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CRYSTAL VIOLET DYE ADSORPTION ON RASPBERRY LEAVES POWDER - KINETIC AND THERMODYNAMIC STUDIES

BY

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Abstract. In this study, kinetics and thermodynamics of crystal violet dye adsorption onto raspberry leaves powder was investigated. The effect of contact time, temperature and ionic strength was studied. The values of the equilibrium time and the experimental adsorption capacity were better that values for other similar adsorbents obtained from vegetal wastes. The thermodynamic study showed that the adsorption process is endothermic, spontaneous and favourable, and physical adsorption is the main mechanism implied in the dye retention on the adsorbent surface.

Keywords: adsorption, crystal violet, raspberry leaves, contact time, thermodynamic parameters.

1. Introduction

Natural waters are affected by discharges of untreated or improperly treated industrially wastewater. Thus, various chemicals resulting from industrial activities reach in the natural waters and negatively affect the environment

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(Chakraborty *et al.*, 2021; Franco *et al.*, 2020; Pang *et al.*, 2019; Shakoor and Nasar, 2018).

Many industries use significant amounts of dyes in technological processes. Over 10000 different dyes are produced annually in quantities exceeding 7×10^5 tons. Of this amount, about 10-15% end up in wastewaters that are discharged into the environment. Dyes are a category of toxic, carcinogenic and mutagenic substances. Therefore, eliminating them from industrial effluents is a priority (Bagchi and Ray, 2015; Chakraborty *et al.*, 2021; Loulidi *et al.*, 2020; Pang *et al.*, 2020).

Many methods of removing dyes from water have been used, but adsorption is evidenced by high efficiency, flexibility, selectivity and low costs. The costs of the adsorption process depend mainly on the adsorbent material which represents 70% of the total costs (Chahinez *et al.*, 2020; Franco *et al.*, 2020; Loulidi *et al.*, 2020; Pang *et al.*, 2019).

The current trend is to find efficient adsorbent materials, available in large quantities and at the lowest possible cost. This category includes industrial wastes, agricultural wastes, bioadsorbents and natural materials (minerals and vegetable). Various biomasses and vegetable wastes have been used successfully to remove dyes from aqueous solutions (Georgin *et al.*, 2018; Georgin *et al.*, 2020; Keereerak and Chinpa, 2020; Kulkarni *et al.*, 2017; Loulidi *et al.*, 2020; Franco *et al.*, 2020; Pang *et al.*, 2019; Pang *et al.*, 2020; Shakoor and Nasar, 2018; Nieva *et al.*, 2020).

The raspberry (*Rubus idaeus*) is a fruiting shrub that is part of the *Rosaceae* family. It grows spontaneously or cultivated in countries with temperate climate. The fruits are used in the food, pharmaceutical and cosmetic industries or for consumption, due to their taste and content rich in vitamins, minerals and various substances useful for human health (Chevallier, 2016; Yang *et al.*, 2022).

In this paper, raspberry leaves were used as easy available and low-cost adsorbent to remove from aqueous solutions one of the most widely used cationic dyes, namely violet crystal. The main objective was to study the kinetics and thermodynamics of the adsorption process.

2. Experimental

The absorbent material was obtained from dried raspberry leaves that were bought from StefMar, Ramnicu Valcea. The obtaining method of adsorbent did not involve the use of a chemical or heat treatment and was presented in detail in a previous study (Mosoarca *et al.*, 2022).

In each adsorption experiment, 0.1 g of adsorbent material was mixing with 50 mL of dye solution, with initial concentration of 50 mg/L. Kinetic studies were performed by varying the contact time from 2 to 50 minutes, while thermodynamic studies were conducted at temperatures of 281, 288, 297, 308 and 320 K. The influence of ionic strength on adsoption capacity was establish using different amount of NaCl as background electrolyte. The final concentration of crystal violet was quantified using a UV-VIS spectrophotometer Specord 200 PLUS, at 590 nm.

The adsorption capacity at equilibrium, (q_e), was determined with Eq. (1):

$$q_e = \frac{(C_0 - C_e) \cdot V}{m} \tag{1}$$

where: C_0 and C_e are the initial dye concentration and the dye concentration at equilibrium (mg/L); V is the solution volume (L) and m is the mass of adsorbent (g).

