

Optimization of crystal violet adsorption on common lilac tree leaf powder as natural adsorbent material

Giannin Mosoarca¹, Cosmin Vancea^{1*}, Simona Popa^{1*}, Sorina Boran¹

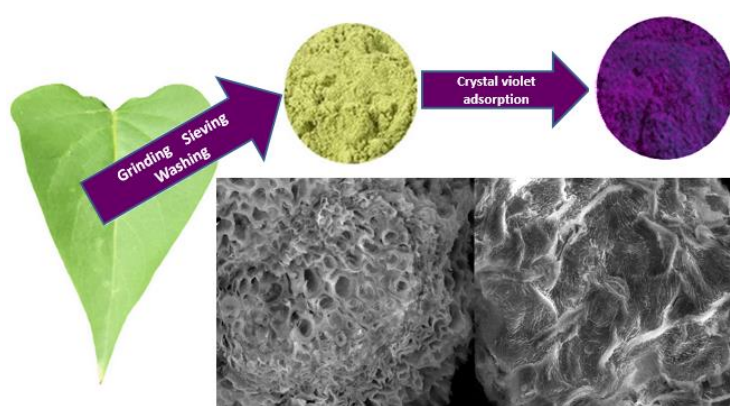
¹Politehnica University Timisoara, Faculty of Industrial Chemistry and Environmental Engineering,

Bd. V. Parvan No. 6, 300223, Timisoara, Romania

*Corresponding author:

E-mail: cosmin.vancea@upt.ro, tel.: +40256404194; simona.popa@upt.ro, tel.: +40256404212

GRAPHICAL ABSTRACT



ABSTRACT

In this study the efficiency of crystal violet dye removal from aqueous solutions using lilac tree leaf powder as adsorbent material was tested. After adsorbent surface analysis by SEM and FTIR technics, the influence of six controllable factor on dye adsorption process was assesses using a L27 Taguchi experimental design. The optimum adsorption conditions and the controllable factors significance were established by evaluation of signal-to-noise ratio (S/N). ANOVA analysis revealed the percentage contribution of each factor in crystal violet removal. Langmuir isotherm and pseudo-second order kinetic model characterized the process and the maximum adsorption capacity was higher than other similar adsorbents previously used in literature. The thermodynamic parameters reveal a favorable, spontaneous and endothermic adsorption based on physisorption mechanism.

Keywords: adsorption, crystal violet, isotherm, kinetic, Taguchi optimization, lilac tree leaf powder

1. Introduction

Dyes represent a wide range of chemicals that are widely used in various industries such as textiles, ceramics, food, cosmetics, pharmaceuticals, paper and printing (Aysu & Kucuk, 2015; Fabryanty et al., 2017; Pang et al., 2019; Zamouche et al., 2020). Some dyes are not dangerous to humans and can be used in the food industry while others can severely affect the environment and human health.

Crystal violet dye is used to color textiles and paper and also in veterinary medicine as a biological stain and as an animal drug. It must be removed from wastewater because it is non-biodegradable, persists in the environment for longer time and can have toxic, mutagenic and carcinogenic effects (Aysu & Kucuk, 2015; Chahinez et al., 2020; Chakraborty et al., 2011; Fabryanty et al., 2017; Zamouche et al., 2020; Franco et al., 2020; Shukat et al., 2017). Therefore, choosing an efficient and cheap treating method of these waters is essential to avoid pollution and protect the environment (Pang et al., 2020; Abbasi et al., 2020).

Several methods including coagulation-flocculation, catalytic reduction, photocatalytic degradation, microorganism degradation, membrane separation, membrane bioreactors, biological and electrochemical treatments have been used to removing dyes from aqueous solutions, but the relatively low efficiency of these methods, the need for special equipment and high energy consumption represent significant technical and economic disadvantages (Abbasi et al., 2020; Behboudi et al., 2021; Kosar Hashemi et al., 2021; Mosaviniya et al., 2019; Nouri et al., 2020; Tanzifi et al., 2020; Tanzifi et al., 2018a; Tanzifi et al., 2018b).

Adsorption is a successfully applied method for dyes removal from wastewater having many advantages: high efficiency, simplicity of operation, wide range of adsorbent material available and reduced costs (Chowdhury et al., 2013; Georgin et al., 2020; Rosly et al., 2021; Zehra et al., 2016). In recent years, the use of vegetable wastes (leaves, seeds, various peels and husk of fruit and agricultural products) as adsorbents material have become of particular interest, due to the fact that they are found in abundance, are ecological and have extremely low costs (Ali & Muhammad, 2008;

Bharathi & Ramesh, 2012; Chakraborty et al., 2012; El-Sayed, 2011; Georgin et al., 2018; Ghazali, 2018; Jain & Jayaram, 2010; Keereerak & Chinpa, 2020; Kulkarni et al., 2017; Lim et al., 2015; Loulidi et al., 2020; Muhammad et al., 2019; Nieva et al., 2020; Pavan et al., 2014; Rammel et al., 2011; Saeed et al., 2010; Saha et al., 2012; Silveira et al., 2014; Smitha et al., 2012; Suyamboo & Srikrishnaperumal, 2014; Wang et al., 2008).

Common lilac (*Syringa vulgaris* L.) is a large shrub or small tree, which grows up to a few meters tall, with a high ornamental value, much appreciated for the attractiveness of its wonderful and fragrant flowers. They can have shades ranging from pure white to purple intense. Originally from the Balkan Peninsula, it was naturalized, cultivated and used as an ornamental plant, in the temperate zone of Europe, Asia and North America. The bark, leaves, buds and flowers are used for phytotherapeutic purposes due to their anti-inflammatory, antimicrobial and antihypertensive properties (Ba & Kang, 2019; Jedrzejuk et al., 2016; Varga et al., 2019).

In this study the crystal violet dye was removed from the water by adsorption using as adsorbent material the common lilac leaf powder. One of the main objectives was to establish the optimum conditions of adsorption using the Taguchi method, following the influence of some controllable factors on the process efficiency and the signal/noise ratio (S/N). The contribution percentage of each parameter on dye removal process was determined by ANOVA analysis. Also, the accuracy of Taguchi model forecast was established. In addition, isotherms, kinetics and thermodynamics studies were conducted to evaluate the dye adsorption mechanism.

