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Adsorption of Reactive Black 5 dye onto two kinds of Poly (vinyl imidazole) in aqueous solutions.

Muhand K. Al Temimi [1] and Murat Erdem [2]

[1] *Basrah University, Marin Science Center, Iraq, Basrah*
[2]*Anadolu University, Faculty of Science, Department of Chemistry, Eski sehir, Turkey*
E mail mohanad7m@yahoo.com

Abstract

Removal of a Reactive Black 5 dye, from an aqueous solution was studied by adsorption onto two adsorbent: *poly* (*vinyl imidazole*) synthesized by using free radical polymerization and quaternized partially by 1-bromo hixa dekan (p-VImC$_{16}$). *Also cross linked poly* (*vinyl imidazole*) (p-VIm hydrogel) was synthesized by using free radical polymerization. The effects of pH, the initial Black 5 concentrations, and temperature in the adsorption process of Black 5 onto polymers were studied and from the obtained data; the isotherm, kinetic and thermodynamic parameters of adsorption were calculated. Adsorption equilibrium was studied by Langmuir and Freundlich isotherm models. Kinetic evaluations were performed by Lagergren-first-order, pseudo-second-order and intraparticular diffusion kinetic models changes of enthalpy, free energy and entropy and activation energy of the adsorption process were also calculated by using kinetic parameters.

Keywords: Adsorption , Dye, polymer , Kinetic , Thermodynamic

1.Introduction

Dye effluents, discharged from the dyestuff manufacturing, dyeing, printing, and textile industries may contain chemicals that exhibit toxic effects toward microbial populations and can be toxic and/or carcinogenic to mammals [1]. Dyes usually have a synthetic origin and complex aromatic molecular structures which make them more stable and more difficult to biodegrade. Today there are more than 10,000 dyes available commercially [2]. Reactive dyes are widely used in many textile-based industries because of their favorable characteristics, such as bright color [3]. However, up to 50% of reactive dyes are lost through hydrolysis during the dyeing process, and therefore, a large quantity of the dyes appears in wastewater [4]. Conventional methods for the removal of dyes in effluents include physical, chemical, and biological processes [5]. Many adsorbents have been tested for the possibility of lowering dye concentrations from aqueous solutions, such as activated carbon [6,7] peat [8,9], chitin [10,11], silica [12] and synthesized polymeric resin [13], agricultural waste [14], and polysaccharide-based materials [15]. These to name a few, are widely studied adsorbents. Hydrophilic and hydrophobic polymers have found wide applications in chemical separation [16,17].

Poly (1-vinylimidazole) (PVIm) is a hydrophilic polymer with several important properties and it is used for the preparation of polymeric dyes, catalysts, and ion-exchange resins [18]. Poly (1-vinylimidazole) contains two active centers a lone electron pair of pyridine N atom which exhibits were donated properties and an unsaturated pay-system of heterocyclic, which is characterized by withdrawing properties [19].

Because of the hydrophilic effect of PVIm is solvable in water. Being so it cannot be easily used as an adsorbent without modification. PVIm can be modificated by the reaction with alkyl halide has a long hydrocarbon chain with hydrophobic effect, So, the producted polymer will not be solvable in water and it can be used as adsorbent seccessfilly. PVIm hydrogel can also be used as a good adsorbent, hydrogels are three-dimensional cross-linked polymer networks that swell by adsorbing water, alcohol or other polar solvents may increase their size more than a hundred times. When dried, they shrink and recover their original volume.Although hydrogels are hydrophilic, they are insoluble in water because of their large molecular size. When hydrogels come in contact with aqueous solutions, they adsorb and retain the dissolved substances. For this reason, they have been in several studies proposed as water purification agents [20-25]. Hydrogels are prepared mostly by crosslinking copolymerization with a water-soluble divinyl monomer. Methylene bis-acrylamide (MBA) is the most common and commercially available co-monomer for crosslinking of water-soluble polymers [26].