The pseudo-first-order, pseudo-second-order, Elovich, general order and Avrami kinetic models were tested to characterize the adsorption process. The higher value of determination coefficient (R^2) and the lowest values for sum of square error (SSE), and average relative error (ARE) were employed to find out the proper kinetic model. The equations that describe the tested kinetic models and error functions are follows:

Pseudo-first-order model: $$q_t = q_e(1 - \exp^{-k_1 \cdot t}) \tag{2}$$

Pseudo-second-order model: $$q_t = \frac{k_2 \cdot t \cdot q_e^2}{1 + k_2 \cdot t \cdot q_e} \tag{3}$$

Elovich model: $$q_t = \frac{1}{a}\ln(1 + a \cdot b \cdot t) \tag{4}$$

General order model: $$q_t = q_n - \frac{q_n}{[k_n \cdot (q_n)^{n-1} \cdot t \cdot (n-1) + 1]^{1/1-n}} \tag{5}$$

Avrami model: $$q_t = q_{AV}[1 - \exp(-k_{AV} \cdot t)^{n_{AV}}] \tag{6}$$

Determination coefficient $$R^2 = 1 - \frac{\sum_{i=1}^{n}\left(y_{i,exp} - y_{i,mod}\right)^2}{\sum_{i=1}^{n}\left(y_{i,exp} - \overline{y_{i,exp}}\right)^2} \tag{7}$$

Sum of square error $$SSE = \sum_{i=1}^{n}\left(y_{i,exp} - y_{i,mod}\right)^2 \tag{8}$$

Average relative error $$ARE = \frac{100}{n}\sum_{i=1}^{n}\left|\frac{y_{i,exp} - y_{i,mod}}{y_{i,mod}}\right| \qquad (9)$$

where: q_t is the crystal violet amount adsorbed at time t; k_1, k_2, k_n and k_{AV} are the rate constants of pseudo-first-order, pseudo-second-order, general order and Avrami kinetic models; q_e, q_n and q_{AV} are the theoretical values for the adsorption capacity; a is the desorption constant of Elovich model; b is the initial velocity; n is the general order exponent, n_{AV} is a fractional exponent, $y_{i,exp}$ is the experimental value; $y_{i,mod}$ is the modeled value; $\overline{y_{i,exp}}$ is the mean values and n is the total amount of information (Filho *et al.*, 2017; Dotto *et al.*, 2017; Netto *et al.*, 2021; Piccin *et al.*, 2017; Zaidi *et al.*, 2018).

The equations of specific thermodynamic parameters (standard Gibbs free energy change ΔG^0, standard enthalpy change ΔH^0 and standard entropy change ΔS^0) were calculated with the Eq. 10 and Eq.11:

$$\Delta G^0 = -RT\ln K_L \qquad (10)$$

$$\ln K_L = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{RT} \qquad (11)$$

where: R is the universal gas constant; K_L is the Langmuir constant and T is the absolute temperature (Filho *et al.*, 2017; Dotto *et al.*, 2017).

3. Results and Discussion

3.1. Influence of contact time on adsorption capacity

Fig. 1 show the influence of contact time on the adsorption capacity. The value of this parameter increases with increasing of contact time until equilibrium is reached at 30 minutes. Initially, the adsorption rate is quite fast because a large number of adsorption sites are available on the adsorbent surface for crystal violet dye retention. In time, these adsorption sites are gradually occupied and adsorption capacity increase slower until the equilibrium was established (Keereerak and Chinpa, 2020; Lim *et al.*, 2014; Shakoor and Nasar, 2018).

The equilibrium time and the experimental adsorption capacity obtained for crystal violet dye adsorption, using similar adsorbents, are presented in Table 1 and Table 2, respectively. The value of these two parameters is better that values obtained in the case of other adsorbent materials.