2. Materials and methods

In order to obtain the adsorbent material, mature leaves of common lilac tree were collected from Buzias resort, located in Timis County, Romania. The leaves powder obtaining process was described elsewhere (Mosoarca et al., 2020). The point of zero charge (PZC) for the adsorbent was 5.77 (Mosoarca et al., 2020). The characteristics of adsorbent material, before and after adsorption,

were examined by SEM analysis (Quanta FEG 250 scanning electron microscope at 1600x magnitude) and FTIR spectroscopy (Shimadzu Prestige-21 FTIR spectrophotometer).

The influence of various factors that influencing the adsorption process, on dye removal efficiency was studied, in batch system, according to the Taguchi approach (standard orthogonal array L27 as experimental design). These controllable factors and their levels are summarized in Table 1. The evaluation of the signal/noise ratio (S/N) was realized using the "larger is the better" option (Ghosh & Mondal, 2019; Fernandez-Lopez, 2019; Santra, 2014; Zolgharnein & Rastgordani, 2018). The percentage contribution of each factor to the dye removal efficiency was calculated with ANOVA analysis (Fernandez-Lopez, 2019; Rahmani et al., 2018; Zolgharnein & Rastgordani, 2018). The mathematical operations were performed with the Minitab 19 Software.

All experiments were performed in the batch system, at a constant stirring intensity provided by a shaker. In order to obtain the best conditions for dye removal, the effect of changes of various parameters that may affect the adsorption process was examined. NaOH (0.1 N) and HCl (0.1 N) solutions were used for pH adjustment. The dye concentration was determined with a UV-VIS spectrophotometer (Specord 200 PLUS) at 590 nm. Three independent replicates were performed for each adsorption test.

Langmuir, Freundlich, Temkin, Sips and Redlich-Peterson isotherms non-linear equations (Dotto et al., 2017; Fabryanty et al., 2017; Filho et al., 2017; Lim et al., 2015; Piccin et al., 2017; Zaidi et al., 2018) and pseudo-first order and pseudo-second order kinetic models nonlinear equations (Dotto et al., 2017; Fabryanty et al., 2017; Filho et al., 2017; Lim et al., 2015; Piccin et al., 2017; Zaidi et al., 2018) were tested to analyze the adsorption equilibrium and kinetics. Also, the intraparticle kinetic model was analyzed and discussed (Abbasi et al., 2020; Suyamboo and Srikrishnaperumal, 2014). In order to have a better precision in the analysis of the results, besides the determination coefficient (R^2), the error functions sum of square error (SSE), chi-square (χ^2) and average relative error (ARE) were also used (Piccin et al., 2017).

In order to characterize the process thermodynamically, the specific parameters (standard Gibbs free energy change, standard enthalpy change and standard entropy change) were computed at three temperatures (281, 294 and 308 K) according with other previous studies (Ali et al., 2015; Fabryanty et al., 2017; Filho et al., 2017; Liu & Xu, 2007; Liu & Liu, 2008; Liu, 2009).

Table 1. Controllable factors and their levels

Factor	Level 1	Level 2	Level 3
pH	2	6	10
Time (min)	5	15	30
Adsorbent dose (mg L ⁻¹)	0.5	1.5	2.5
Initial dye concentration (mg L ⁻¹)	50	150	250
Temperature (K)	281	294	308
Ionic strength (mol L ⁻¹)	0	0.1	0.2

3. Results and Discussion

3.1. Adsorbent characterization

The SEM image of the adsorbent material before adsorption (Figure 1a) shows an irregular surface with channels and many pores able to provide adsorption sites for dye molecules. After adsorption the SEM image is much different (Figure 1b), the surface has become smoother, and the pores are covered by dye molecules.

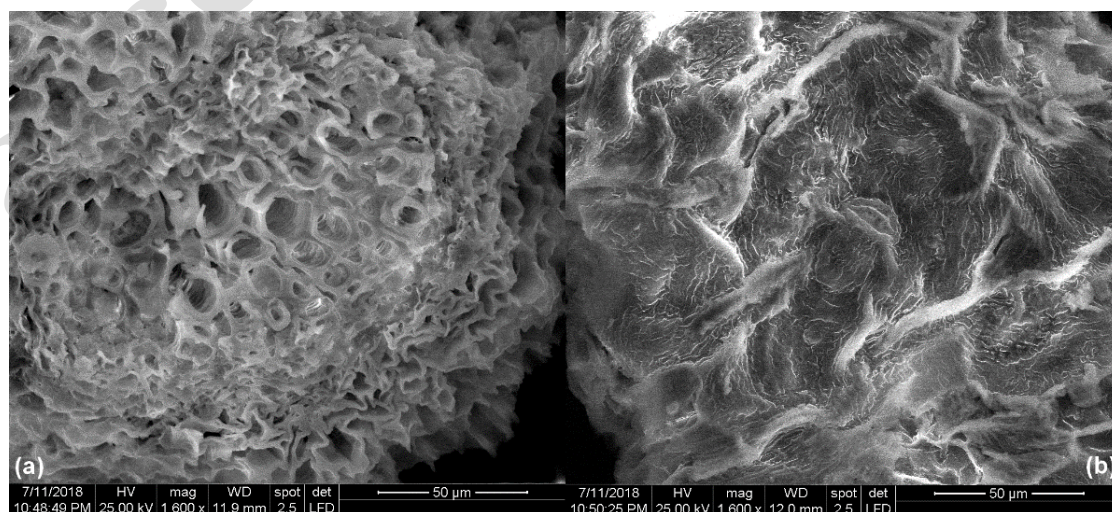


Figure 1. SEM image of adsorbent surface before (a) and after (b) crystal violet adsorption.