2. Materials and Methods

2.1.Chemicals

Vinylimidazole (VIm) was purchased from Aldrich (99%) and used as the monomer. N, N'-methylenebisacrylamide (MBAAM) was obtained from Aldrich Chemical Company to used as cross limked material. Ltd. (99%) and used as the crosslinking agent. Azobisisobutyronitrile (AIBN) was purchased from Fluka used as an initiator. 1-Bromo hex Dekan (Aldrich), And the Black 5 dye obtained from (Merck) Company.

2.2. Synthesis of Crosslinked Poly (1-vinylimidazole) hydrogels

(0.624 mol) 1- vinylimidazole (VIm), and (9.0×10^{-4} mol) AIBN were mixed. AIBN was not soluble in water. Firstly, AIBN was dissolved in 1-vinylimidazole then (150 ml) of water were added. The (7.78×10^{-3} mols) of crosslinking agent BAAm were added to these aqueous solutions. The mixture was poured into (250 mls) round flask and placed in a water bath at 90 °C under a nitrogen atmosphere and reflected with mixing for 3 hours. After synthesis, the hydrogel was taken out of the round, cut into pieces of the desired size and shapes and washed repeatedly with an excess amount of deionized water, to remove the non-polymerized 1-vinylimidazole and other water soluble material. Clean hydrogel was dried in air and put in vacuum at 50 °C for two days and ground into the powder form[27].

2.2. Synthesis of Poly (1-vinylimidazole C_{16})

A Poly (1-vinylimidazole) was synthesized in a way similar to the last step without addition of crosslinking agent beam.

B Grounded p-VIm 10 gm and alkyl halogen(1-bromo hixa dekan) 0.266 mol were mixed by using 250 ml round-bottom with a single - neck flask on magnetic stirrer and refluxed with 150 ml THF. The mixing process continued for 80 hours.

The partially quartinized $PVImC_{16}$ that obtained was cleaned from unreacted alkyl halogens by using the soxhlette method with THF as a solvent and rinsed with adequate acetone on Whatman filter paper and finally dried at 50 ºC up to 48 hours in the vacuum oven[28].

2.3. Batch adsorption experiments

20mgs of $PVImC_{16}$ were transferred separately into 250 mls screw capped volumetric flask containing 50 mls of 500 mgs/l of black 5 dye (solved in distilled water), the mixture was stirred magnetically at 300 rpm at 20 C for 24 hrs. The PH was adjusted (2,3,4,5,6,7,8,9,10,11,12) by using 0.1 M of HCl and NaOH solution. After equilibrium, the solutions were filtered through filter paper and the concentration of dye in the filtrate was determined spectrophotometrically at 597 nm.

Adsorption Isotherm experiments were carried out at optimum conditions, the 20 mg of PVImC16 were transferred into 250 ml screw capped volumetric flask containing 75 ml of (400, 500, 600, 700, 800, 900, 1000) mgs/l of Black 5 dye . The mixture was stirred magnetically at 300 rpm at 20 C for 24 hrs . The pH adjusted at 2. After equilibrium, the solutions were filtrated through filter paper and the concentration of dye in the filtrate was determined spectrophotometrically.

The Kinetic studies were carried out with 20 mgs of PVImC16 transferred into 250 mls screw capped volumetric flask containing 75 ml of 700 mg/l of black 5 dye, the mixture was stirred magnetically at 300 rpm at (25,40,55,70) C by using water – bath controlling temperature and optimum PH, then 1ml of the mixture solution was taken out at different periods of time and was diluted to 25 mls to measure the concentration of black 5 dye spectrophotometrically at (597) nm .The batch adsorption experiments had been repeated for cross-linked PVIm hydrogel by the same procedures mentioned above.

3. Results and discussion

3.1. Characterization of the polymers

The FT-IR characteristic bands for PVIm appeared at 1500 cm^{-1}, at around 1290 cm^{-1} and 920 cm^{-1} due to C=N, C—N and ring stretching vibrations, respectively [29]. Qquaternized polymers at 2930 -2960 cm^{-1} due to CH_2N vibrations showed [30]. Elemental analysis (CHN) showed the percentage of Nitrogen for the modificated (p-VIm-C_{16}) used to compute the yield of modification percentage. It was %71.