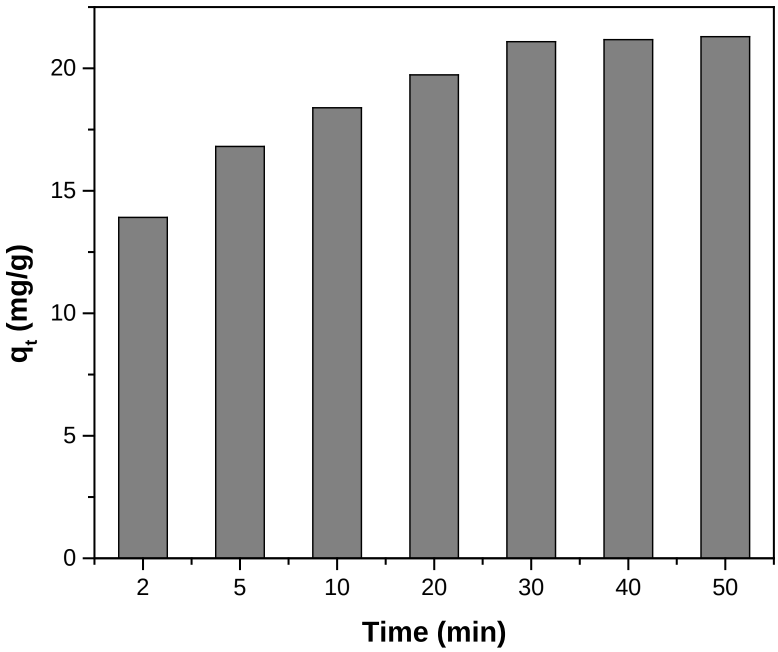

Fig. 1 − The influence of contact time on adsorption capacity.

Table 1

The equilibrium times obtained for crystal violet dye adsorption on similar adsorbents

Adsorbent material	Equilibrium times (min)	Reference
olive leaves powder	20	Elsherif *et al*., 2021
raspberry leaves powder	**30**	**This study**
cedar cone	30	Zamouche *et al*., 2020
pará chestnut husk	40	Georgin *et al*., 2018
grapefruit peel	60	Saeed *et al*., 2010
Moringa oleifera pod husk	60	Keereerak and Chinpa, 2020
almond shell	90	Loulidi *et al*., 2020
modified rice husk	90	Chakraborty *et al*., 2011
Ocotea puberula bark powder	120	Georgin *et al*., 2020
water hyacinth	120	Kulkarni *et al*., 2017
Terminalia arjuna sawdust	120	Shakoor and Nasar, 2018
Araticum seed powder	120	Franco *et al*., 2020
pinus bark powder	120	Ahmad, 2009
Punica granatum shell	120	Silveira *et al*., 2014
yeast-treated peat	150	Zehra *et al*., 2016
Eragrostis plana Nees	180	Filho *et al*., 2017
Artocarpus altilis skin	210	Lim *et al*., 2014

Table 2

The experimental adsorption capacity obtained for crystal violet dye adsorption on similar adsorbents, at 50 mg/L initial dye concentration

Adsorbent material	Experimental adsorption capacity (mg/g)	Reference
cedar cone	2.00	Zamouche *et al.*, 2020
Terminalia arjuna sawdust	7.97	Shakoor and Nasar, 2018
almond shell	8.50	Loulidi *et al.*, 2020
pinus bark powder	8.90	Ahmad, 2009
olive leaves powder	12.32	Elsherif *et al.*, 2021
yeast-treated peat	17.13	Zehra *et al.*, 2016
Punica granatum shell	19.30	Silveira *et al.*, 2014
raspberry leaves powder	**21.29**	**This study**
Eragrostis plana Nees	23.40	Filho *et al.*, 2017
modified rice husk	41.48	Chakraborty *et al.*, 2011
Moringa oleifera pod husk	73.85	Keereerak and Chinpa, 2020

3.2. Influence of temperature on adsorption capacity

The influence of temperature on adsorption capacity is illustrate in Fig. 2. The increase in temperature has a positive influence on the value of the adsorption capacity indicating that the adsorption process is endothermic in nature (Loulidi *et al.*, 2020; Saeed *et al.*, 2010).

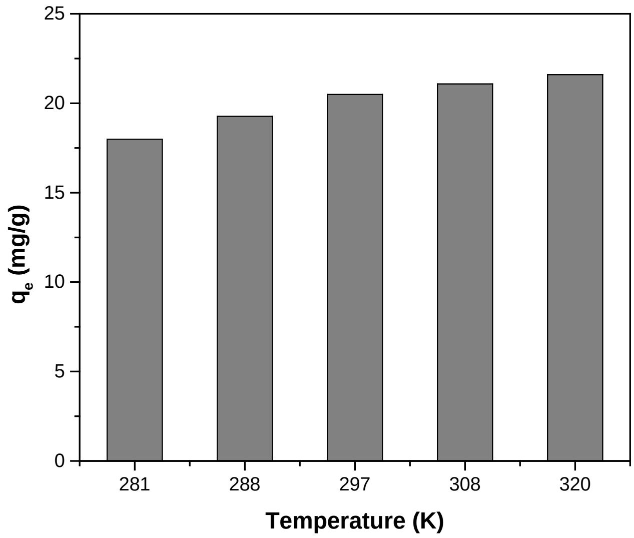

Fig. 2 − The influence of temperature on adsorption capacity.

