

## Thermodynamic Study of Adsorption of Azure Dyes on Iraqi Porcelanite Rocks

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### Abstract

Removal of Azure dyes, A, B, and C have been carried out by using Iraqi Porcelanite rocks as an adsorbent . A series of experiments were undertaken in a batch adsorption technique to access the effect of the process variable i.e. contact time , initial pH , adsorbent dose and temperature. The removal data have been analyzed using Langmuir and Freundlich models of adsorption at various temperature varying from 298 to 328 K .Thermodynamic parameters such as change in free energy ( $\Delta G$ ) the enthalpy ( $\Delta H$ ) and the entropy ( $\Delta S$ ) were also evaluated. The overall adsorption process of Azure A, B, and C dyes onto porcelanite rocks were spontaneouse, exothermic, and physisorption.

**Keyword:** Porcelanite Rocks, Organic dyes Azure A, B and C, Adsorption isotherm.

### (I) Introduction

Dyes have long been used in dyeing, paper, pulp, textiles, plastics, leather, paint, cosmetics and food industries[1,2]. Nowadays, more than 100.000 commercial dyes are available with a total production of 700.000 tones manufactured all over the world annually. About 10-15% of dyes are being disposed off as a waste into the environment after the completion of dyeing process[2]. Dyes also affect human and animal health[3].In addition dyes can cause allergic dermatitis, skin irritation , cancer and mutations[2]. Hence treating waste water with dyes is one of a prime importance, there are several methods for dye removals such as adsorption, oxidation-ozonation, coagulation, coagulation-flocculation and biological methods[4-7]. .Adsorption process provides an attractive treatment of waste water containing dyes over other treatment techniques due to economic consideration, its availability and easy to operate as well as greater efficiency,[4,8]. The Iraqi porcelanite rocks represent one of the most and great adsorbents, because it is containing high percentage of silicon reached to 50% [9,10]. However , our search through the literature reveals that limit works have been done on the adsorption of organic dyes by using Iraqi porcelanite rocks as adsorbent.

### (II) Experiments

#### 1.Materials used :

Porcelanite rocks in Iraq are from an industrial bed of (0.5 to 1.3m) thickness in the sefra, and Trafawi site of the Jeed formation in Al-Rutba region, western of Iraq .The adsorption capacity of porcelanite is due to the large surface area within the composition of critobalite and tridymite [11,12]. Some of the chemical and physical analysis for the porcelanite is shown in Table (1).

**Table (1) chemical and physical analysis for the porcelanite samples of Traifawi Site**

Chemical composition	SiO <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	CaO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O	L.O.I
%	83.57	0.62	4.45	0.01	1.82	1.64	0.5	0.16	0.22	5.9
Specific gravity (SG)	Range for 5 Sample				Average			Recommendation		
Porosity	1.5-1.61				1.554			OK		
					0.52					

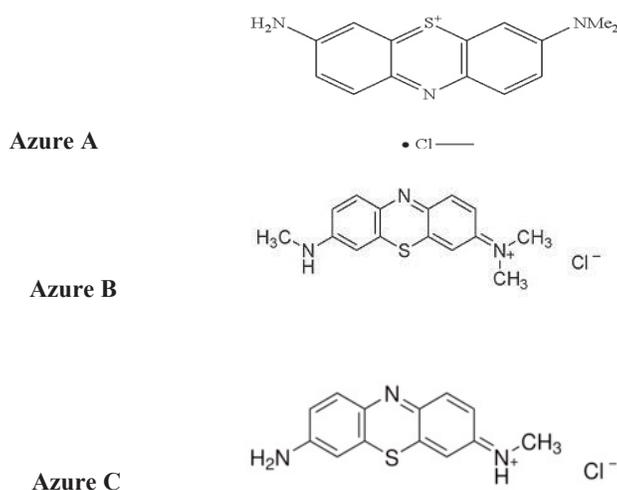
The crushed and milled samples of porcelanite was washed several times with deionised water then dried to constant weight at (110 C) by using drying oven. Each sample was sieved and the fraction of 75 µm and below was collected for adsorption experiments.

Organic Azure A, B, and C dyes, were used. All the chemicals were of high purity, commercially available AR grade. The properties of the selected dyes are given in Table (2) and their structures are shown in Fig. (1).

**Table (2): Details of selected dyes**

Dyes	Molecular formula	Molecular weight(g/mol)	Wavelength $\lambda_{max}$ (nm)	C. I. No.
Azure A	C <sub>14</sub> H <sub>14</sub> ClN <sub>3</sub> S	291.7991	633	52005
Azure B	C <sub>15</sub> H <sub>16</sub> ClN <sub>3</sub> S	305.83	648	52010
Azure C	C <sub>13</sub> H <sub>14</sub> ClN <sub>3</sub> S	277.77	616	52002

**Fig. (1): Molecular structures of organic dyes**



## 2.Batch Adsorption Experiments:

The adsorption experiments were carried out by agitating(0.025 mg) adsorbent with (25 ml) of dye solutions (50)mg/L at (150) rpm on an thermostated shaker water bath. centrifuge (universal make) at 5000 rpm for (60) min and unabsorbed supernatant liquid was analysed for the residual dye concentration using Shimadzu UV-Vis 1700 digital double beam at a wavelength corresponding to the  $\lambda_{max}$  of each dye. The effect of pH was studied by the adjusting of pH( 7 ) of the adsorptive solution using dilute HCl and NaOH solutions. The effect of temperature was studied using five different temperatures ( 298 – 328 K ).