3.2. Effect of pH:

The effect of pH on PVImC$_{16}$, and cross-linked PVIm hydrogel adsorption is apparent Figure 1. shows the effect of PH on the removal of black 5 dye by polymers adsorption. The initial concentration of black 5 was 500 mg/L. The adsorption of dyes was strongly pH dependent. For all polymers the highest black 5 adsorption capacity was experimentally observed at pH 2.

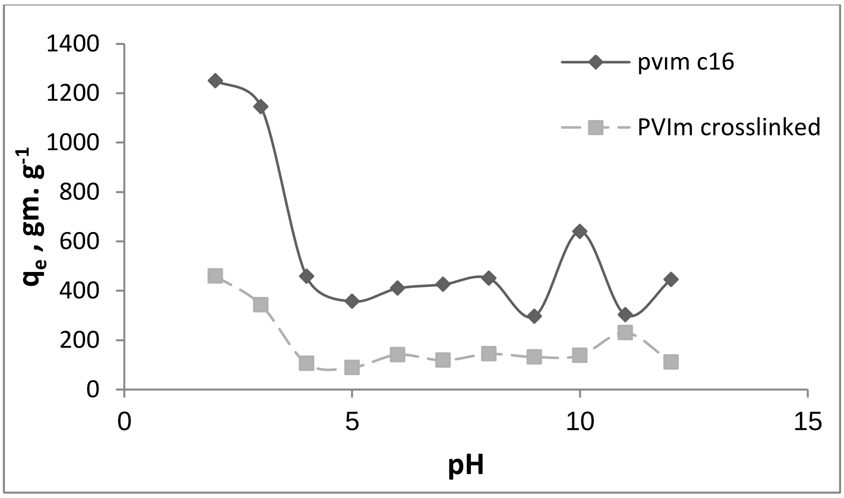

Figure 1. Effect of pH for the adsorption of black 5 on polymers (C=500 ppm, T=20)

3.3. Adsorption isotherms

The equilibrium adsorption isotherm is important for describing the mechanism of adsorption systems.

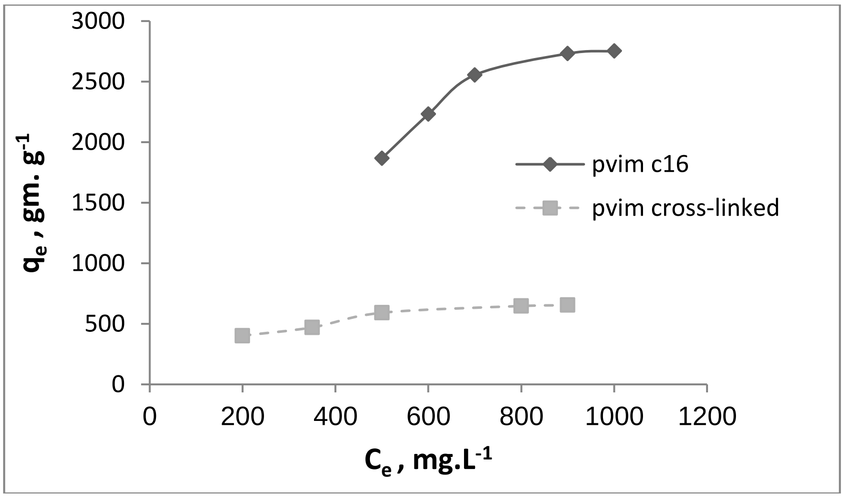

Figure 2. Effect of initial concentration for the adsorption of black 5 on polymers , [black 5] = 400 mg L^{-1}-1000 mg L^{-1}, *V*= 75 mL, pH= 2.0.

The equations of the Langmuir and freundlich models are commonly used equations to apply the adsorption isotherms. [31]. The Langmuir isotherm theory is based on the assumption of adsorption on a homogeneous surface [32]. Langmuir equation is generally expressed as follows

$$C_e / q_e = 1/ q_{\max} k_L + C_e / q_{maax} \qquad (1)$$

where Ce (in mg. L^{-1}) is the equilibrium dye concentration in the solution, q_e (in mg. g^{-1}) is the equilibrium dye concentration on the adsorbent, KL (in L. mg^{-1}) is a direct measure for the intensity of the adsorption process, and $q_{\max}$ (in mg. g^{-1}) is a constant related to the area occupied by a monolayer of adsorbate reflecting the adsorption capacity. From the slope, y-intercept of a plot of C_e/q_e versus C_e, $q_{\max}$ and KL can be determined [33].