Similar observation was reported in other studies regarding the crystal violet adsorption on adsorbent materials obtained from vegetal wastes such as: *Ocotea puberula* bark powder (Georgin *et al.*, 2020), almond shell (Loulidi *et al.*, 2020) and grapefruit peel (Saeed *et al.*, 2010).

When temperature increases, the solution viscosity is reduced and the mobility of dye molecules increase favouring the adsorption process (Ghazali *et al.*, 2018; Zehra *et al.*, 2016).

3.3. Influence of ionic strength on adsorption capacity

In addition to dyes, wastewater also contains other chemical compounds that increase ionic strength and can influence the adsorption process. The effect of ionic strength (simulated by the addition of NaCl) on adsorption capacity is shown in Fig. 3.

A competition appear between Na^+ ions and dye cations to occupy the available adsorption sites on the adsorbent surface and thus the dye adsorption process becomes more and more difficult (Aryee *et al.*, 2020; Chakraborty *et al.*, 2011). The unfavourable effect of increasing the ionic strength on the adsorption capacity has been highlighted by other researchers who have used similar adsorbents (Abu Elella *et al.*, 2019; Zaidi *et al.*, 2018).

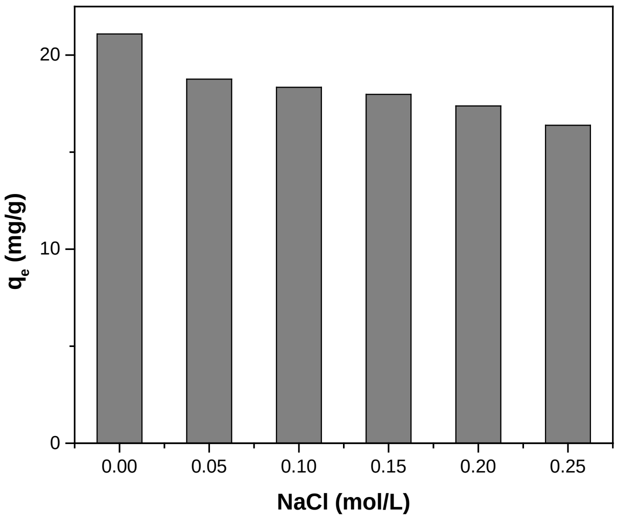

Fig. 3 − The influence of ionic strength on adsorption capacity

3.4. Kinetic modelling

The crystal violet adsorption kinetic was evaluated using pseudo-first order, pseudo-second order, Elovich, general order and Avrami models. Nonlinear fits of the tested kinetic models are presented in Fig. 4, while the models constants and the corresponding error functions are summarized in Table 3.

