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Kinetics studies on the adsorption behaviour of Basic Blue 9 dye on macroporous ion exchanger resins

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ABSTRACT

Among various types of adsorbents used in the treatment of wastewaters impurified with dyes, the ion exchange resins still have an important place. The aims of this paper are: to evaluate the influence of contact time on the dye adsorption from aqueous solutions on Purolite macroporous ion exchange resins for strong acid (C-145) and for weak acid (C-107E), to understand the mechanisms that govern dye removal and to find an appropriate kinetic model to describe the process. In order to investigate the mechanism of adsorption and the potential rate controlling steps, pseuso-first- and second-order equations, the intraparticle diffusion equation and the Elovich equation have been used to test experimental data. The kinetics of the adsorption process of Basic Blue 9 dye onto Purolite ion exchanges follows a pseudo-second- order rather than pseudo-first- order model, and both the film and the intraparticle diffusion mechanisms are involved in the adsorption processes, and may be the rate determining step. The value of activation energy (E_a) confirms the endothermic nature of the adsorption process.

Keywords: Adsorption; Basic Blue 9 dye; Kinetic study; Purolite resins

1. Introduction

Nowadays, surface water and groundwater are polluted with many types of organic and inorganic pollutants. Among them, a special place is that of the dyes which are present in industrial effluents from different industries (textile, leather, paper, or chemical synthesis). Their presence, be it in small quantities, has a negative impact on the aquatic environment since they reduce the oxygenation processes, and they can be decomposed into by-products with a much stronger toxic effect compared to the initial dyes.

Dyes are organic substances with a complex structure; most times they are not readily biodegradable substances and they could be decomposed into small molecules with higher toxicity and carcinogenicity compared to the considered dyes [1]. Thus, the dyes must be removed from the industrial effluents before their discharge in the environment or in the surface

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waters. In this context, along the time, various methods for their removal from the aqueous medium were developed and tested, such as: chemical oxidation, coagulation/flocculation, ozonation, ion exchange, electrochemical reduction, adsorption and anaerobic biological treatment [1–10]. Among all of these, adsorption is one of the most efficient methods due to the low cost for implementation, the simplicity of design, the ease of operation and the possibility of using different types of materials as adsorbents. Some of the adsorbents used to remove dyes from wastewater are: activated carbon (the most common), ion exchangers, polymeric adsorbents, zeolites, silica, and low-cost materials such as peat, bentonite clay, fly ash, agricultural by-products and industrial wastes [1,11–21].

Despite the high production cost, the ion exchanger resins are adsorbents preferred nowadays for industrial scale applications because they provide high efficiency of the discoloration process, are resistant to the presence of various organic and inorganic compounds present in industrial effluents alongside dyes, can be regenerated many times, fact which reduces the total cost of the wastewater treatment process [13,14,22–26].

This article presents the kinetic considerations about the final adsorption of the cationic Basic Blue 9 dye in batch conditions onto Purolite cation exchange resins. For this purpose, the resin loading with dye was monitored according to the contact time of the phases, and the obtained data were processed using some models from the literature in order to establish the mechanism of the process and the rate determining step.

2. Materials and methods

2.1. Materials

The experiments were carried out using two types of commercially available macroporous cation exchange resins provided from Purolite International LTD (UK) [13] (Fig. 1):

1. **C 154**—presented in Na^+ ionic form, has a macroporous structure based on polystyrene crosslinked with divinylbenzene and sulphonic acid ($-SO_3^-$) as functional group;
2. **C 107E—**presented in H^+ ionic form, has a macroporous structure based on polymethacrylic crosslinked with divinylbenzene and carboxylic acid ($-COO^-$) as functional group.

The Basic Blue 9 dye is a phenothiazine type (C.I. 52015; Standard Fluka AG) and was used without further purification, as commercial salt with exchangeable chloride ions. Having molecular formula $C_{16}H_{18}N_3SCl$, is characterized by MW = 319.85 g/mol and maximum wavelength $\lambda_{max} = 660$ nm. The aqueous stock solution of 320 mg/L was obtained by dissolving the corresponding amount of dye in double distilled water, and working solutions were prepared by appropriate dilutions.

