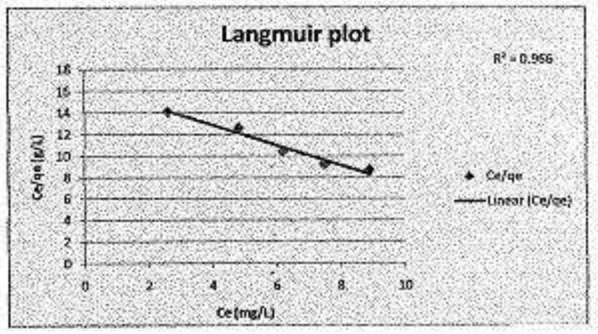


***Figure. VI:** Langmuir isotherm for dye removal.*

The R^2 value suggested that the Langmuir isotherm provides a good model of the adsorption. The favourability of adsorption of the dyes ions on the cassava peels waste biomass was tested using the essential features of the Langmuir isotherm model, expressed in terms of a dimensionless constant called separation factor, S_F proposed by Putshaka *et al.* (2005). The separation factor S_F is defined by the following relationship.

$$S_F = \frac{1}{I+K_LCo}$$

Where K_L = Langmuir isotherm constant Co = initial dye ion concentration of l0 mg/L. The parameter indicates the shape of the isotherm as follows:

$S_F > 1$ unfavourable isotherm
$S_F = 1$ linear isotherm
$S_F = 0$ irreversible isotherm
$0<S_F<1$ favourable isotherm

The separation parameters for the dye is less than unity indicating that cassava peel waste biomass is an excellent adsorbent for ultramarine blue ions. The separation parameter and other Langmuir isotherm parameters are shown in table I below.

Table I: Linear Langmuir Isotherm Parameters

Dye ion	qm (mg^{-1})	K_L	R^2	S_F
U.B	0.952	0.058	0.966	0.633

(b) *Freundlich Isotherm*

The Freundlich model was chosen to estimate the adsorption intensity of the adsorbate on the adsorbent surface. The linear Freundlich isotherms for the adsorption of the dye ion onto cassava peel waste biomass are presented in figure VIII. Examination of the plot reveals that the Freundlich isotherm is also an appropriate model for the adsorption process under consideration. Table II shows the linear Freundlich sorption isotherm constant and the coefficient of determination (R^2).

Based on the value of R^2, the linear plot form of the Freundlich isotherm appears to produce a reasonable model for the adsorption process under consideration.

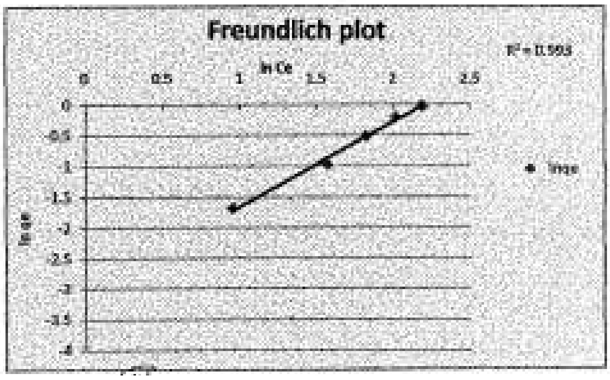


Figure VII: Freundlich Equilibrium Isotherm Model for Dye Removal.

Table II: Freundlich isotherm Constant

Dye in	1/n	K_F	R^2
UB	1.978	0.045	0.993

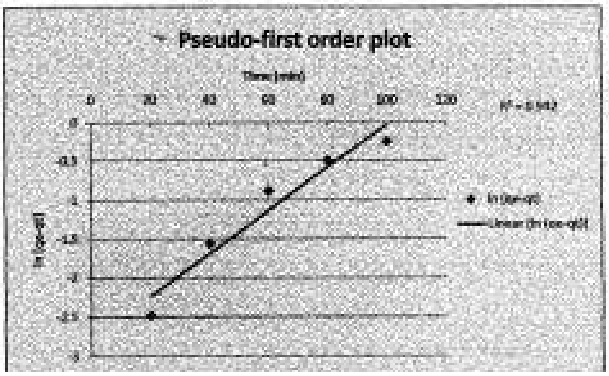
(c) *Adsorption Kinetics*

I. Pseudo – First Order Plot

The kinetic of adsorption is probably the most important factor in predicting the rate at which adsorption takes place for a given system.

A plot of In(qe-qt) against t (Figure VIII) gave the pseudo- first order kinetics. From the plot, it is observed that the relationship between dye ion diffusivity, In(qe-qt) and time is linear which confirm the model. The value of coefficient of determination R^2 is shown below which indicates that pseudo-first order model provide a good description for the adsorption of ultramarine blue on the cassava peel biomass.

Figure VIII: *Pseudo - first order plot.*



However in confirming the linearity of the pseudo-first order model, the same observation has been reported by Gunusamy (2002) for the adsorption of basic dye on strongly chelating polymer.