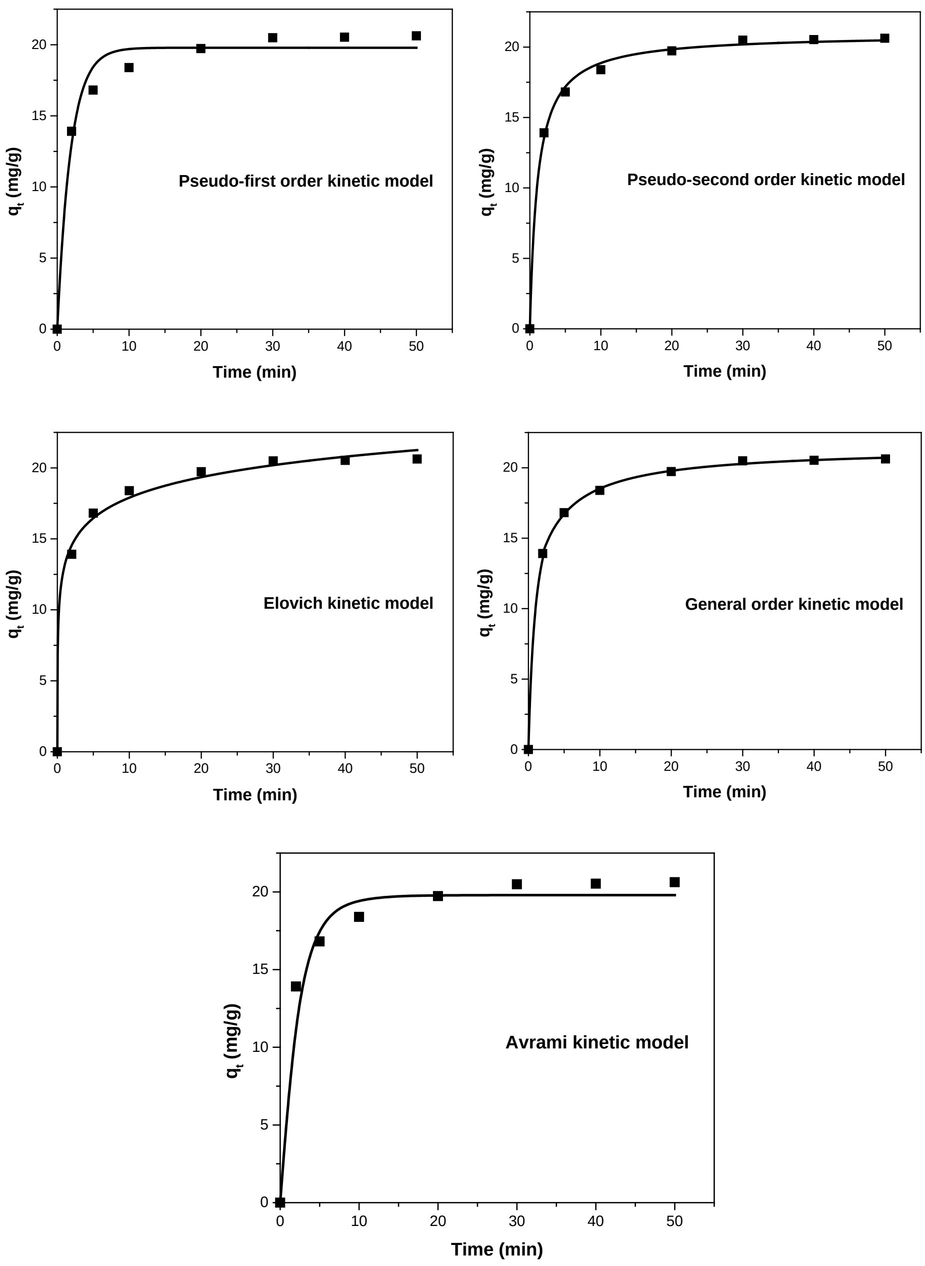

Fig. 4 − Tested kinetic models for crystal violet adsorption on raspberry leaves powder.

Table 3
The tested kinetic models constants and the corresponding error functions

Kinetic model	Parameter	Value
Pseudo-first order	k_1 (1/min)	0.535
	$q_{e,calc}$ (mg/g)	19.75
	R^2	0.9724
	SSE	7.81
	ARE (%)	18.27
Pseudo-second order	k_2 (1/min)	0.043
	$q_{e,calc}$ (g/mg·min)	20.92
	R^2	0.9981
	SSE	0.64
	ARE (%)	13.87
Elovich	a (g/mg)	0.452
	b (mg/g·min)	701.76
	R^2	0.9947
	SSE	1.98
	ARE (%)	14.64
General order	k_N (1/min·(g/mg)$^{n-1}$)	0.001
	q_n (mg/g)	21.42
	n	2.72
	R^2	0.9997
	SSE	0.08
	ARE (%)	0.39
Avrami	k_{AV} (1/min)	0.883
	q_{AV} (mg/g)	19.79
	n_{AV}	0.61
	R^2	0.9799
	SSE	6.88
	ARE (%)	16.76

The obtained data show that general order model best describes the adsorption process (the highest value of R^2 and the lower values for SSE and ARE). This model assumed that the order of an adsorption process should follow the same logic as in a chemical reaction, where the order is experimentally measured (Al-Azabi *et al.*, 2018; Ribas *et al.*, 2014).

3.5. Thermodynamic parameters

Table 4 presents the values of thermodynamic parameters ΔG^0, ΔH^0 and ΔS^0. The last two parameters were calculated from the slope and the intercept of ln K_L versus 1/T (Fig. 5).