All experiments were carried out in duplicate and the mean values are reported. The effect of each parameter (contact time, PH value, temperature and adsorbent dosage) were evaluated in an experiment by varying the parameter, while keeping the other parameters as constant. The amount of dye on porcelanite adsorbent was calculated using the following equation.

$$Q_e = (C_0 - C_e)V/m \dots(1)$$

Where  $Q_e$  (mg/g) is the amount of dye adsorbed at equilibrium (adsorbent capacity),  $C_0$  and  $C_e$  are (mg/L) the concentrations of dye at initial and equilibrium respectively,  $V$  and  $m$  is the volume of the solution (L) and the adsorbent mass (g) respectively.

### (III) Results and Discussion

#### Characterization Studies:

The XRD spectrum (Fig.2) indicates existence of certain amount of Quartz (silicon dioxide) and Opal; which are known as irregular interferences between two phases, kretoplyte and traidamayte-alpha; and also some other (kaolin and samkrite) which can be found together with fine size silica.

The surface morphology of porcelanite rocks was visualized via scanning electron microscope (SEM) at 10000 magnification. Examination of SEM micrographs of the porcelanite rocks particles showing clearly the porous nature of the sample surface (Fig.3)

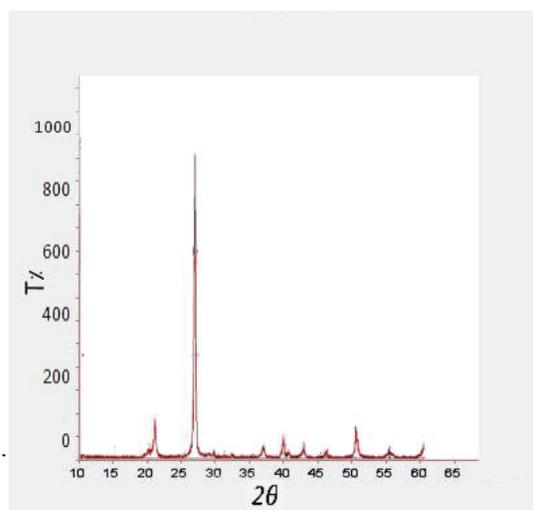


Fig.(2): XRD of Iraqi Porcelanite rocks

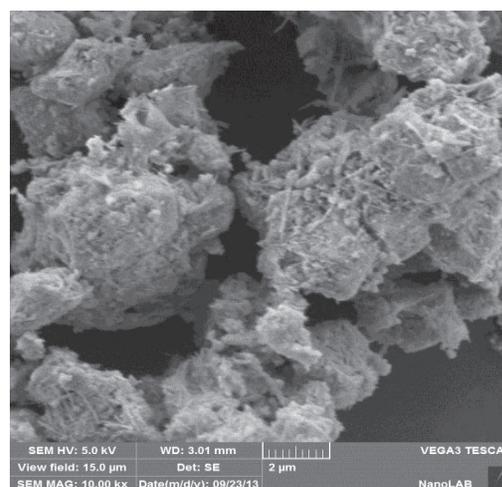


Fig.(3): The SEM photograph of Porcelanite rocks

The FT-IR spectra (Fig.4) shows existence of the terminal silanol-OH and to the bright Si-OH-Si.

The band at  $(1600 \text{ cm}^{-1})$  could be attributed to the deformation of water molecules  $\beta(\text{H}_2\text{O})$ . The band at  $(1100 \text{ cm}^{-1})$  that appear as strong band can be assigned to the symmetric ( $\nu$ Si-O-Si) or asymmetric ( $\nu$ Si-O-Si) stretching vibrations. The bands at  $(796 \text{ cm}^{-1})$  are characteristic of Quartz and those at  $(455 \text{ cm}^{-1})$  can be attributed to  $\beta(\text{Si-O-Si})$  or  $\beta(\text{Si-O-Si})$  bending vibrations [13].