Following the FTIR analysis it was found that cellulose, hemicellulose and lignin are the main components of the adsorbent material (Mosoarca et al., 2020). The FTIR spectra of adsorbent before and after adsorption, shown in Figure 2, indicates specific peaks for main functional group existing in cellulose (3382 cm^{-1} , 2933 cm^{-1} , 1026 cm^{-1} , 609 cm^{-1}) (Ali et al., 2015; Garg et al., 2007; Lupoi et al., 2015; Miraboutalebi et al., 2017; Moosavinejad et al., 2019), hemicellulose (2933 cm^{-1} , 1647 cm^{-1} , 1255 cm^{-1} , 1026 cm^{-1}) (Lupoi et al., 2015; Moosavinejad et al., 2019; Saravanakumar et al., 2014; Senthamaraikannan et al., 2019; Shaharuddin et al., 2014; Weng et al., 2009) and lignin (3382 cm^{-1} , 2933 cm^{-1} , 1647 cm^{-1} , 1422 cm^{-1}) (Miraboutalebi et al., 2017; Moosavinejad et al., 2019; Senthamaraikannan et al., 2019; Shaharuddin et al., 2014; Chang et al., 2015; Sahoo et al., 2011).

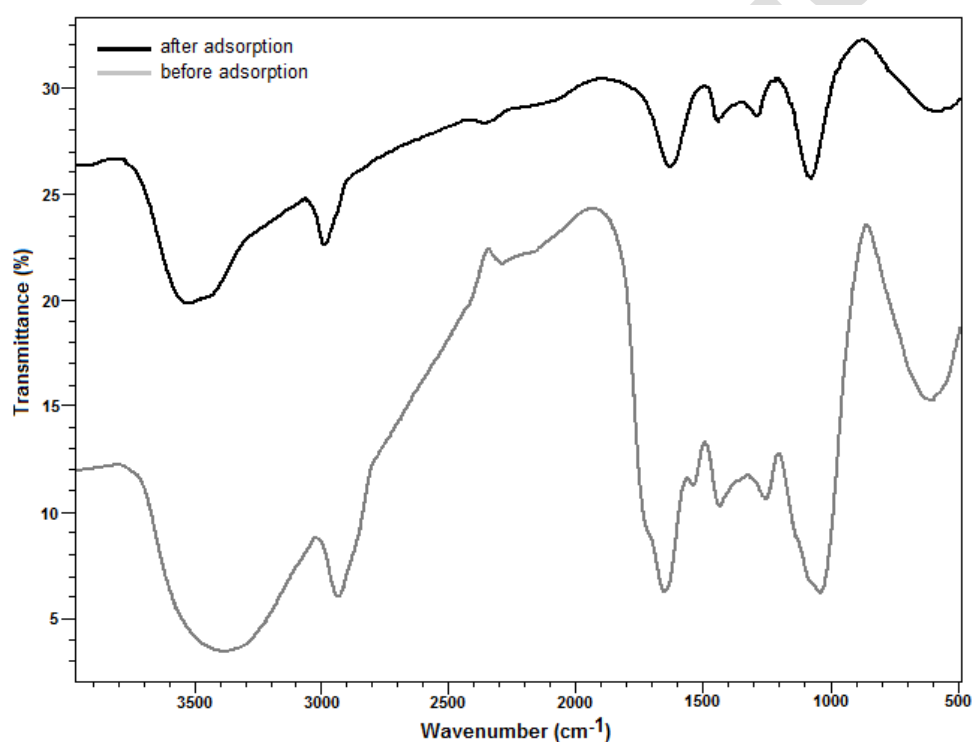


Figure 2. FTIR spectrum of common lilac tree leaves powder before and after crystal violet adsorption

3.2. Optimization parameters of adsorption process using Taguchi approach

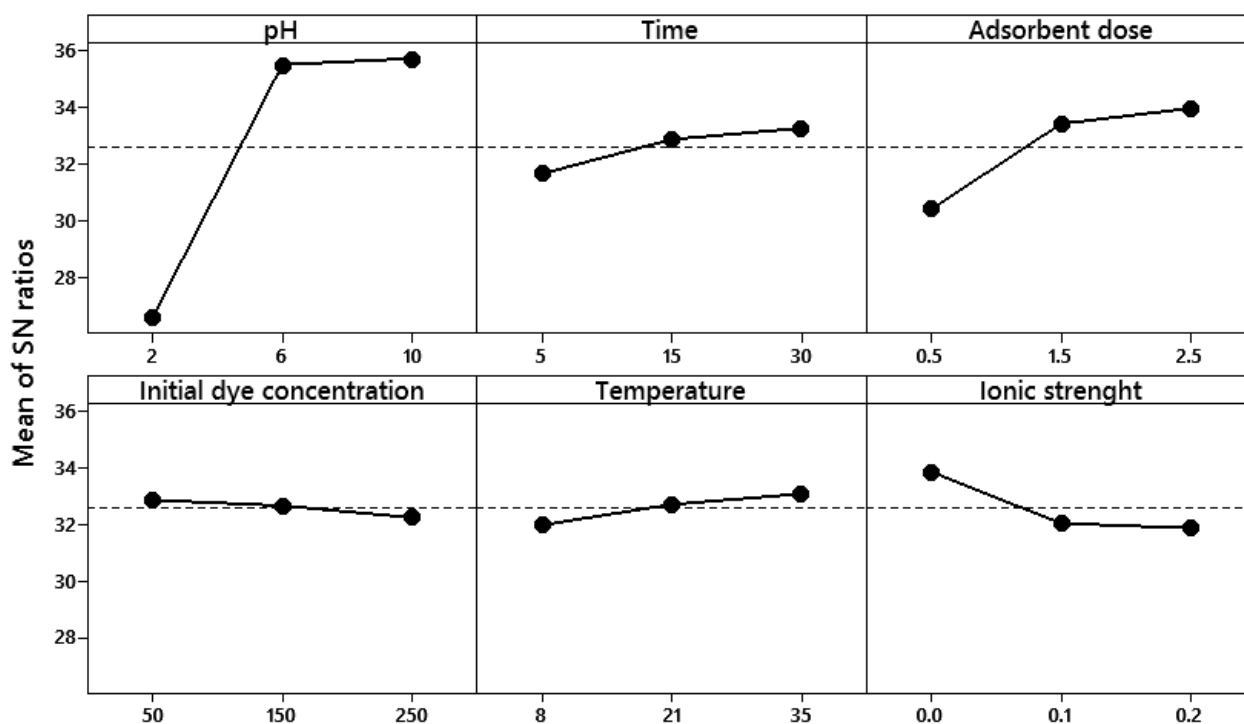
The optimum conditions for crystal violet removal from water were established using a L27 orthogonal array Taguchi design. The effect of 27 experiments, conducted according to the specified orthogonal array table, on the dye removal efficiency and the signal to noise (S/N) ratios is reveal in Table 2.