2.2. Kinetic sorption methodology

The effect of phase contact time was studied using the "limited batch" technique, which implies contacting a sample of 0.4 g resin with 100 mL solution of dye with 51.2 mg/L initial concentration, and pH adjusted to 10 value. The system, under intermittent stirring, was maintained at 2, 25 and 45°C temperature. After established time intervals (from 2.5 to 300 min) volumes of 0.5 mL were spectrophotometrically analyzed (respecting the conditions of Lambert–Beer law) in order to determine the dye content, using a JK-VS-721N VIS spectrophotometer. The adsorption capacity of the resins was evaluated by means of the amount of retained dye:

$$q = \frac{C_0 - C}{G} \cdot V, \text{ (mg of dye/g of resin)} \tag{1}$$

and the extent of adsorption was expressed by the fractional attainment of equilibrium, *F*:

$$F = \frac{q_t}{q_e} \tag{2}$$

where C_0 and C are the initial and the equilibrium (residual) concentration of dye in solution (mg/L), G is the amount of resins (g) and V is the volume of solution (L), q_t and q_e (mg/g) are the amounts of dye adsorbed at time t and at equilibrium (24 h), respectively.

3. Results and discussions

It was established that the absorption of Basic Blue 9 onto C 145—strong acid and C 107E—weak acid Purolite cation exchangers resin depends on the physico-chemical properties of the resin, the amount of adsorbent, the initial dye concentration, the pH value, the temperature and the phase contact time [13]. Also, the modelling of the experimental data according to

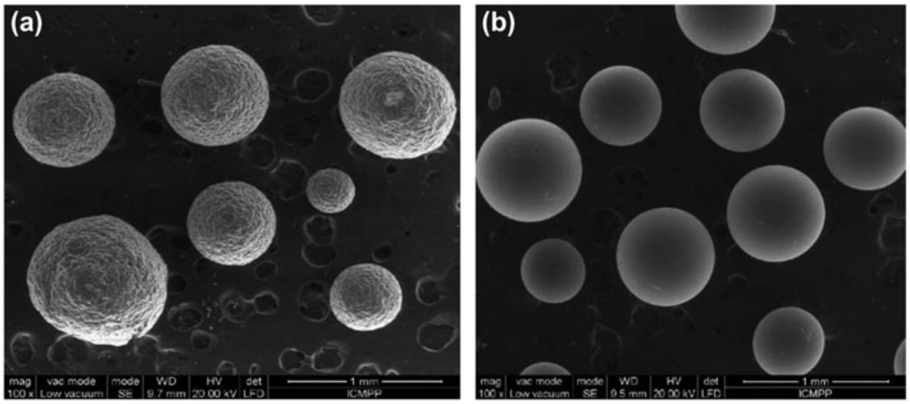

Fig. 1. Macroporous structure charcterization by ESEM microphotographs: C 107-E (a) and C 145 (b).

the Freundlich, Langmuir, and Dubinin–Radushkevich adsorption isotherms (Fig. 2), suggests that the Langmuir isotherm is the best one in simulating the adsorption equilibrium.