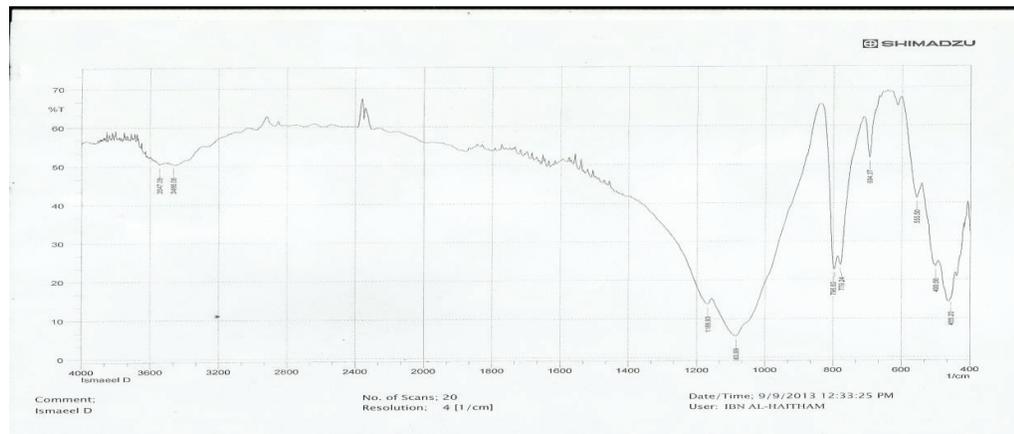


Fig.(5): FT-IR spectrum of Iraqi Porcelanite rocks

### Effect of Contact Time

The relationship between contact time and adsorption capacity of Azure a, b and c dyes is conducted through batch experiments to achieve the equilibrium as shown in (Fig.5). The results showed that the equilibrium time was reached within 60 mins.

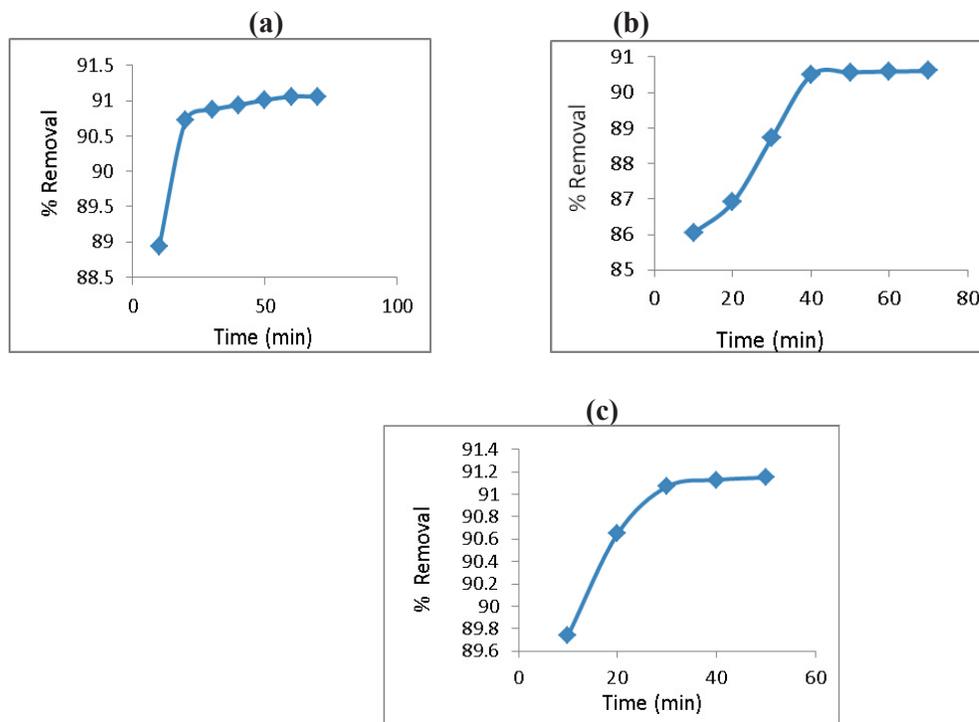
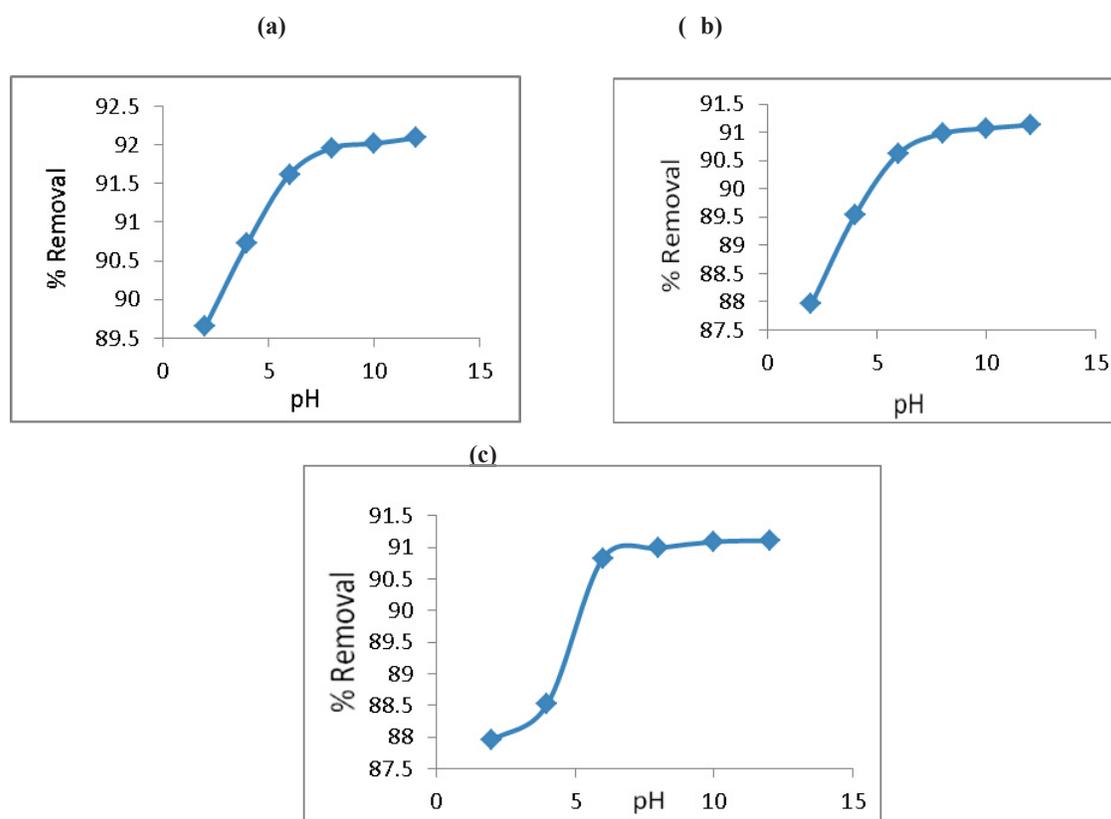