Table 2. Experimental layout of L27 orthogonal array and results obtained for removal efficiency and S/N ratios

pH	Time (min)	Adsorbent dose (g L ⁻¹)	Initial dye concentration (mg L ⁻¹)	Temperature (K)	Ionic strength (mol L ⁻¹)	Removal efficiency (%)	S/N ratio
2	5	0.5	50	281	0	16.69	24.44
2	5	0.5	50	294	0.1	14.73	23.36
2	5	0.5	50	308	0.2	14.81	23.41
2	15	1.5	150	281	0	26.27	28.38
2	15	1.5	150	294	0.1	23.19	27.30
2	15	1.5	150	308	0.2	23.85	27.54
2	30	2.5	250	281	0	27.90	28.91
2	30	2.5	250	294	0.1	24.62	27.82
2	30	2.5	250	308	0.2	25.33	28.07
6	5	1.5	250	281	0.1	49.30	33.85
6	5	1.5	250	294	0.2	52.70	34.43
6	5	1.5	250	308	0	69.02	36.77
6	15	2.5	50	281	0.1	64.78	36.22
6	15	2.5	50	294	0.2	69.24	36.80
6	15	2.5	50	308	0	90.70	39.15
6	30	0.5	150	281	0.1	44.05	32.87
6	30	0.5	150	294	0.2	47.08	33.45
6	30	0.5	150	308	0	61.67	35.80
10	5	2.5	150	281	0.2	55.57	34.89
10	5	2.5	150	294	0	75.63	37.57
10	5	2.5	150	308	0.1	64.25	36.15
10	15	0.5	250	281	0.2	40.41	32.12
10	15	0.5	250	294	0	54.99	34.80
10	15	0.5	250	308	0.1	46.72	33.39
10	30	1.5	50	281	0.2	64.20	36.15
10	30	1.5	50	294	0	87.37	38.82
10	30	1.5	50	308	0.1	74.23	37.41

Depending on the combination of the controllable factors the removal efficiency ranged from 14.73 to 90.70 %. The value of S/N ratio increase with the increasing of pH, time, adsorbent dose and temperature, while the increase of the initial dye concentration and ionic strength has a negative effect on this parameter (Figure 3). Also, Figure 3 reveals that the strongest variation in S/N ratio occurred with pH followed by adsorbent dose, ionic strength, contact time, temperature and initial dye concentration. Therefore, the controllable factor with the greatest influence was the pH and the factor with the least influence was the initial dye concentration. The optimum conditions for dye removal, indicate by the maximum mean of S/N ratio were: pH = 10, contact time 30 minutes, adsorbent dose 2.5 (g L⁻¹), initial dye concentration 50 (mg L⁻¹), temperature 308 K and ionic strength 0.0 (mol L⁻¹).

Main Effects Plot for SN ratios
Data Means



Signal-to-noise: Larger is better

Figure 3. Signal-to-noise main effect plots for each controllable factors

The results of the analysis of variance (ANOVA), carried out to assess the contribution of each controllable factor to the process, are presented in Figure 4. They confirm the same order of influence for the controllable factors, the pH having the highest contribution percentage and the initial concentration the lowest percentage.

In order to establish the accuracy of Taguchi model forecast, the predicted values of dye removal efficiency were correlated with the experimental ones. Figure 5 indicate a very good correlation between the values and confirm the Taguchi method precision.