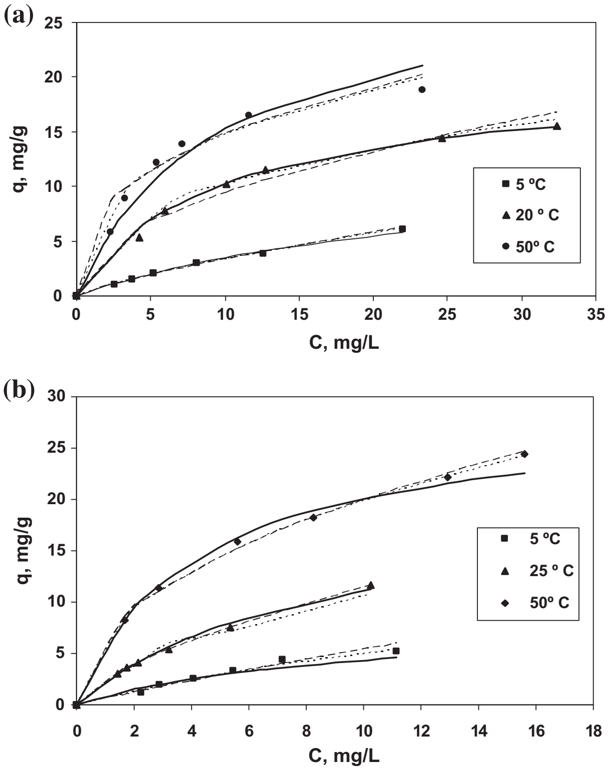

Fig. 2. Adsorption isotherms of BB 9 on the C 145—strong (a) and C 107E—weak acid (b) Purolite cation exchangers resin at three temperatures with the results fitted to the Freundlich equation (—), Langmuir equation (—) and D–R equation (……); ♦, ▲, ■: experimental points. Conditions: 6 g/ L adsorbent dose, 24 h contact time and pH 10.

Taking into account these few preliminary conclusions, the kinetic experiments were realized in aqueous solution with pH 10 with an initial working dye concentration corresponding to the saturation value of the sorbent: $C_{0,\mathrm{MB}} = 96$ mg/L and three temperatures: 2, 25 and 45˚C.

3.1. The phase contact time in adsorption process

An important operating parameter used to study the adsorption kinetics is the contact time of the liquid phase with the solid phase. Studying the influence and the effect of the contact time on the amount of dye retained allow establishing the time necessary to reach equilibrium (Fig. 3(a) and (c)), the time required for the retention of half the dye ($t_{1/2}$) (Fig. 3(b) and (d)), values used then in kinetic modelling.

As seen in Fig. 3(a) and (c), the values of the amounts of adsorbed dye at equilibrium and the fractional attainment of equilibrium rapidly increase with the contact time, especially during the first 500 min, and after that, the rate of both dye adsorption processes becomes slower and the time period required for maximum removal of dyes was found to be up to 1,500 min. Also, the sorption half-time ($t_{1/2}$) decreases with increasing the temperature from 750 min at 2˚C to values around 500 min at 45˚C. The obtained results confirmed the endothermic nature of the adsorption processes studied.

3.2. Adsorption kinetic modelling

The adsorption of dyes onto solid ion exchange resins must be considered as a liquid–solid phase reaction and the most important steps involved are [22,26]:

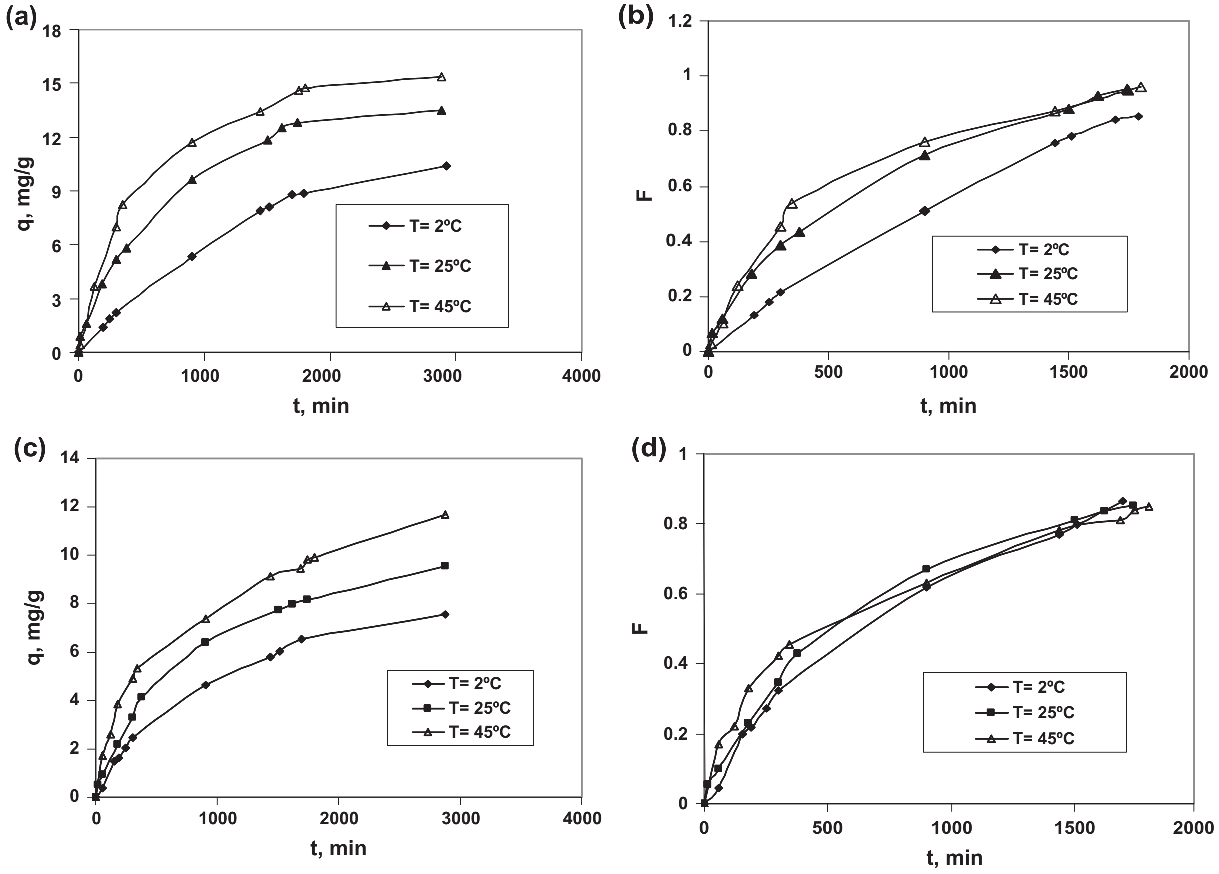

Fig. 3. Effect of phase contact time on the Basic Blue 9 cationic dye removal by C 145—strong (a and b) and C 107E—weak (c and d) acid Purolite cation exchangers resin: 96 mg/L; 6 g/L adsorbent dose and pH 10.

(1) diffusion of the dye molecules through the liquid layer on the surface of adsorbent (*film diffusion*);
(2) diffusion in the pores of the adsorbent (*pore diffusion* or *intraparticle diffusion*);
(3) *chemical reaction* (ion exchange, complexation) which may occur between the molecules of dye and the functional groups on the surface of the ion exchangers.

The adsorption kinetic is governed by the slowest of these processes. The study of adsorption kinetics is important for: (i) establishing the mechanism of the process; (ii) obtaining useful information for desorption process in the idea of using resin as pharmaceutical carriers for Basic Blue 9 which favour the controlled release of dye in physiological conditions in the human body.

In order to investigate the type of mechanism (particle diffusion/film diffusion or chemical reaction) involved in the adsorbtion of Basic Blue 9 dye onto the considered Purolite cation exchangers and to establish the rate determining step of the process, the kinetic data were processed and interpreted using three of the most used kinetic models presented in the literature: *pseudo-first order*—implies that adsorption is preceded by diffusion through boundary (Lagergren model, Eq. (3)), *pseudo-second order*—assumes that the rate limiting step may be chemical adsorption involving valence bonds or covalent bonds between adsorbent and adsorbate (Ho model, Eq. (4)) and *Elovich equation*—used in chemical adsorption without desorption of products (Eq. (5)) [27,28].

$$\lg(q - q_t) = -\frac{k_1}{2.303}t + \lg q \qquad (3)$$

$$\frac{t}{q_t} = \frac{1}{k_2 q^2} + \frac{1}{q} \cdot t \qquad (4)$$

$$q_t = \frac{1}{\beta}\ \ln(\alpha \cdot \beta) + \frac{1}{\beta}\ \ln\ t \qquad (5)$$

where q and q_t are the amounts of adsorbed dye (mg/g) at equilibrium (24 h) and at any time t (min), respectively, and k_1 is the Lagergren rate constant of the first-order sorption (min^{-1}); k_2 is the rate constant of second-order sorption (g/mg min); $q^2 \cdot k_2 = h$ can be regarded as the initial sorption rate (mg/g min) as t approaches zero; β is the desorption constant (g/mg) and α is the initial sorption rate (mg/g min).