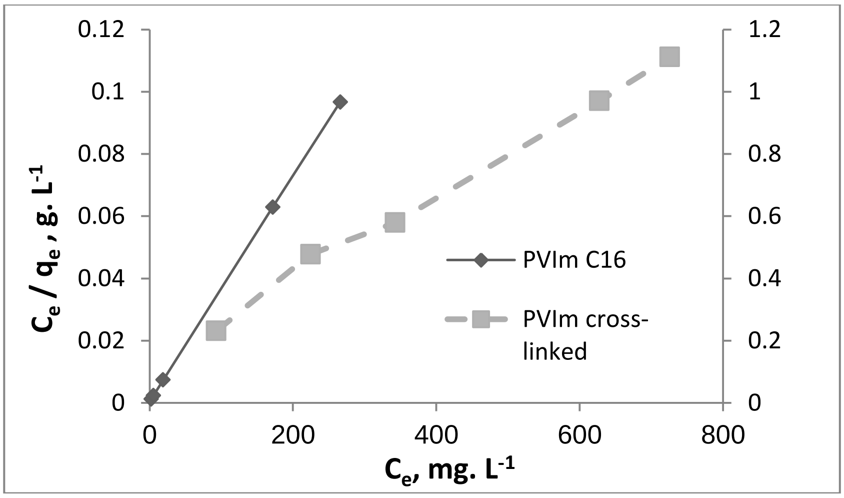

Figure 3. Langmuir adsorption isotherms for black 5 dye adsorption of polymers at 20 C° V= 75 ml, pH= 2,0.

The Freundlich isotherm describes adsorption where the adsorbent has a heterogeneous surface with adsorption sites that have different energies of adsorption. The energy of adsorption varies as a function of the surface coverage (q_e) and is represented by the Freundlich constant KF (L. g^{-1}) [34]. The equation is generally given by

$$\ln q_e = \ln K_f + n^{-1} \ln C_e \qquad (2)$$

where KF (in L. mg^{-1}) is a constant for the system defined as the adsorption or distribution coefficient and represents the quantity of an adsorbate adsorbed onto adsorbent for a unit equilibrium concentration. The slope 1/n is a parameter whose value changes from 0 to 1.

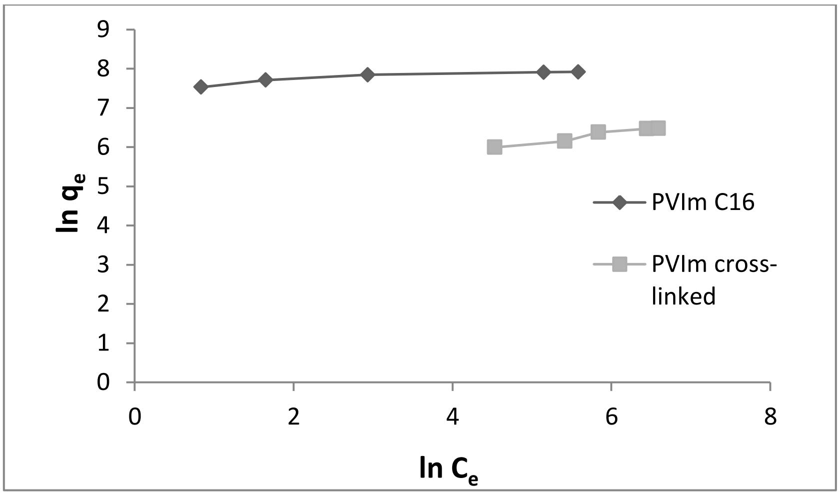

Figure 4. Freundlich adsorption isotherms for black 5 adsorption of polymers at 20 C° V=75 ml, pH= 2.0.