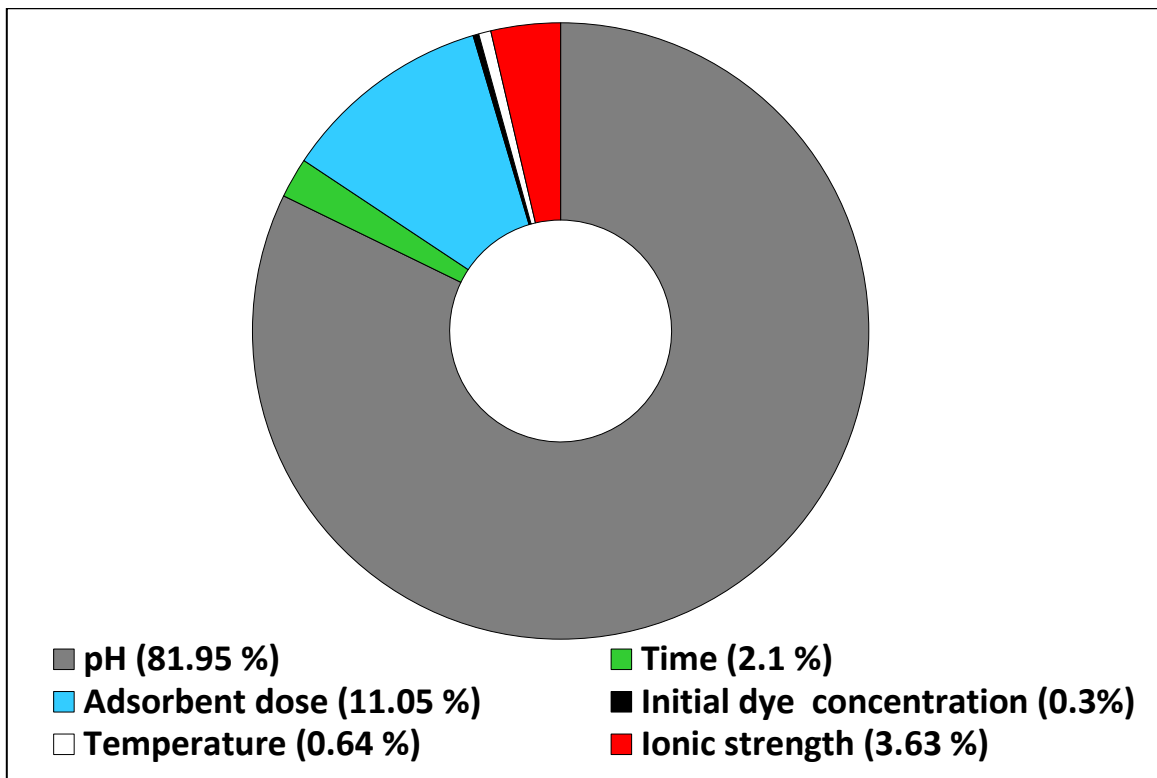


Figure 4. Contribution percentage of controllable factor influence on dye removal establish by ANOVA analysis

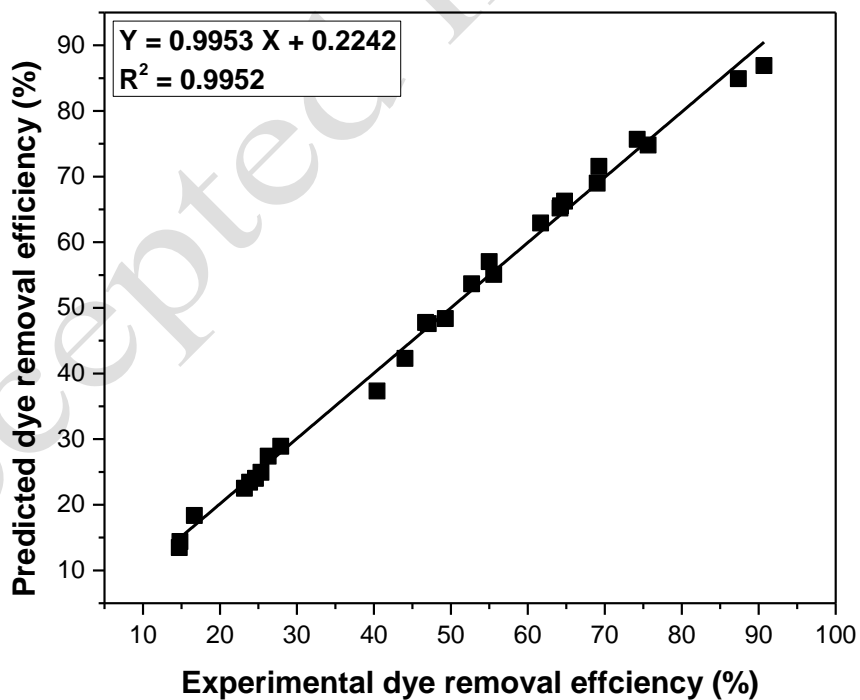


Figure 5. Comparison of experimental and predicted dye removal efficiency

3.3. Equilibrium and kinetic studies

Langmuir, Freundlich, Temkin, Sips and Redlich-Peterson adsorption isotherms (Figure 6) were used to study the interactions between the dye and the adsorbent material as well as to obtain information about the adsorption mechanism (Alizadeh et al., 2017; Aysu & Kucuk, 2015; Laskar & Kumar, 2018; Lim et al., 2015; Mirza & Ahmad, 2020; Zaidi et al., 2018).

The values for determination coefficient (R^2), sum of square error (SSE), chi-square (χ^2) and average relative error (ARE) are summarized in Table 3. The data suggest that the adsorption process is best described by the Langmuir isotherm. In addition, the value for the R_L parameter (0.26) was calculated and this indicated that the adsorption is favorable.