The kinetic parameters related to each model, calculated from the intercepts and slopes of the corresponding linear plots (Fig. 4), are presented in Table 1. The fitting of each model to the experimental data was estimated using the linear regression correlation coefficient, R^2.

As it can be seen in Fig. 4(a) and (b) the plot ln $(q - q_t)$ vs. t gives straight lines which did not pass through the origin of axes. Also, the q_{exp} values did not agree with the calculated q, all of these suggesting that the pseudo-first- order model is not well fitted for modelling of kinetic data.

The linearity of plots of t/q_t vs. t (Fig. 4(a) and (b)), the values of q calculated are much closer to the experimental value, q_{exp}, and the very higher values of the correlation coefficient, R^2, suggested that adsorption kinetics on both resins follows a pseudo-second-order kinetic model, and the dye adsorption processes could be controlled by chemical adsorption or chemisorption involving valence forces through sharing or exchange of electrons between the two phases involved. Similarly, previous studies of different research teams showed that the pseudo-second- order kinetic model is often applied with success to describe the kinetics of adsorption process of dyes from aqueous media [23,25,26,29,30].

The data were also processed using Elovich equation in order to verify if the studied processes can be described as a chemical adsorption. The R^2 values greater than 0.95 for the studied adsorption system suggest that the data fit well the Elovich equation. But, the linear representation q vs. $\ln t$ which do not

