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ADSORPTION PROCESS OF CYAN PRINTING DYE ONTO ACTIVATED CARBON: OPTIMIZATION AND KINETIC STUDIES

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Abstract: *The potential use of commercial activated carbon for the removal of water-based phthalocyanine printing dye (Cyan) form aqueous solution was investigated. Screening and optimization of four variables (initial dye concentrations, adsorbent mass, pH of the aqueous solution and reaction time) influencing the adsorption process are performed using a new design of experiments methodology, the definitive screening design (DSD). Maximum removal efficiency of 88.5% for Cyan dye was achieved under following conditions: 32.8 mgL^{-1} of the initial dye concentration, 0.09 g of the adsorbent mass, pH 6 and 20 minutes of the reaction time. The kinetic adsorption modelling followed the Elovich model, indicating monolayer chemisorption process on a heterogeneous adsorbent surface, with possible van der Waals interactions, hydrogen and halogen bonding, as an integral part of adsorption mechanisms of printing dye on activated carbon.*

Key words: Cyan dye, activated carbon, adsorption, optimization, kinetic models, mechanism

1. INTRODUCTION

Synthetic organic dyes are becoming increasingly important in most industries, including paper, plastics, pharmaceuticals, cosmetics and textiles, due to their ease of synthesis and speed of production, their stability and variety of colours (Boulika et al., 2023; Nabih et al., 2023; Rožić et al., 2023; Adamović et al., 2022). Organic dyes owe their colour to the content of certain chromophores, which are mainly acidic and basic functional groups. These dyes are carcinogenic, mutagenic and highly toxic to humans and aquatic organisms. They can also reduce light transmission and interfere with photosynthesis, which can disrupt the ecological balance, and they can break down into toxic, mutagenic or carcinogenic compounds that damage the aquatic body (Raji et al., 2023; Reddy et al., 2023). Due to their high stability and low biodegradability, wastewater containing such dyes can cause various problems leading to visual and chemical pollution. Therefore, it is important to remove these dyes from wastewater or treat it in a way that minimizes its impact on the environment (Boulika et al., 2023).

To resolve these problems, different conventional techniques for water treatment have been proposed, including coagulation-flocculation (Ihaddaden et al., 2022), electrochemical degradation (Abo-Ayad et al., 2024), photocatalysis (Zhao et al., 2024), biodegradation (Das et al., 2021), membrane filtration (Hou et al., 2023) and chemical oxidation (Neto et al., 2024). Some of these techniques, although effective, are restricted by secondary waste, expense, or complexity (Suhaimi et al., 2022). Among them, adsorption technology is widely recognized as an important way to remove pollutants due to several technical merits: ease of implementation, high efficiency, low material and operating costs, high performance and the ability to treat different dye concentrations (Raji et al., 2023; Suhaimi et al., 2022). On the other hand, the adsorption technique depends on the performance of a particular adsorbent for different types of dyes (Reddy et al., 2023).

Currently, different type of adsorbents like: composites (Cui et al., 2024), nanoparticles (Ali et al., 2024), polymers and resins (Elabboudi et al., 2023), clays and minerals (Cigeroglu et al., 2024), biochar (Mancuso et al., 2024), and biosorbents (Elboughdiri et al., 2024) are used for removal of dyes. Among the various adsorbents, activated carbon is emerged as the most commonly and widely utilized adsorbent due to its outstanding properties like adsorption capacity, microporous, high surface area, abundant functional groups, thermal and radiation stability (Reddy et al., 2023). In view of that, activated carbon can be used as a commercial product, or can be prepared from ubiquitous, inexpensive, and renewable precursors, such as rice husk (Zhang et al., 2023), moringa oleifera seed husk (Escobar et al., 2021), date palm waste (Arangadi et al., 2022), grass (Tu et al., 2021), pineapple leaves (Arumugasamy et al., 2022), pomegranate peel (Saadi et al., 2022), banana peel (Tripathy et al., 2021), and coconut shells (Widanarto et al., 2022).

The objective of this study is to investigate the effectiveness of commercial activated carbon for the removal of Cyan printing dye, by screening and optimizing the factors influencing the adsorption process, in order to obtain a maximum removal rate. Furthermore, the adsorption kinetics and mechanism are examined by using the pseudo-first and pseudo-second order models, as well as the Elovich model.

2. MATERIALS AND METHODS

2.1 Materials and chemicals

The used chemicals and reagents were: synthetic solution of Cyan flexographic dye (Flint group), activated carbon (AC, Norit Row 0.8 Supra), sodium hydroxide (>98.8% POCH, Poland), hydrochloric acid (>96%, J.T. Baker - Fischer Scientific, USA), deionized water. Figure 1 shows the molecular structure of Cyan dye (Phthalocyanine type, C.I. PB15:3, CAS number: 147-14-8, λ_{max} = 636 nm) and Table 1 presents certain physico-chemical properties of activated carbon.