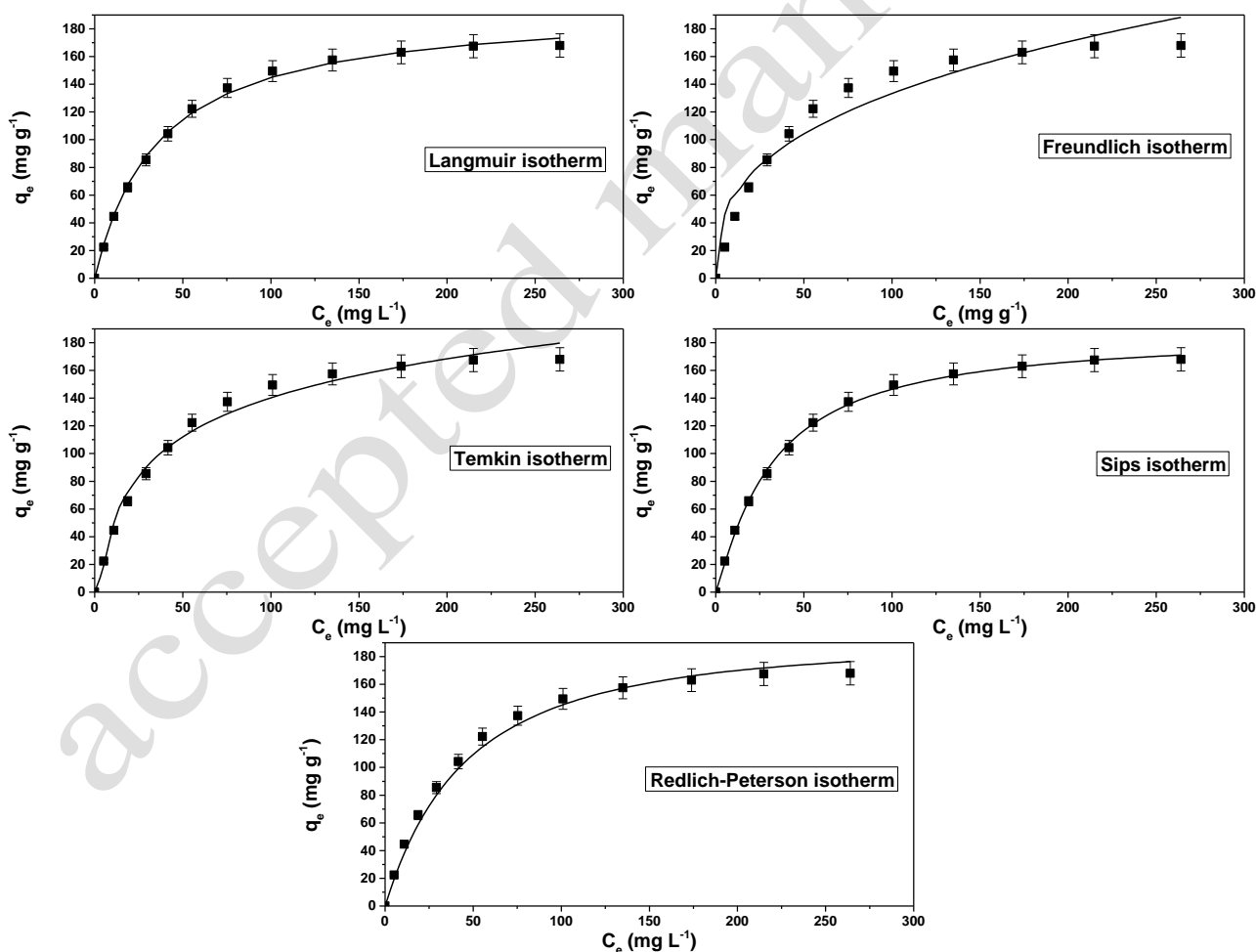


Figure 6. The tested adsorption isotherms (non-linear forms) for the crystal violet adsorption on common lilac tree leaves powder

Table 3. Adsorption isotherms models constants and the corresponding error functions

Isotherm model	Parameters	Value
Langmuir non-linear	K_L (L mg ⁻¹)	0.027 ± 0.001
	q_{max} (mg g ⁻¹)	196.7 ± 4.21
	R^2	0.9984
	χ^2	0.55
	SSE	48.01
	ARE (%)	2.16
Freundlich non-linear	K_f (mg g ⁻¹)(L ^{1/n} mg ^{-1/n})	9.07 ± 1.29
	1/n	0.66 ± 0.03
	R^2	0.9256
	χ^2	28.82
	SSE	2215
	ARE (%)	13.02
Temkin non-linear	K_T (L mg ⁻¹)	0.313 ± 0.001
	b (kJ g ⁻¹)	60.06 ± 2.14
	R^2	0.9838
	χ^2	4.40
	SSE	470
	ARE (%)	6.03
Sips non-linear	Q_{sat} (mg g ⁻¹)	186.9 ± 5.1
	K_S (L mg ⁻¹)	0.019 ± 0.002
	n	1.13 ± 0.03
	R^2	0.9981
	χ^2	0.89
	SSE	95.12
	ARE (%)	2.69
Redlich-Peterson non-linear	K_{RP} (L g ⁻¹)	4.12 ± 0.03
	a_{RP} (L mg ⁻¹)	0.014 ± 0.001
	β_{RP}	1.06 ± 0.02
	R^2	0.9947
	χ^2	4.46
	SSE	365
	ARE (%)	6.59

Table 4 compares the maximum adsorption capacities for different low-cost adsorbents used for crystal violet adsorption. The value of the maximum adsorption capacity obtained for common lilac tree leaves powder, 196.75 (mg g⁻¹), is higher than other similar adsorbents previously reported in the literature.

Table 4. The maximum adsorption capacities for different low-cost adsorbents used for crystal violet adsorption

Adsorbent material	Adsorption capacity (mg g ⁻¹)	Reference
<i>Calotropis procera</i> leaf	4.14	(Ali & Muhammad, 2008)
Pineapple crown leaves	6.49	(Nieva et al., 2020)
Corn stalk	9.64	(Muhammad et al., 2019)
Almond shells	12.20	(Loulidi et al., 2020)
Peel of Cucumis sativa fruit	34.24	(Smitha et al., 2012)
Date palm leaves powder	37.73	(Ghazali et al., 2018)
Rice bran	41.68	(Wang et al., 2008)
Jackfruit leaf powder	43.39	(Saha et al., 2012)
Punica granatum shell	50.21	(Silveira et al., 2014)
Eragrostis plana nees	60.10	(Filho et al., 2017)

Laminaria japonica	66.64	(Wang et al., 2008)
Wheat bran	69.15	(Wang et al., 2008)
Pineapple leaf powder	78.22	(Chakraborty et al., 2012)
Palm kernel fiber	78.90	(El-Sayed, 2011)
Pará chestnut husk	83.60	(Georgin et al., 2018)
Papaya seeds	85.99	(Pavan et al., 2014)
Wood apple shell	129.87	(Jain & Jayaram, 2010)
Breadfruit skin	145.80	(Lim et al., 2015)
Moringa oleifera pod husk	156.25	(Keereerak & Chinpa, 2020)
Chaetophora elegans alga	158.70	(Rammel et al., 2011)
Common lilac tree leaves powder	196.75	This study
Grapefruit peel	254.16	(Saeed et al., 2010)
Water hyacinth root powder	322.58	(Kulkarni et al., 2017)

The pseudo-first-order, pseudo-second-order and intra-particle diffusion models were used to study the adsorption kinetics (Figure 7 and Figure 8). The kinetic parameters of these models can provide useful information needed to predict the adsorption rate and design the adsorption processes (Abbasi et al., 2020; Alizadeh et al., 2017; Laskar & Kumar, 2018; Patil et al., 2020). Kinetic constants and the corresponding error functions presented in Table 5 indicate that the pseudo-second-order model best characterizes the process. The similar conclusion has been reported in other articles concerning the dyes adsorption on low-cost adsorbent materials (Chakraborty et al., 2012; Filho et al., 2017; Loulidi et al., 2020; Pavan et al., 2014; Smitha et al., 2012; Zamouche et al., 2020).