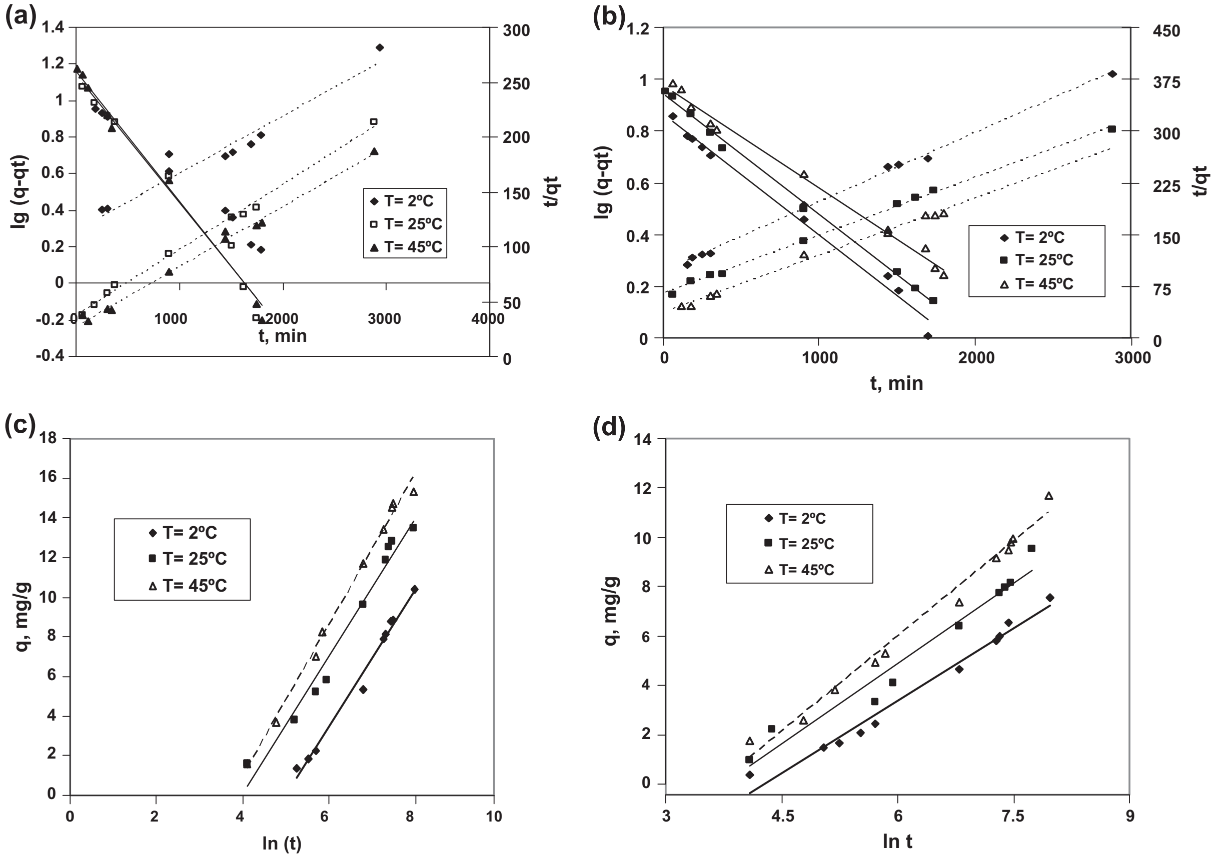

Fig. 4. Applicability of the pseudo-first (___), pseudo-second (…..) kinetic models (a and b) and Elovich equation (c and d) to cationic dye Basic Blue 9 sorption on Purolite resins: 96 mg/L; 6 g/L adsorbent dose and pH 10: C-145 (a and c) and C-107E (b and d).

Table 1
Kinetic parameters for the Basic Blue 9 cationic dye sorption on Purolite resins

	T, °C					
	2		25		45	
Kinetic model	C 145	C 107E	C 145	C 107E	C 145	C 107E
$(q_{\text{exp}})^{13}$, mg/g	21.83	17.46	31.984	27.77	50.984	41.768
Pseudo first- order kinetic						
k_1 (min^{-1})	0.001151	0.00115	0.00161	0.00115	0.00161	0.000921
q (mg/g)	11.66	7.326	13.583	8.75	14.524	9.52
R^2	0.9884	0.9892	0.982	0.9969	0.9838	0.9922
Pseudo second- order kinetic						
k_2 (g/mg min)	2.07×10^{-7}	8.95×10^{-4}	9.53×10^{-5}	1.119×10^{-4}	1.086×10^{-4}	1.85×10^{-4}
q (mg/g)	19.157	9.95	16.778	11.83	18.349	12.106
R^2	0.9632	0.9969	0.9952	0.9979	0.9952	0.994
Elovich model						
α (mg/g min)	0.00684	0.027	0.0659	0.052	0.0904	0.064
β (g/mg)	0.29	0.511	0.289	0.46	0.263	0.388
R^2	0.986	0.9799	0.9724	0.961	0.9937	0.9859

intersect the origin of the axes, confirmed that chemisorption cannot be the only step that controls the studied adsorption processes, conclusion in accordance with the previously calculated Langmuir parameters [13] and the diffusion processes remain the step that could control the adsorption dye onto both resins.

3.3. Diffusion mechanism

In order to establish the diffusion mechanism (*film diffusion* or *intraparticle*) which could be the rate determining step on the studied adsorption processes, the kinetic data were furthermore processed using two of the most used models: the *intraparticle diffusion model* (Eq. (6)), and *film diffusion model* (Eq. (7)) [31].

The *intraparticle diffusion model*, proposed by Weber and Morris (where k_{d} is the rate constant for intraparticle diffusion, mg/g min$^{1/2}$; c is intercept to the y axis):

$$q_t = k_{\text{d}} t^{1/2} + c \tag{6}$$

suggests that the intraparticle diffusion is involved in the sorption process if the plot q_t vs. $t^{1/2}$ (Fig. 5, Table 2) is linear and controls the adsorption process if the line passes through origin.

The *film diffusion model*, proposed by McKay (where k_{f} is the rate constant for film diffusion, min^{-1}; F is the fractional attainment):

$$\ln(1 - F) = k_{\text{f}} t \tag{7}$$

Specifies that the film diffusion is involved in the adsorption process if the plot $\ln(1 - F)$ vs. t (Fig. 5) is linear, and it is the rate-limiting step if the line passes through origin.

In Fig. 5 it can be noticed that the lines from the graphical representation are linear but none pass through the origins, which underline the assumption that both diffusion mechanisms are involved in the adsorption processes, and may be the rate determining step.