Fig.(5) Effect of contact time on adsorption of: (a) Azure A (b) Azure B (c) Azure C by Iraqi Porcelanite rocks. Temperature= 298, pH= 7, agitation speed (5000 rpm) and adsorbent Dosage = 0.025 g.

### Effect of pH

The pH is the important factor which controls the adsorption process especially for Azure dyes[14 ]. Fig.(6) shows the effect of pH for the adsorption of Azure A, Azure B and Azure C on to Iraqi Porcelanite rocks over a pH range of 2-12. As elucidated in (Fig. 6), the dyes removal were minimum at pH 2 and the dyes adsorbed increased as the pH was increased from 2 to 7. Then, beyond pH 7 there was no notable change. For this reason, pH 7 was selected for further experiments. After adsorption experiments, it was found that at low pH and at high pH, the dye become protonated, the electrostatic repulsion between the protonated dyes and positively charged adsorbent sites result in decreased adsorption. Higher adsorption at pH 7 may be due to increased protonation by the neutralization of the negative charges at the surface of the adsorbent, which facilitates the diffusion process and provides more active sites for the adsorbent.

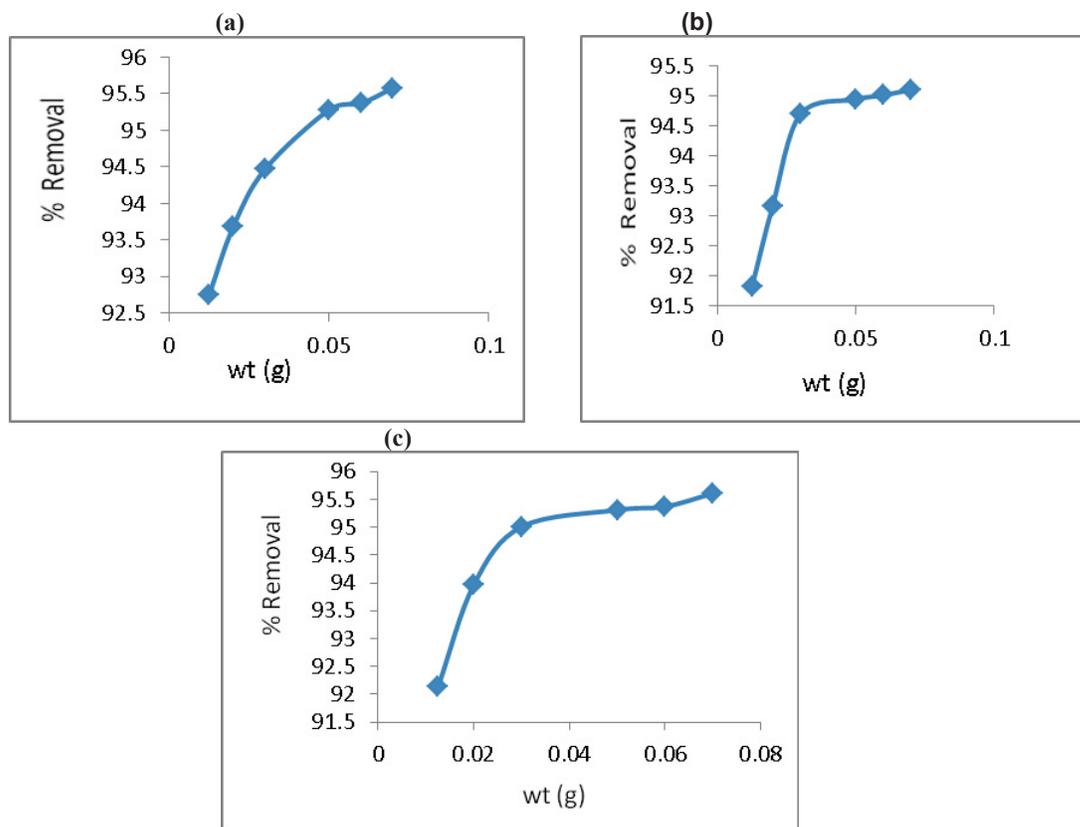


**Fig.(6)** Effect of pH on the adsorption of: (a) Azure A (b) Azure B (c) Azure C by Iraqi Porcelanite rocks. Temperature= 298, agitation speed (5000 rpm) and equilibrium time = 60 min.