Table 1. Langmuir and Freundlich isotherms parameters for adsorption of black 5 onto polymers at 20 C° V= 75 ml, pH= 2,0.

	Langmuir isotherm			Freundlich isotherm		
Adsorbent	q_{max}, mg.g^{-1}	K_L, L. mg^{-1}	R^2	K_F, L. mg^{-1}	1/n	R^2
p-VIm	2762.43	0,7343	0,9999	1901.12	0.0724	0,851
p-VIm-C_{18}	740.74	0,0103	0,9952	127.83	0,251	0,955

3.4. Adsorption kinetics

In order to understand the mechanism of the adsorption process of black 5, the kinetics of the adsorption data was evaluated by means of the Lagergren first-order, pseudo –second order, and intraparticle kinetic models.

Lagergren first-order rate equation is

$$\ln(q_e - q_t) = \ln q_1 - k_1 t \qquad (3)$$

Where q_e and q_t (in mg.g^{-1}) are the amounts of the black 5 adsorbed at equilibrium and at time t (min), respectively, and k_1 (in min^{-1}) is the lagergren first order rate constant for adsorption. (Figure 5). The values of k_1 and q_1 were calculated from the plots slope and y-intercepts.

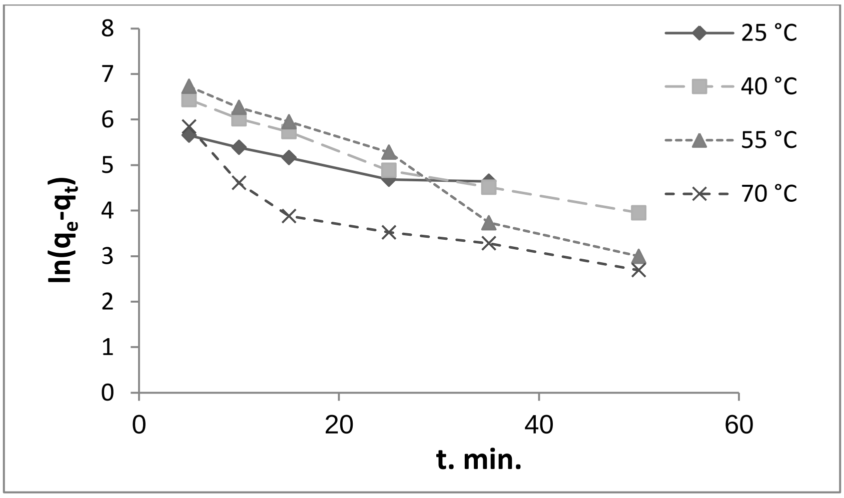

Figure 5. Lagergren-first-order Kinetic plots for the adsorption of black 5 onto p-VımC$_{16}$ at different temperatures.

The pseudo-second- order kinetic equation is

$$\frac{t}{q_{\mathrm{t}}} = \frac{1}{k_2 q_{\mathrm{m}}^2} + \frac{1}{q_{\mathrm{m}}} t \qquad (4)$$

where (in mg.g^{-1}) is the maximum adsorption capacity for the pseudo-second-order adsorption, q_t (in mg. g^{-1}) is the amount of the adsorbed adsorpate at equilibrium at time t (min) and k_2 (in g. mg^{-1}. min) is the equilibrium rate constant of pseudo-second- order adsorption. Fig. 6. gives the plots of t/q_{t} versus t for the adsorption of black 5 onto PVImC$_{16}$ at different temperatures. The values of k_2 and q_{m} of the pseudo-second- order kinetic model were calculated from the slope and y-intercepts.