The intra-particle kinetic model includes three steps. At the beginning of the process, the adsorption proceeds at high speed, the dye molecules immediately entering the large pores on the surface of the adsorbent. In step 2 the intra-particles diffusion proceeds with lower speed, the dye molecules entering the small pores of the adsorbent and then, in step 3, the equilibrium is reached. The present finding indicate that intra-particle diffusion intervene in process but it is not the only one mechanism involved in adsorption (Abbasi et al., 2020; Suyamboo and Srikrishnaperumal, 2014).

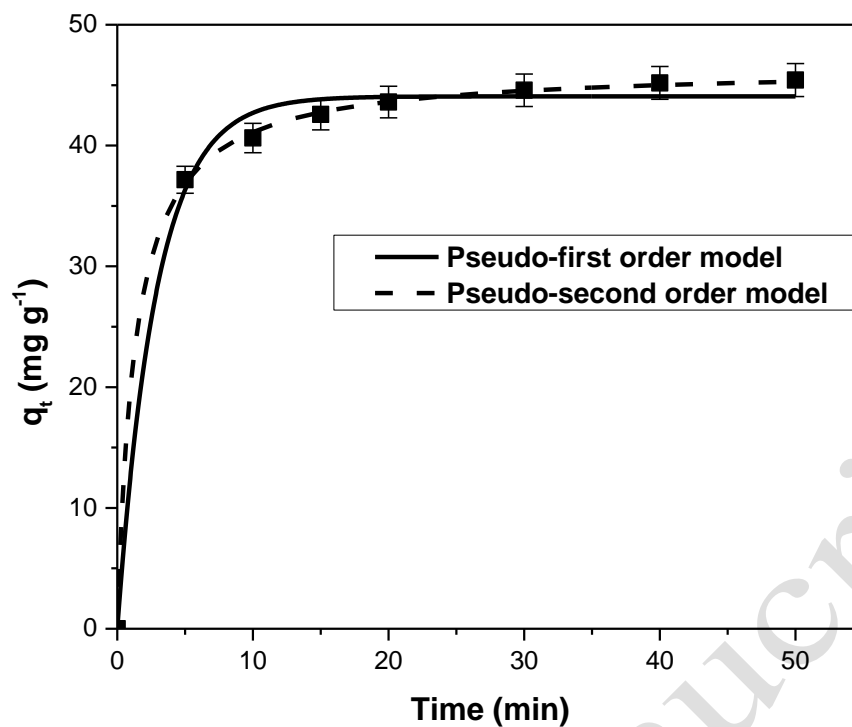


Figure 7. Pseudo-first-order and pseudo-second-order kinetic models (non-linear forms) for the crystal violet adsorption on common lilac tree leaves powder

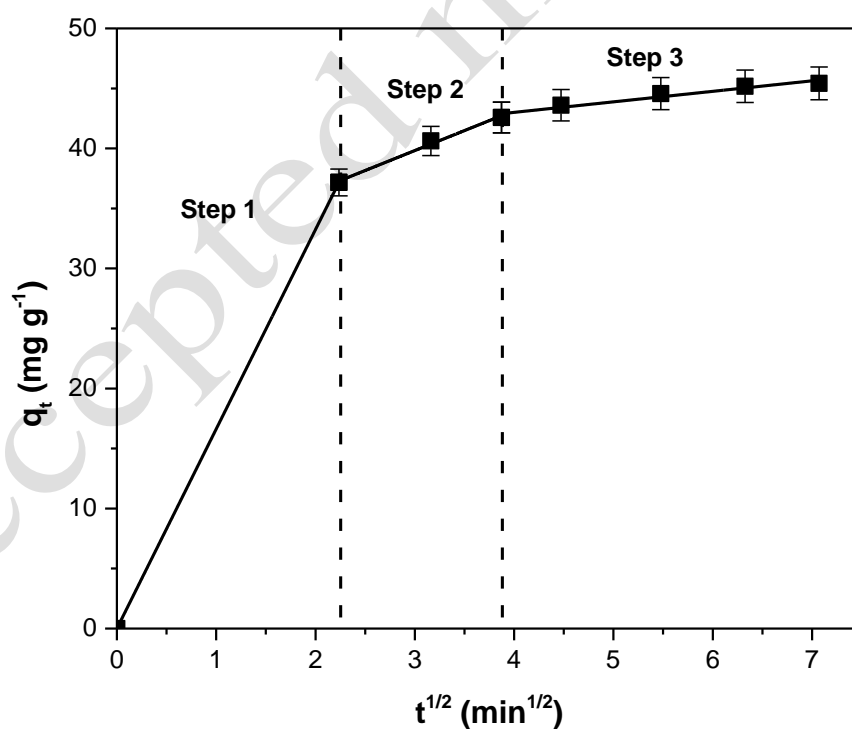


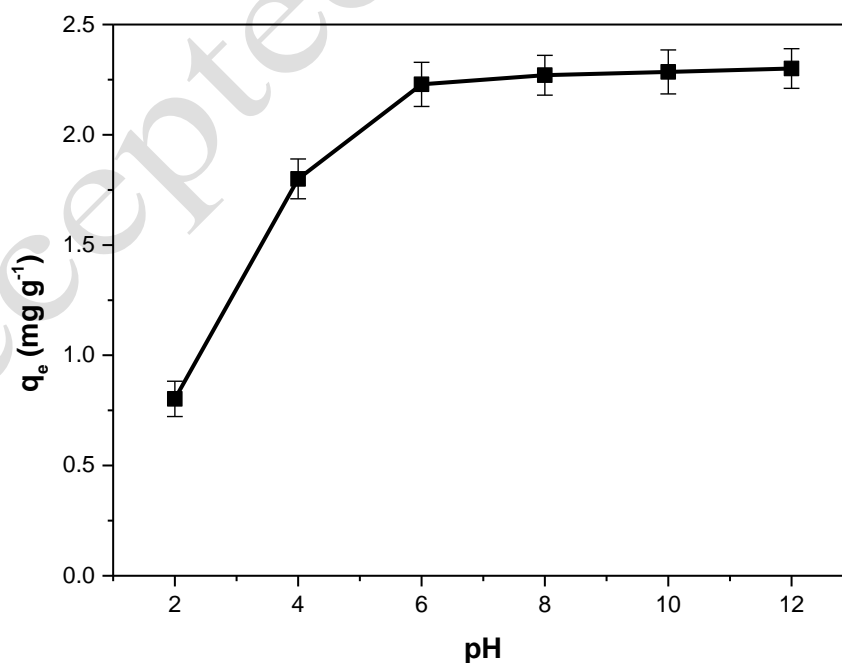
Figure 8. Fitted three-step intra-particle kinetic model for the crystal violet adsorption on common lilac tree leaves powder

Table 5. Kinetic models constants and the corresponding error functions.