### Effect of Adsorbent Dos

Effect of adsorbent dose on removal dye is studied by varying the dose of adsorbent (0.0125, 0.02, 0.03, 0.05, 0.06 and 0.07 g) in the test solution while keeping the initial dye concentration  $50 \text{ mg L}^{-1}$  (Temperature  $25 \pm 1$ ) at pH 7. Experiments were carried out at different contact times for 70 mins. As shown in (Fig. 7), the percent of the adsorption increased with increasing adsorbent doses. The increase in the percent removal of dyes with the increase in adsorbents dosage is due to the availability of larger surface area with more active functional groups[15]. Initially the rate of increase in the percent dye removal has been found to be rapid which slowed down as the dose increased. This phenomenon can be explained, based on the fact that at lower adsorbent dose the adsorbate (dye) is more easily accessible and because of this, removal per unit weight of adsorbent is higher. With rise in adsorbent dose, there is less commensurate increase in adsorption,

resulting from many sites remaining unsaturated during the adsorption.[15]. The result obtained indicate that the Porcelanite rocks has a large potential as an adsorbent for dye removal.



**Fig.(7)** Effect of adsorbent dosage on the percentage removal of: (a)Azure A (b)Azure B (c)

Azure C by Iraqi Porcelanite rocks .Temperature = 298 K, initial dye concentration= 50 mg/L, pH= 7, contact time (60 min) and agitation speed (5000 rpm).

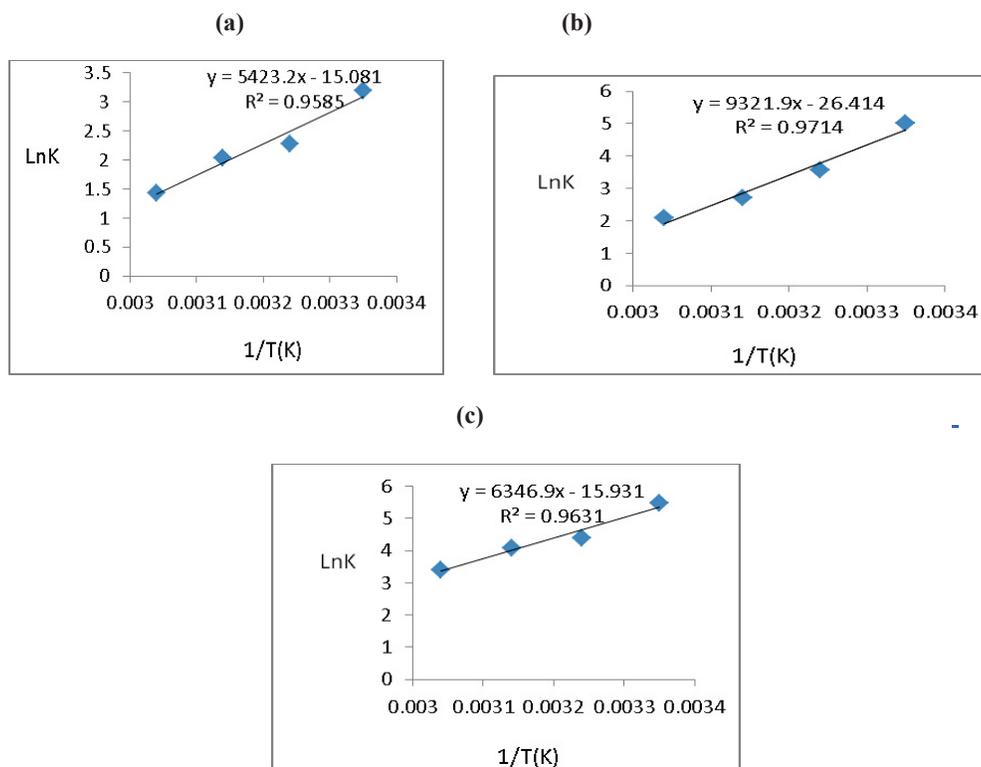
### Effect of Temperature

The removal of Azure A, B, and C dyes using Iraqi Porcelanite rocks has been studied at 298 to 328 K. Determine the adsorption isotherms and thermodynamic parameters, which is presented in (Fig.8) Thermodynamic parameters, i.e. free energy ( $\Delta G$ ), enthalpy ( $\Delta H$ ) and entropy ( $\Delta S$ ) changes were also calculated using eqs. (2-4)[16] and are given in Table ( 3 )

$$\Delta G = -RT \ln K \dots \dots \dots (2)$$

$$\ln K = \left( -\frac{\Delta H}{RT} \right) + \text{const.} \dots \dots \dots (3)$$

$$\Delta S = \frac{\Delta H - \Delta G}{T} \dots \dots \dots (4)$$



**Fig.(8)** Temperature dependence of the adsorption (a)Azure A (b)Azure B (c)Azure C on the Iraqi Porcelanite rocks.

**Table(3)** Thermodynamic function  $\Delta G, \Delta S$  and  $\Delta H$  of Azure A, B and C dyes on the adsorbent surface Iraqi Porcelanite at (298-328)K