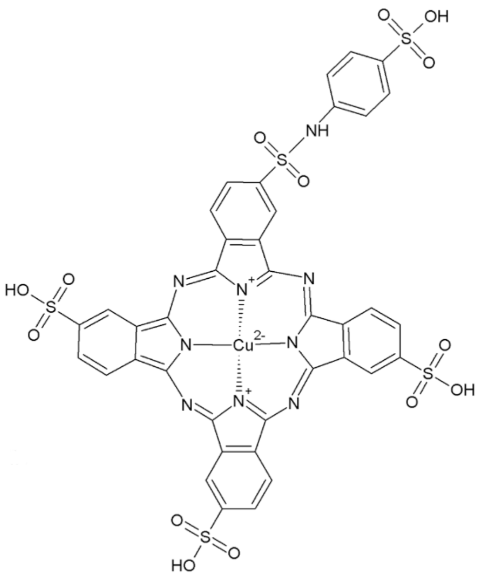

Figure 1: Chemical structure of Cyan dye

Table 1: Physico-chemical properties of activated carbon

Characteristic	Value
Iodine number (mgg^{-1})	1050
Specific surface area (m^2g^{-1})	1150
Ash content (%)	7
Moisture content (%)	2
pH_{PZC}	4.6
Density (kgm^{-3})	390

2.2 Experimental design

In order to optimize the experiment and to identify significant variables and eliminate irrelevant variables, a new generation statistical method, Definitive Screening Design (DSD), was used (Mohamed et al., 2017). DSD provides a compelling balance between the number of variables to be modelled and the number of experiments to be performed, with an ability to detect main, interaction, and quadratic effects without the need for effect aliasing (Boulika et al., 2023). The influence of four process parameters was studied: initial dye concentration (20, 100 and 180 mgL^{-1}), adsorbent mass (0.01, 0.055 and 0.1 g), pH (2, 6 and 10) and reaction time (1, 30.5 and 60 min). The selected software JMP 13 (SAS Institute, USA) was used for the experimental design and the complete statistical processing of the obtained results. The optimization of the adsorption treatment using the statistical DSD method with the selection of process parameters combination enables the achievement of the highest efficiency of adsorption process.

2.3 Adsorption experiments

Synthetic solution of Cyan dye was obtained from one flexographic printing facility in Novi Sad. A certain mass of activated carbon (0.01 - 0.1 g) was added to a 50 mL of sample volume of synthetic dye solution (concentration of 20 - 180 mgL^{-1}) and the pH value of the reaction medium was adjusted (2 - 10) with the addition of HCl (0.1 M) or NaOH (0.1 M) solution. The experiments were carried batchwise at room temperature (23° C), at a stirring speed of 240 rpm (IKA® Orbital shaker KS 130 Digital) with the estimated time (1 - 60 min), to ensure good dispersion of the solid particles of the activated carbon. After mixing, the samples were filtered through a cellulose acetate membrane filter with a porosity of 0.45 mm, and the absorbance of the solution was determined using a UV/VIS spectrophotometer (Genesys 10S, Thermo Fisher) (Tubić et al., 2023). The residual dye concentration was established immediately by measuring the absorbance of the aqueous solutions at 636 nm and the efficiency of adsorption treatment was calculated according to Equation (1):

$$E(\%) = A_0 - A/A_0 * 100 \tag{1}$$

where: A_0 is the initial absorbance of the aqueous solution sample before adsorption treatment and A is the absorbance of the aqueous solution sample after adsorption treatment.

2.4 Kinetic experiments

The adsorption kinetics and the mechanism of adsorption of Cyan dye on activated carbon were studied in a batch mode at room temperature (25°C) in laboratory conditions. In these experiments, the volume of the synthetic aqueous matrix was 50 mL, and the initial dye concentration was 100 mgL^{-1}. A dye concentration of 100 mgL^{-1} was selected for the adsorption kinetic experiments due to the Cyan dye concentration detected in printing wastewater samples, which was 94.16 mgL^{-1} (Tubić et al., 2023). A mass of 0.1 g of activated carbon was chosen based on the literature data (Raji et al., 2023). Complete suspension mixing was achieved using a digital mixer IKA® Orbital shaker KS 130 Digital) at 150 rpm at room temperature (25°C). The change in the concentration of the printing dyes after defined contact periods with the activated carbon (10 min, 30 min, 1, 2, 3, 5, 24 h) and filtration through a 0.45 μm cellulose acetate membrane filter was monitored using a UV/VIS spectrophotometer (Genesys 10S, Thermo Fisher).
The adsorption kinetic model could determine the relationship between adsorption time and adsorption capacity and could be used to analyze the adsorption mechanism of printing dye on activated carbon. The pseudo-first-order kinetics (Equation 2) and pseudo-second-order kinetics (Equation 3) and the Elovich model (Equation 4) were used in this study (Boulika et al., 2023; Li et al., 2021).

$$log(q_e - q_t) = log q_e - \frac{K_1}{2.303} t \tag{2}$$

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{t}{q_e} \tag{3}$$

$$q_t = \frac{1}{\beta} ln(\alpha\beta) + \frac{1}{\beta} ln\,(t) \tag{4}$$

where: q_e is the unit adsorption capacity of printing dye at adsorption equilibrium (μgg^{-1}), q_t is the unit adsorption capacity of printing dye at any time (μgg^{-1}), K_1 is the pseudo-first-order kinetics rate constant (1/h), K_2 is the pseudo-second-order kinetics rate constant (g/(μg·h)), α is the initial adsorption rate (mg/g·min), β is the desorption constant (g/mg), and t is the reaction time (min).