II. Pseudo - Second Order Model

A plot of t/qt against t (Figure IX) gave the pseudo-second order kinetics. From the plot, it is observed that the relationship between t/qt and t is linear which confirms the model. Also, the initial sorption rate ho, the equilibrium adsorption capacity qe, the pseudo-second order rate constant K_2 and the coefficient of determination R^2 are presented in Table III. Based on the value of coefficient of determination R^2, the pseudo-second order model provides a better description for the adsorption process batter than pseudo-first order model. This observation has been reported by Che (2004) for the adsorption of basic blue, acid blue and direct red dyes using clay-based and activated carbon adsorbent.

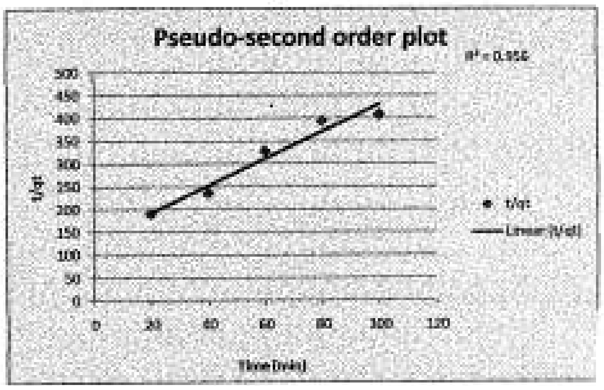


Figure IX: *Pseudo - second order plot.*

Table III: Values of pseudo-second order kinetic paramet.

Dye ion	Ho mgg^{-1}	K_2 mgg^{-1}	qe mgg^{-1}	R^2
U.B	0.008	0.072	0.333	0.956

(d) *Thermodynamic Parameters*

The values of thermodynamic parameters like free energy (Δ°G), enthalpy (Δ^0H) and entropy (Δ°S) of the adsorption process were calculated from the Langmuir constant K using the following equations.

$$\Delta G^o = -RT\ \text{In}k_D$$

$$\Delta \text{In}k = \frac{\Delta S}{R} - \frac{\Delta H}{RT}$$

$$\Delta G^o = \Delta H - T\Delta S.$$

The value of ΔH° and ΔS° were calculated from the slope and intercept of the linear variation of ink with the reciprocal of temperature (1/T) and are given in figure X.

From the table III, the values of ΔG° at different temperatures are positive, which shows that the adsorption process is unspontaneous. The values of enthalpy are also positive, which reveals the endothermic nature of the process, the values of entropy are negative. Meanwhile, the same observation has been reported in regards to the endothermic nature of the process by Hajira et al (2008) for the removal of basic dye methylene blue by using bioadsorbents.

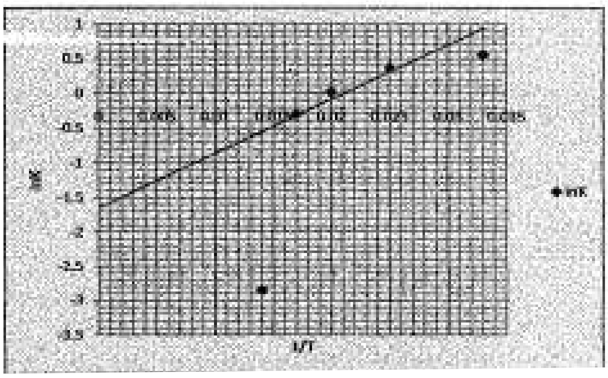


Figure X: *Thermodynamic Plot.*

CONCLUSION

The effects of ultramarine blue ion concentration on adsorption capacities show that cassava (*mannihot esculanta cranz*) peel waste biomass adsorbed the dye ions from solution, with an increase in adsorption capacity of the biomass with increased dye ion concentration. The actual percentage removal of the dye ions from solution also increase with increase initial dye ion concentration. On the effect of adsorbent dosage on the adsorption of ultramarine blue by the cassava peel waste biomass there is also adsorbed and also increase in the actual percent removal. The effect of contact time and temperature showed the same trend in the increase on the amount of dye ion adsorbed and the actual percent removal. The pH profile studies showed a decreased in the amount of the dye ion adsorbed when acidic but a sudden increase at pH of 10. However, the equilibrium data fitted the Langmuir and Freundlich isotherms very well, and the separation factor or equilibrium parameter obtained from the Langmuir isotherm showed that adsorption of the dye ions onto the cassava peel waste biomass is favourable.

The kinetic data clearly established that pseudo-second order model provide a more appropriate description of the dye ions adsorption process ultramarine blue. Moreso, the thermodynamic parameter showed that the adsorption process was endothermic. Though not spontaneous, on the whole, the data showed that cassava peel waste biomass was successful as adsorbent for removing dyes from wastewater and may serve as an alternative adsorbent to conventional means. Hence *manihot esculanta cranz* peel waste is not only inexpensive and readily available, but it also has the potential for dye removal from wastewaters or contaminated waters.

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