298 K			
Adsorb.	$\Delta G$ (KJ/Mol)	$\Delta H$ (KJ/Mol)	$\Delta S$ (KJ/Mol)
Azure A	-7.9252	-45.0868	0.1247
Azure B	-12.3976	-77.4947	0.2150
Azure C	-6.0955	-52.7606	0.1565
308 K			
Adsorb.	$\Delta G$ (KJ/Mol)	$\Delta H$ (KJ/Mol)	$\Delta S$ (KJ/Mol)
Azure A	-5.8179	-45.0868	0.1274
Azure B	-7.8503	-77.4947	0.2261
Azure C	-11.2490	-52.7606	0.1347
318 K			
Adsorb.	$\Delta G$ (KJ/Mol)	$\Delta H$ (KJ/Mol)	$\Delta S$ (KJ/Mol)
Azure A	-6.1702	-45.0868	0.1223
Azure B	-6.3900	-77.4947	0.2235
Azure C	-6.3652	-52.7606	0.1458
328 K			
Adsorb.	$\Delta G$ (KJ/Mol)	$\Delta H$ (KJ/Mol)	$\Delta S$ (KJ/Mol)
Azure A	-6.6103	-45.0868	0.1576
Azure B	-8.4507	-77.4947	0.2105
Azure C	-9.2558	-52.7606	0.1326

Thermodynamic parameter, like  $\Delta H$ ,  $\Delta S$  and  $\Delta G$  were determined from the slope and intercept of Van't Hoff's plot of  $\ln K$  versus  $1/T$ . Table show the thermodynamic parameters  $\Delta H$ ,  $\Delta S$  and  $\Delta G$  for the removal of Azure A, B and C by Iraqi porcelanite rocks respectively. The  $\Delta G$  values are found to be negative at all tested temperatures (25- 55°C), indicating that the adsorption of Azure A, B and C dyes on to Iraqi Porcelanite rocks are spontaneous and thermodynamically favorable. It has been reported that  $\Delta G$  up to -20 KJ/mol are due to electrostatic interaction between sorption sites and the metal ion (physical adsorption), while  $\Delta G$  values more negative than -40 KJ/mol involve charge sharing or charge transfer from the biomass surface to the metal ion to form a coordinate bond(chemical adsorption)[17]. The negative values of apparent enthalpy change shoe an exothermic physical adsorption favoured by increased temperature [18], i.e., physisorption conducted with van der waals forces. The apparent entropy change values are almost constant over the temperature range. The positive entropy characterize an increased disorder of the system due to the loss of water which surrounding the dye molecules at the sorption on the Iraqi Porcelanite rocks. It can be suggested that the driving force for adsorption process is an entropy effect[19].

### Adsorption isotherms

The experiment equilibrium adsorption data were analyzed using Langmuir and Freundlich adsorption isotherm models [20,21]:

The Langmuir isotherm is represented by the following equation:

$$\frac{C_e}{Q_e} = \frac{1}{ab} + \frac{C_e}{a} \dots\dots(5)$$

Here  $C_e$  ( $\text{mg}\cdot\text{L}^{-1}$ ) is the equilibrium concentration of dye,  $Q_e$  is the amount of dye adsorbed at equilibrium ( $\text{mg}\cdot\text{g}^{-1}$ ).  $a$  and  $b$  are Langmuir constants related to the adsorption capacity and energy of adsorption respectively [22]. The linear plots of  $C_e/Q_e$  versus  $C_e$  suggest the applicability of the Langmuir isotherms (Fig.9). The values of  $a$  and  $b$  were determined from from slope and intercept of the plot Table (4).