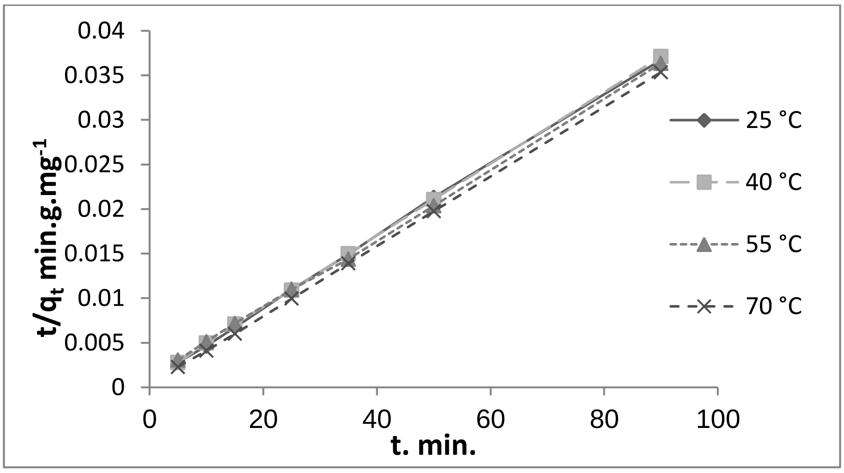

Figure6. Pseudo-second- order kinetic plots for the adsorption of black5 onto p-VımC$_{16}$ at different temperatures.

The intrapartical diffusion kinetic model can be represented by following equation:

$$q_{\mathrm{t}} = k_{\mathrm{p}} t^{1/2} + C \qquad (5)$$

where C is the intercept and k_{p} is the intrapartical diffusion rate constant in (mg. g^{-1}. min$^{-1/2}$). By using this model, the plot of the uptake, q_{t}, versus the square-root of time, $t^{1/2}$ in Figure 7. can presents intrapartical diffusion plots for the adsorption of black 5 onto p-VIm C$_{16}$ at different temperatures.

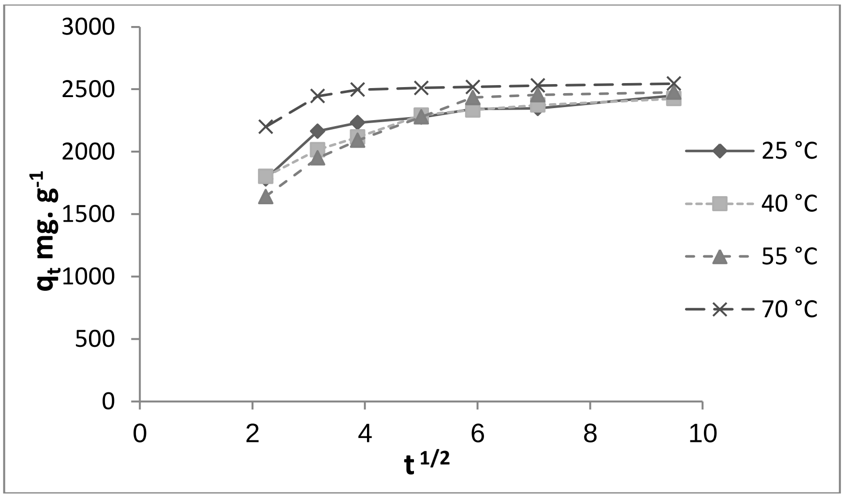

Figure 7. Intraparticle diffusion for the adsorption of black 5 onto p-VımC$_{16}$ at different temperatures.

The three kinetic models were also evaluated for the adsorption of black 5 onto cross-linked p-VIm hydrogel and the results are given in table 2.

The results show that the adsorption of black 5 onto PVImC16 and cross-linked p-VIm hydrogel follow the pseudo second order kinetic model because the highest R^2 value compared to the other kinetic models has been studied.

Table 2. Kinetic parameters of three models for black 5 onto adsorbents at (PH=2)

		Lagergren first order			Pseudo second order			Intraparticle diffusion		
adsorbent	ºC	k1 min^{-1}	q_1 (mg g^{-1})	R^2	K_2 (g. mg^{-1} min^{-1})	q_2 (mg g^{-1})	R^2	k_p(mg g^{-1} min$^{-1/2}$)	C	R^2_P
PVImC16	25	0.0366	2450.15	0.799	0.0001976	2487.6	100	72.554	1846.6	69.4
	40	0.0557	2424.67	0.968	0.0001807	2481.4	100	81.806	1764.3	80.95
	55	0.0867	2475.64	0.975	0.000134	2570.1	100	111.24	1605.4	77.95
	70	0.0593	2545	0.808	0.000676	2564.1	100	34.561	2282.1	50.48
PVIm cross-linked	25	0.0668	354.885	0.902	0.000783	374.5	99.6	19.925	217.49	98.15
	40	0.0685	391.292	0.954	0.000691	414.9	99.9	26.574	216.86	86.51
	55	0.0577	475.03	0.966	0.000487	505.1	99.7	31.504	258.48	98.54
	70	0.077	484.132	0.9595	0.000571	512.82	99.8	29.655	285.99	97.84