In the graphical representation of the dye amount adsorbed by Purolite ion exchange vs. $t^{1/2}$ (Fig. 5(a) and (b)) two line segments can be highlighted for both resins at the three temperatures. The first part is generally associated with the external mass transfer (film diffusion), while the second linear part indicates intraparticle diffusion into the porous structure of the adsorbent [27,32].

Also, from the graphical representation (Fig. 5(c) and (d)) it was noticed that by increasing the temperature the *film diffusion* could become the rate determining step of the adsorption process, as straight lines are very close to the intersection with the axis origin.

The studied adsorption processes take place very fast until reaching the saturation on the solid surface, followed by adsorption of the dye molecules inside the porous adsorbent particles, a process that takes place at a much lower speed.

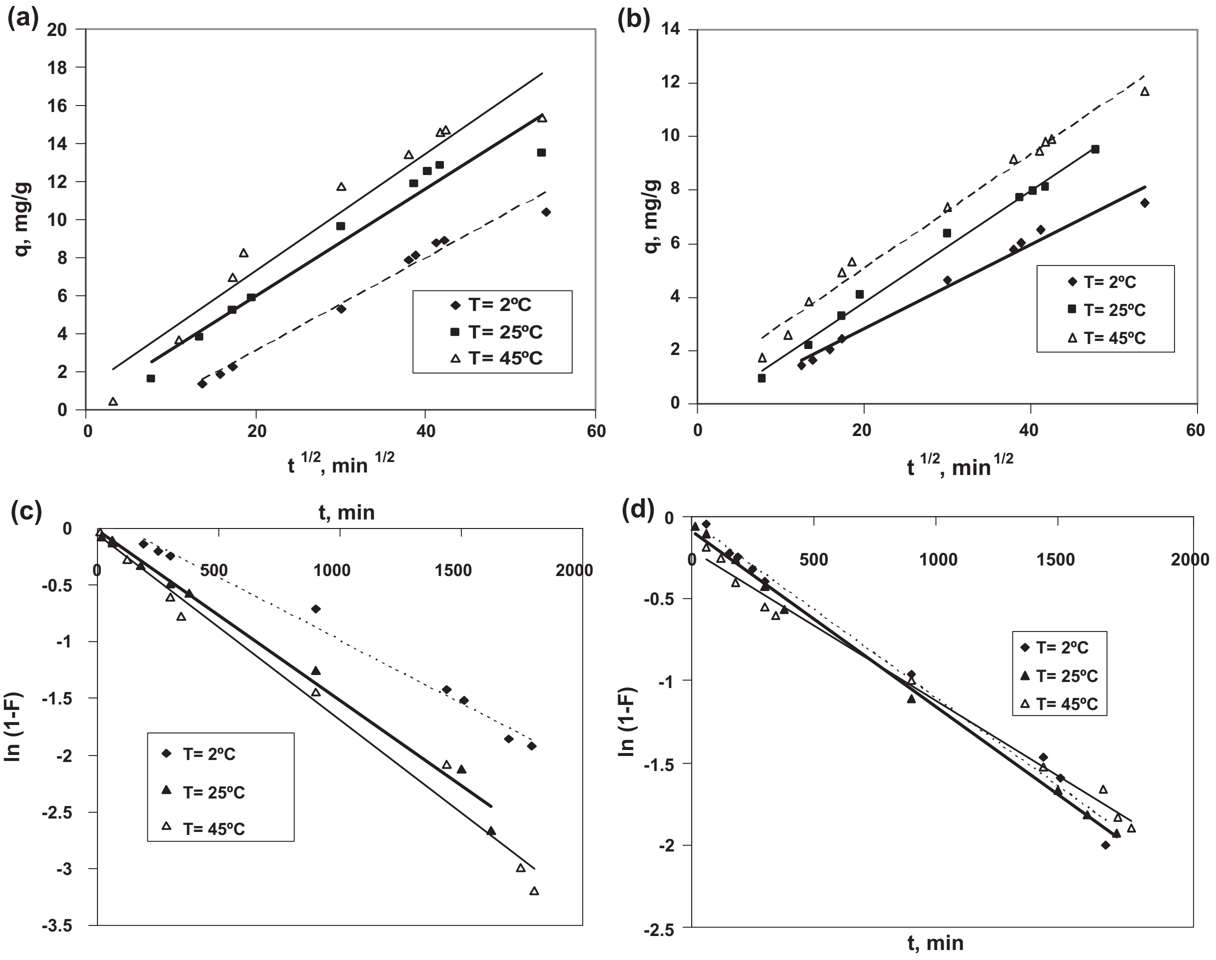

Fig. 5. Applicability of intraparticle diffusion kinetic model (a and b) and film diffusion model (c and d) in BB 9 dye sorption onto Purolite C 145 (a and c) and Purolite C 107 (b and d): C_0 = 96 mg/L; 6 g/L adsorbent dose and pH 10.

Table 2
Diffusion data for the BB 9 dye adsorption onto Purolite ion exchange

	Ion exchange/*T*, ℃					
	C 145			C 107		
Kinetic model	2	25	45	2	25	45
Intra-particle diffusion						
k_d (mg/g mg$^{0.5}$)	0.245	0.2827	0.3087	0.1578	0.2097	0.214
c	1.8396	0.2962	1.1325	0.3414	0.3841	0.7701
R^2	0.9796	0.9549	0.9399	0.9843	0.9918	0.9844
Film diffusion						
k_f (min^{-1})	0.0011	0.0015	0.0016	0.0011	0.0011	0.0009
R^2	0.9883	0.9875	0.9838	0.9892	0.9969	0.9919

3.4. Evaluation of activation energy

From a thermodynamic point of view, the batch adsorption process of cation dye onto studied ion exchangers seems to be an endothermic and spontaneous process ($\Delta G^\circ < 0$ and $\Delta H^\circ > 0$) [13].

The values of the mean free energy (E_{C145} = 8.909–10.314 kJ/mol; E_{C107E} = 8.7706–10.314 kJ/mol) obtained from the Dubinin–Radushkevich model, suggested that the proposed ion exchange is the main mechanism of the adsorption process [13].