Kinetic model	Parameters	Value
Pseudo-first-order non-linear	k_1 (min^{-1})	0.39 ± 0.03
	$q_{e,\text{calc}}$ (mg g^{-1})	42.68 ± 0.57
	R^2	0.9925
	χ^2	0.46
	SSE	19.92
	ARE (%)	15.35
Pseudo-second-order non-linear	k_2 ($\text{g mg}^{-1} \text{min}^{-1}$)	0.017 ± 0.007
	$q_{e,\text{calc}}$ (mg g^{-1})	46.23 ± 0.18
	R^2	0.9997
	χ^2	0.01
	SSE	0.53
	ARE (%)	13.00

3.4. Influence of initial solution pH on adsorption capacity

The effect of initial pH of solution on the adsorption capacity is shown in Figure 9. The highest values of this parameter were obtained at $\text{pH} > 6$. Taking into account that pH_{PZC} of adsorbent materials is 5.77 (Mosoarca et al., 2020), the better results can be explained by the fact that at higher pH values than pH_{PZC} adsorbent surface is negative and binding of dye cations is favorable due to the electrostatic attractions (Fabryanty et al., 2017; Lim et al., 2015; Rahmani et al., 2018; Zolgharnein and Rastgordani, 2018).

**Figure 9.** The effect of initial solution pH on adsorption capacity

3.5. Thermodynamic study

Thermodynamic parameters were calculated from the slope and the intercept of $\ln K_L$ versus $1/T$ plot (Figure 10), where: K_L represents the Langmuir constant and T represents the absolute temperature (Ali et al., 2015; Fabryanty et al., 2017; Filho et al., 2017; Liu & Xu, 2007; Liu & Liu, 2008; Liu, 2009). The obtained values for these parameters are presented in Table 6. The positive value of ΔH^0 and the negative value of ΔG^0 indicates that the dye adsorption on the adsorbent material is a favorable, spontaneous and endothermic process. In addition, the positive value of ΔS^0 reveal the affinity of the adsorbent for the crystal violet dye (Aysu & Kucuk, 2015; Fabryanty et al., 2017; Loulidi et al., 2020). The same observations have been reported in other studies in which similar adsorbents were used to remove crystal violet dye from water (Filho et al., 2017; Kulkarni et al., 2017; Loulidi et al., 2020; Zaidi et al., 2018). The value of ΔH^0 less than 20 (kJ mol^{-1}) shows that the dye removal mechanism is mainly physical adsorption and van der Waals interaction is involved in the physisorption (Jiang & Hu, 2019; Loulidi et al., 2020; Zehra et al., 2016).

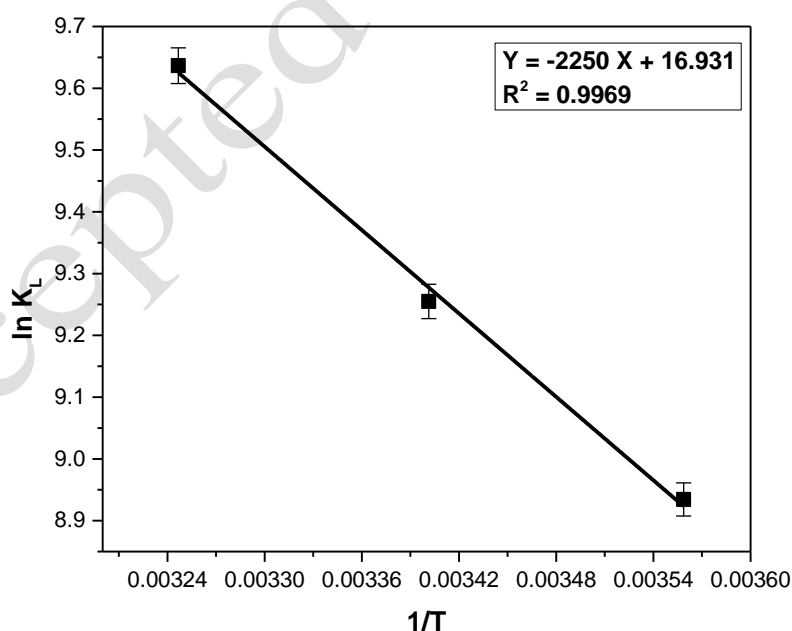


Figure 10. Plot of $\ln K_L$ vs. $1/T$ for the crystal violet adsorption on common lilac tree leaves powder

Table 6. Thermodynamic parameters for the crystal violet adsorption on common lilac tree leaves powder

ΔG (kJ mol ⁻¹)			ΔH (kJ mol ⁻¹)	ΔS (J mol ⁻¹)
281 K	294 K	308 K		
-20.87	-22.62	-24.67	2.25	16.93

4. Conclusions

The lilac tree leaf powder has a high efficiency on the crystal violet dye removal from aqueous solution. Using a Taguchi standard orthogonal array L27 as experimental design the optimum condition of adsorption were establish and it was concluded that the controllable factor with the greatest influence on the process was the pH and the factor with the least influence was the initial dye concentration. ANOVA analysis confirmed the results obtained by the Taguchi method. The correlation between the predicted values of dye removal efficiency and the experimental ones indicated a high accuracy of the Taguchi experimental design. The equilibrium and kinetics adsorption data can be best fitted by Langmuir isotherm and pseudo-second order kinetic model, respectively. The adsorbent material had a higher value of maximum adsorption capacity than other similar adsorbents used for crystal violet adsorption. The thermodynamic parameters indicate is a favorable, spontaneous and endothermic physisorption process.

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