3.5. Adsorption thermodynamics

The thermodynamic parameters for the black 5 adsorption process such as enthalpy of adsorption (ΔH°), free energy change (ΔG°) and entropy change (ΔS°) were determined using the following equations:

$$\Delta G^\circ = -RT \ln K_L = -RT \ln \frac{C_A}{C^\circ} \qquad (6)$$

where C_A and C° are the equilibrium concentration of black 5 on the adsorbent (mg. g^{-1}) and in the solution (mg. L^{-1}), respectively.

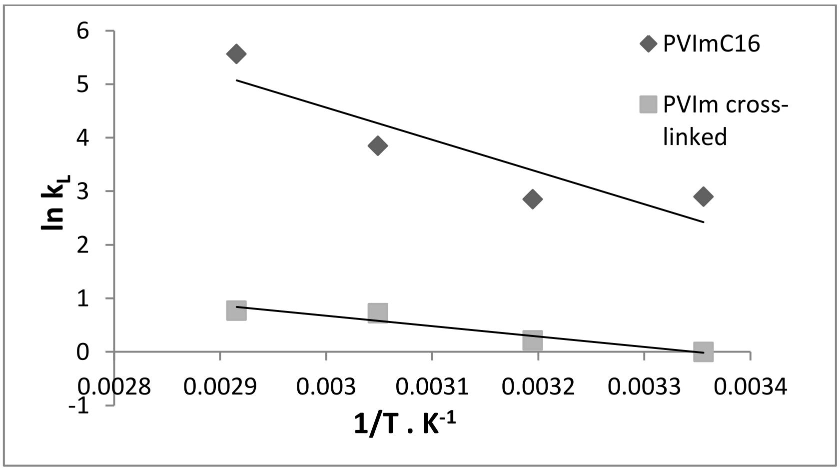

Figure 8. Plot of ln k_L vs. 1/T estimation of thermodynamic parameters for black 5 onto polymers.

$$\ln KL = \frac{\Delta S^\circ}{R} - \frac{\Delta H^\circ}{RT} \qquad (7)$$

ΔS° and ΔH° were determined from the slope and intercept of plot of $\ln KL$ versus $1/T$ as showen in Figure 8 than ΔG° can be determined by the following equation:

$$\Delta G^\circ = \Delta H^\circ - T\ \Delta S^\circ \qquad (8)$$

The positive value of ΔH indicates the endothermic process. It is indicated when the effect of temperature on adsorption capacities is studied The parameter ΔS° is used to identify the spontaneity in the adsorption process. The positive value of ΔS° reflects the affinity of polymers and black 5.

The negative value of ΔG° (table 3), indicates that the adsorption process leads to a decrease in Gibbs free energy and confirms the feasibility of the process and the spontaneous nature of the adsorption with a high preference of black 5 on polymers.

The activation energy (E_a) values of black 5 adsorption onto adsorbents are calculated from Arrhenius equation as below:

$$\ln k_2 = \ln A - \frac{E_a}{RT} \qquad (9)$$

where E_a is the activation energy; k_2 is the pseudo-second-order rate constant; A is the preexponential factor. Plots of $\ln k_2$ of the adsorbents versus $1/T$ are the slop of straight lines equal to Ea/R . Activation energy values of black 5 adsorption onto adsorbents are given in (figure 9) and (table 3).