Based on the values of the adsorption rate constant of the pseudo-second- order adsorption model, k_2, the activation energy, the energy necessary for the adsorbate species to interact with the adsorption sites on the surface of the solid phase, for the adsorption of BB 9 dye onto studied ion exchangers was evaluated using the Arrhenius equation (Eq. (8)) [33,34].

$$k_2 = k_0 \exp\left(-\frac{E_a}{RT}\right), \text{ or in linear form: } \ln k_2 = \ln k_0 - \frac{E_a}{RT} \quad (8)$$

where k_2 is the adsorption rate constant of the pseudo-second- order adsorption model, g/mg min, k_0 is the temperature independent factor, E_a is the activation energy of adsorption (kJ/mol), R is the gas constant (8.314 J/mol K) and T is the temperature in K.

The value of the activation energy and the k_0 factor determined from the slope and intercept of the Arrhenius plot (figure not shown) is 117.418 kJ/mol. The value of activation energy also provides information on the nature—physical or chemical—of the adsorption process. It is known that the activation energy for the physisorption process ranges from 5 to 40 kJ/mol, while chemisorption involves a high activation energy (40–800 kJ/mol) [35]. Additionally, Lazaridis and Asouhidou suggested that low activation energy values (<25–30 kJ/mol) indicate diffusion-controlled processes [36].

4. Conclusions

This paper completes the study of the batch adsorption process of the BB 9 dye onto Purolite ion exchange, and it can underline the following:

(1) For the adsorption process of Basic Blue 9 dye onto Purolite ion exchange C 145 and C 107E it was found that the kinetics follows pseudo-second- order model rather than pseudo-first- order model.

(2) The mechanism of the adsorption process is the *diffusion*, but depending on certain physical parameters (e.g. temperature, type of resin, etc.) the *film diffusion* or *particle diffusion* can be predominant. Both diffusion mechanisms may be the rate determining step at a certain time.

(3) The positive E_a value confirms the previous information about the endothermic nature of BB 9 dye adsorption onto Purolite ion exchangers, and corresponds to a chemisorption process controlled not only by diffusion.

(4) This study highlighted that the studied ion exchange, namely Purolite C 145 and Purolite C 107E can be used in the decolourization of industrial effluents.

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