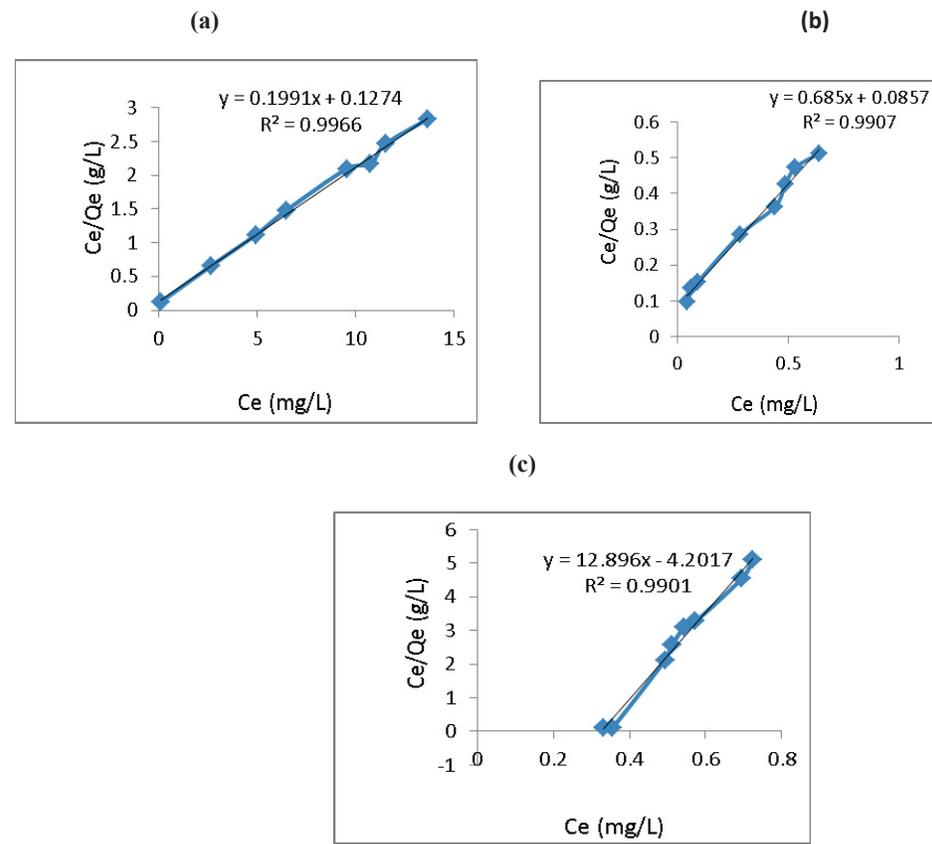


Fig.(9) Langmuir adsorption isotherms for adsorption of (a) Azure A (b) Azure B (c) Azure C on adsorbent Iraqi porcelanite rocks.

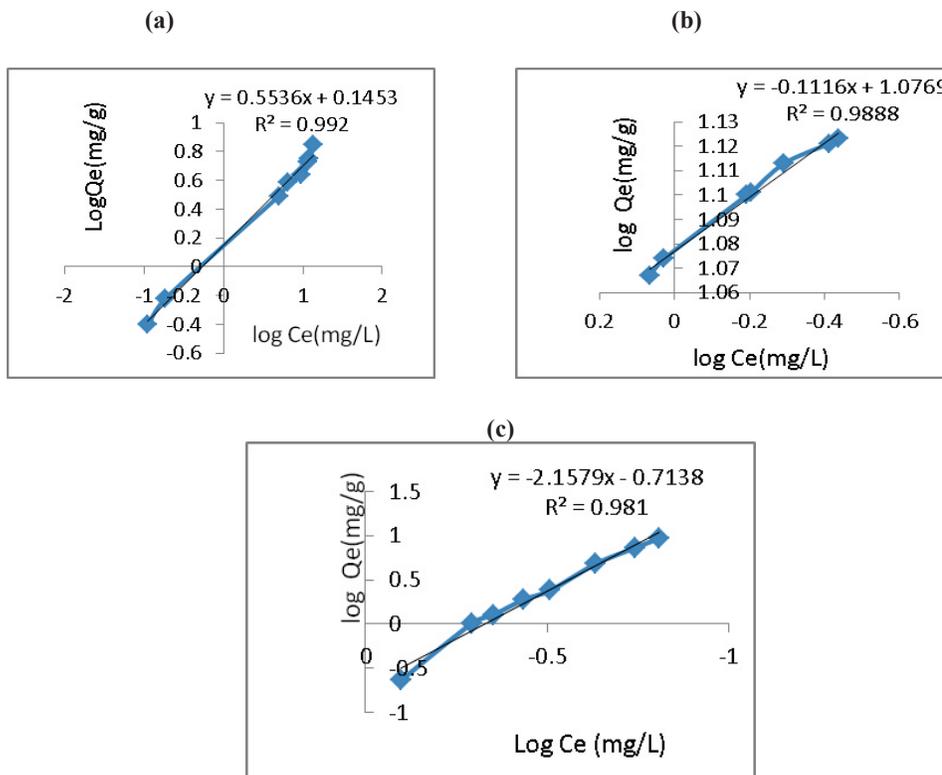
Figure.9 shows a linearized plot of  $C_e/Q_e$  against  $C_e$ . values of a, and b are given in Table (4).The Langmuir adsorption capacities vary from 5.0251 mg/g to 23.8095 mg/g for Azure A, from 1.4598 mg/g to 11.6279 mg/g for Azure B and from 0.4071 mg/g to 5.9880 mg/g for Azure C dyes respectively onto the Iraqi Porcelanite rocks with the increase in temperature from 298K to 328K.This indicates the adsorption at high operating temperature. From the values, it is calculated that the maximum adsorption corresponds to a saturated mono layer of dye molecules on the adsorbent surface with constant energy and there is no transmission of dye molecules on the adsorbent surface [23].The Langmuir isotherm fits quite well with the experimental data with good correlation coefficient. The essential features of Langmuir isotherm can be expressed in terms of dimensionless constant separation factor,  $R_L$  that is calculated by the following equation to confirm the favourability of the adsorption process:

$$R_L = 1 / (1 + bC_0) \dots (6)$$

Where  $C_0$  is the initial dye concentration in solution ( $\text{mg.L}^{-1}$ ) and b is the Langmuir constant ( $\text{Lmg}^{-1}$ ). The value of  $R_L$  indicates the type of the isotherm to be either favourable ( $0 < R_L < 1$ ), unfavourable ( $R_L > 1$ ), linear ( $R_L = 1$ ) or irreversible ( $R_L = 0$ ). The value of  $R_L$  was found to be between 0.0043 and 0.2881 and confirm that the adsorption process is favourable. Freundlich adsorption isotherm model used to explain the adsorption phenomenon is represented by the following equation:

$$\log Q_e = \log K_f + \frac{1}{n} \log C_e \dots (7)$$

Here  $K_f$  and n are constants incorporating all factors affecting the adsorption capacity and intensity of adsorption, respectively. Linear plot of  $\log Q_e$  versus  $\log C_e$  shows that the adsorption of Azure A, B, and C dyes follows the Freundlich isotherm (Fig.10). The values of  $K_f$  and n were calculated from the intercept and slope of the plot. The magnitude of the exponent (n) gives an indication of the favourability and  $K_f$  the capacity of the adsorbent/adsorbate. The values of  $1/n$ , less than unity is an indication that significant adsorption take place at low concentration but the increase in the amount adsorbed with concentration becomes less significant at higher concentration and vice versa [24].



**Fig.(10)** Freundlich adsorption isotherms for adsorption of (a) Azure A (b) Azure B (c) Azure C on adsorbent Iraqi porcelanite rocks.

**Table(4): Langmuir and Freundlich parameters of adsorption isotherms at (298 – 328 )K**

298 K							
Adsor.	Langmuir isotherm				Freundlich isotherm		
	a (mg/g)	b (mg/L)	Correlation coefficient (r <sup>2</sup> )	R <sub>L</sub>	Intercept (K <sub>f</sub> )	Slope (n)	Correlation coefficient (r <sup>2</sup> )
Azure A	5.0251	1.5671	0.996	0.0835	1.3963	1.8083	0.992
Azure B	1.4598	8.0645	0.990	0.0174	11.967	9.0090	0.988
Azure C	0.0775	3.0684	0.990	0.0444	5.1641	0.4636	0.981
308 K							
Adsor.	Langmuir isotherm				Freundlich isotherm		
	a (mg/g)	b (mg/L)	Correlation coefficient (r <sup>2</sup> )	R <sub>L</sub>	Intercept (K <sub>f</sub> )	Slope (n)	Correlation coefficient (r <sup>2</sup> )
Azure A	6.8027	1.9342	0.990	0.0687	0.9527	0.9380	0.986
Azure B	2.8571	0.7642	0.993	0.1574	1.3708	1.5313	0.995
Azure C	4.2016	1.4253	0.967	0.0910	3.6559	0.6265	0.981
318 K							
Adsor.	Langmuir isotherm				Freundlich isotherm		
	a (mg/g)	b (mg/L)	Correlation coefficient (r <sup>2</sup> )	R <sub>L</sub>	Intercept (K <sub>f</sub> )	Slope (n)	Correlation coefficient (r <sup>2</sup> )
Azure A	20.8333	0.3529	0.940	0.2881	0.5902	0.9165	0.960
Azure B	6.3694	0.3747	0.995	0.2760	2.4945	2.8901	0.990
Azure C	0.4071	5.6338	0.955	0.0247	4.1020	0.7513	0.967
328 K							
Adsor.	Langmuir isotherm				Freundlich isotherm		
	a (mg/g)	b (mg/L)	Correlation coefficient (r <sup>2</sup> )	R <sub>L</sub>	Intercept (K <sub>f</sub> )	Slope (n)	Correlation coefficient (r <sup>2</sup> )
Azure A	23.8095	0.0826	0.989	0.0043	0.8279	0.7733	0.988
Azure B	11.6279	0.1945	0.778	0.0121	2.6607	2.2075	0.973
Azure C	5.9880	2.1978	0.883	0.0233	2.2908	4.8302	0.984

**Conclusions:**

The present study shows that the Iraqi porcelanite rocks can be used as adsorbent for the removal of Azure dyes A,B and C from aqueous solutions .The findings are: (1)The Iraqi Porcelanite rocks were characterized by XRD, FT-IR spectroscopy and SEM.(2) The amount of dye adsorbed was found as function of initial pH, adsorbent dose, and contact time.(3) The adsorption equilibrium data were found to fit the Langmuir isotherm ,indicating a monolayer adsorption on a homogenous surface.(4) The negative ΔG values obtained from van 't Hoff plots confirm that the adsorptions of three Azure dyes are spontaneous in nature and the negative ΔH values for the adsorption suggest that the adsorptions of three Azure dyes on Iraqi Porcelanite rocks are exothermic in nature while the positive ΔS values indicates that the degrees of freedom increase at the solid- liquid interface during adsorption of these Azure dyes onto Iraqi Porcelanite rocks.

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