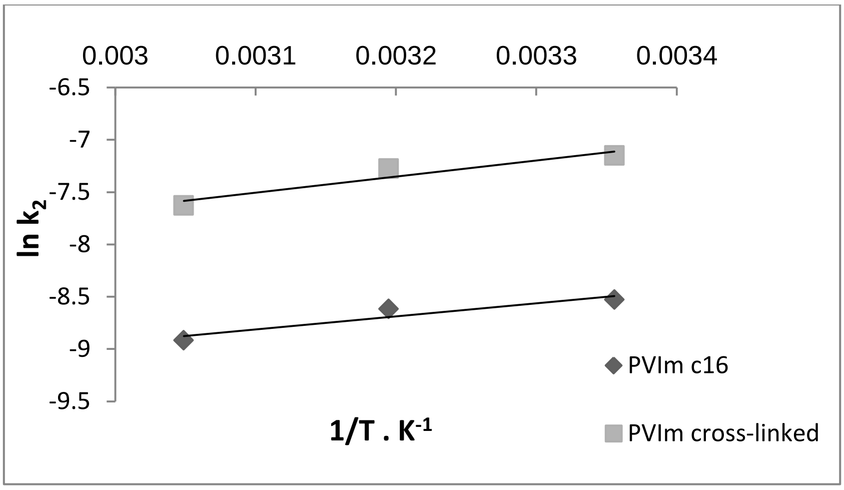

Figure 9. Arrhenius Graphic for black 5 adsorption onto adsorbents in different temperatures.

Table 3 Thermodynamic parameters for adsorption of black 5 onto adsorbents

Polymer	t, °C	E_a, kJ mol^{-1}	ΔH^0, kJ mol^{-1}	ΔS^0, J $K^{-1}mol^{-1}$	ΔG^0, kJ mol^{-1}
p-Vim C16	25	10.42	50.025	188.02	−6.005
	40				−8.826
	55				−11.646
	70				−14.467
p-Vim cross-linked	25	12.76	16.105	53.93,7	−0.033
	40				−0.776
	55				−1.585
	70				−2.394

4. Conclusions

The maximum adsorption of Black 5 on the PVIm C_{16} and PVIm cross linked is obtained at 70 °C, pH 2. The adsorption capacities increase (as temperatures do) in the studied range (25-70) °C.

Langmuir isotherm model and pseudo-second order kinetic model have the correlation coefficients higher than 0.999 and more fitted compared to with other models in the concentrations and temperatures range that have been studied.

Thermodynamic results show that the adsorption of black 5 onto adsorbents is physisorption adsorption mechanism. The positive values of $\Delta H°$, $\Delta S°$ and the negative value of $\Delta G°$ show the endothermic and spontaneous nature of the adsorption process.

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امتزاز الصبغة الفعالة السوداء 5 على نوعين من البولي فينايل ايميدازول في المحلول المائي

مهند كاظم التميمي [1] و مرات اردم [2]

1 مركز علوم البحار / جامعة البصرة/ البصرة
2 قسم الكيمياء / كلية العلوم / جامعة الاناضول اسكيشهير تركيا
الايميل : mohanad7m@yahoo.com

الخلاصة

تم دراسة إزالة الصبغة السوداء5 من المحلول المائي بامتزازها على نوعين من البولي فينايل ايميدازول. حضر الاول باستخدام بلمرة الجذور الحرة وطور الناتج بإضافة برومو هكسا ديكان .حضر البوليمر الثاني بشكل هلامي بإضافة عامل مشبك وكذلك جرت البلمرة باستخدام الجذور الحرة. درس تأثير درجة الحموضة، والتركيز الابتدائي للصبغة ، ودرجة الحرارة في عملية امتزاز الصبغة السوداء 5 على البوليمرات. من البيانات التي تم الحصول عليها تم حساب المعاملات الحركية والحرارية لعملية الامتزاز. تمت دراسة توازن الامتزاز باستخدام طريقتي (Langmuir , Freundlich) وكذلك درست حركية الامتزاز باستخدام ثلاث نماذج هي(الدرجة الاولى والدرجة الثانية الكاذبة و intraparticula) وحسبت منها الدوال الحرارية (التغير بالطاقة الحرة وانثالبي التكوين وحالة العشوائية للنظام وطاقة التنشيط) لعملية الامتزاز على كل من البوليمرين.

الكلمات المفتاحية : امتزاز , صبغة, بوليمر , الحركية, الديناميك الحراري