Table 4
The thermodynamic parameters for crystal violet adsorption on raspberry leaves powder

ΔG^0 (kJ/mol)					ΔH^0 (kJ/mol)	ΔS^0 (J/mol·K)
281 K	288 K	297 K	308 K	320 K	1.63	13.32
-17.55	-18.30	-19.43	-20.61	-21.83		

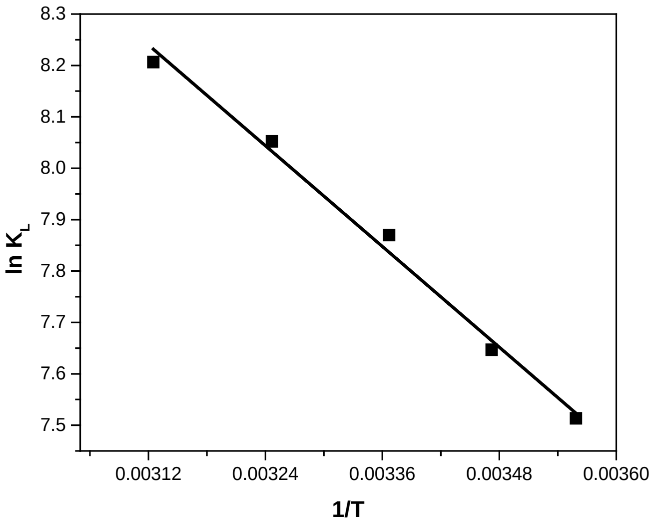

Fig. 5 – Plot of ln K_L vs. 1/T for the crystal violet adsorption on raspberry leaves powder.

The negative value of standard Gibbs free energy change and the positive value of standard enthalpy change and standard entropy change indicate that adsorption process is endothermic, spontaneous and favorable (Filho *et al.*, 2017; Kulkarni *et al.*, 2017; Loulidi *et al.*, 2020; Shakoor and Nasar, 2018; Zaidi *et al.*, 2018; Zehra *et al.*, 2016)

Similar results were obtained at crystal violet adsorption on water hyacinth (Kulkarni *et al.*, 2017), almond shell (Loulidi *et al.*, 2020), *Terminalia arjuna* sawdust (Shakoor and Nasar, 2018), *Artocarpus odoratissimus* leaf-based cellulose (Zaidi *et al.*, 2018) and yeast-treated peat (Zehra *et al.*, 2016).

When ΔG^0 values ranged between -20 (kJ/mol) to 0 (kJ/mol) and ΔH^0 is lower that 40 (kJ/mol) physical adsorption is implied in the process (Loulidi *et al.*, 2020; Singh *et al.*, 2020; Zehra *et al.*, 2016). The obtained value for ΔG^0 and ΔH^0 indicate that physisorption is the main mechanism implied in dye adsorption. van der Waals interaction plays an important role in physisorption fact suggested by the enthalpy change value lower than 20 (kJ/mol) (Jiang and Hu, 2019; Wakkel *et al.*, 2019).

4. Conclusions

During the crystal violet adsorption process on the raspberry leaves powder, the contact time and temperature positively influence the adsorption capacity, while ionic strength has a negative influence.

The kinetic study show that the equilibrium time and the experimental adsorption capacity obtained for crystal violet dye adsorption are better that values obtained in the case of other similar adsorbents. The general order kinetic model best describes the dye adsorption process.

The thermodynamic parameters indicate an endothermic, spontaneous and favourable process, physical adsorption being the main mechanism implied in crystal violet dye adsorption.

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ADSORBȚIA COLORANTULUI VIOLET CRISTAL PE PULBERE DE FRUZE DE ZMEUR – STUDII CINETICE ȘI TERMODINAMICE

(Rezumat)

În acest studiu, a fost investigată cinetica și termodinamica adsorbției colorantului violet cristal pe pulberea de frunze de zmeur. A fost studiat efectul timpului de contact, al temperaturii și al tăriei ionice. Valorile obținute pentru timpul de echilibru și capacitatea de adsorbție experimentală au fost mai bune decât valorile pentru alți adsorbanți similari obținuți din deșeuri vegetale. Studiul termodinamic a arătat că procesul de adsorbție este un proces endotermic, spontan și favorabil, iar adsorbția fizică este mecanismul principal implicat în reținerea colorantului pe suprafața adsorbantului.