3. RESULTS AND DISCUSSIONS

3.1 Optimization of the adsorption process

Table 2 shows the 4-variable DSD matrix and obtained adsorption process efficiencies (%) for 15 experimental runs. The efficiency ranges from 11.53 to 83.78%, whereby it was found that the maximum and minimum decolorization efficiency during the adsorption treatment is achieved under different process conditions, which confirms the assumption that the dye removal process mainly depends on the applied experimental conditions.

Table 2: Experimental design layout and obtained adsorption efficiency

Experimental run	Dye concentration (mgL^{-1})	Adsorbent mass (g)	pH	Reaction time (min)	Adsorption efficiency (%)
1	100	0.1	10	60	11.53
2	100	0.01	2	1	51.40
3	180	0.055	2	60	35.18
4	20	0.055	10	1	81.08
5	180	0.01	6	1	47.04
6	20	0.1	6	60	79.73
7	180	0.1	2	30.5	79.05
8	20	0.01	10	30.5	39.19
9	180	0.1	10	1	71.74
10	20	0.01	2	60	59.46
11	180	0.01	10	60	59.68
12	20	0.1	2	1	83.78
13	100	0.055	6	30.5	63.86
14	100	0.055	6	30.5	66.36
15	100	0.055	6	30.5	67.60

Prediction profiler with optimal settings is shown in Figure 2. The optimization plot graphically illustrates how the adsorption process efficiency changes as a function of one of the variables, while all other variables remain constant. According to optimization results, maximum adsorption process efficiency of 88.5% is obtained for the following process conditions: 32.8 mgL^{-1} of Cyan dye concentration, 0.09 g of adsorbent mass, pH 6 and reaction time of 20 minutes.

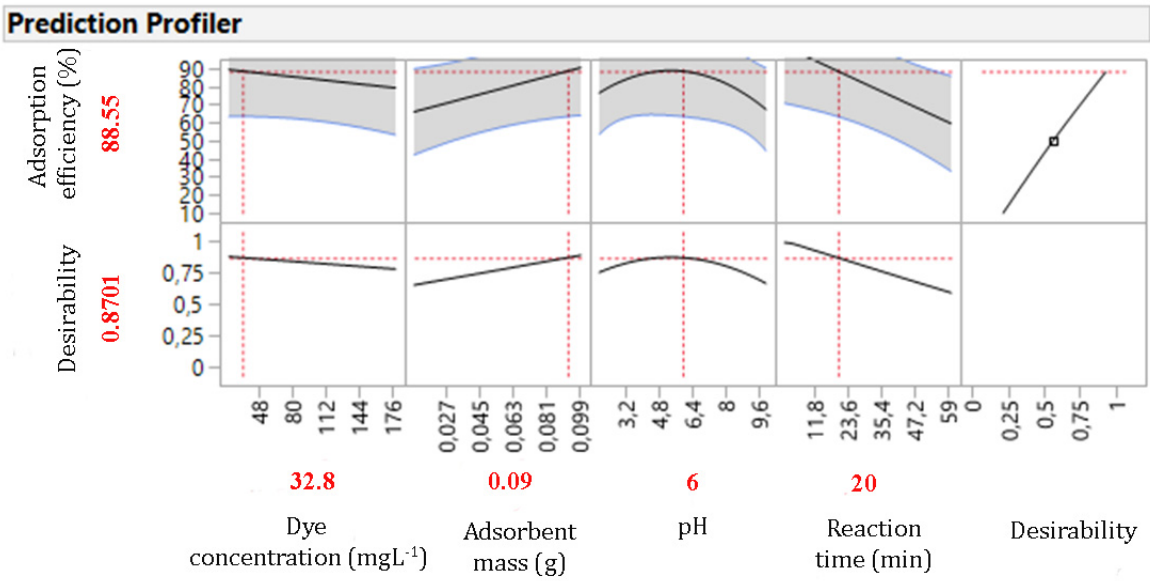

Figure 2: Optimization diagram

The optimization diagram clearly shows the strong influence of adsorbent mass, pH value and reaction time on the performance of the adsorption process. The results obtained confirm that the adsorption efficiency increases with increasing adsorbent mass and decreasing reaction time, due to the initially high number of free active sites, and finally to the coverage of the active sites by adsorbed dye molecules (Tubić et al., 2023).

3.2 Kinetics of adsorption

In order to analyze the Cyan adsorption behaviour on activated carbon, three kinetic models were used: pseudo-first order, pseudo-second order and Elovich model. The modelling curves are presentend in Figure 3. Based on the linear equations, the values of the pseudo-first-order (K_1) and pseudo-second-order (K_2) kinetics rate constants, the adsorption capacity at equilibrium (q_e), the initial reaction rate (h) for the adsorption of prinitng dye on activated carbon, the initial adsorption rate (α) and the desorption constant (β) were determined (Table 3).

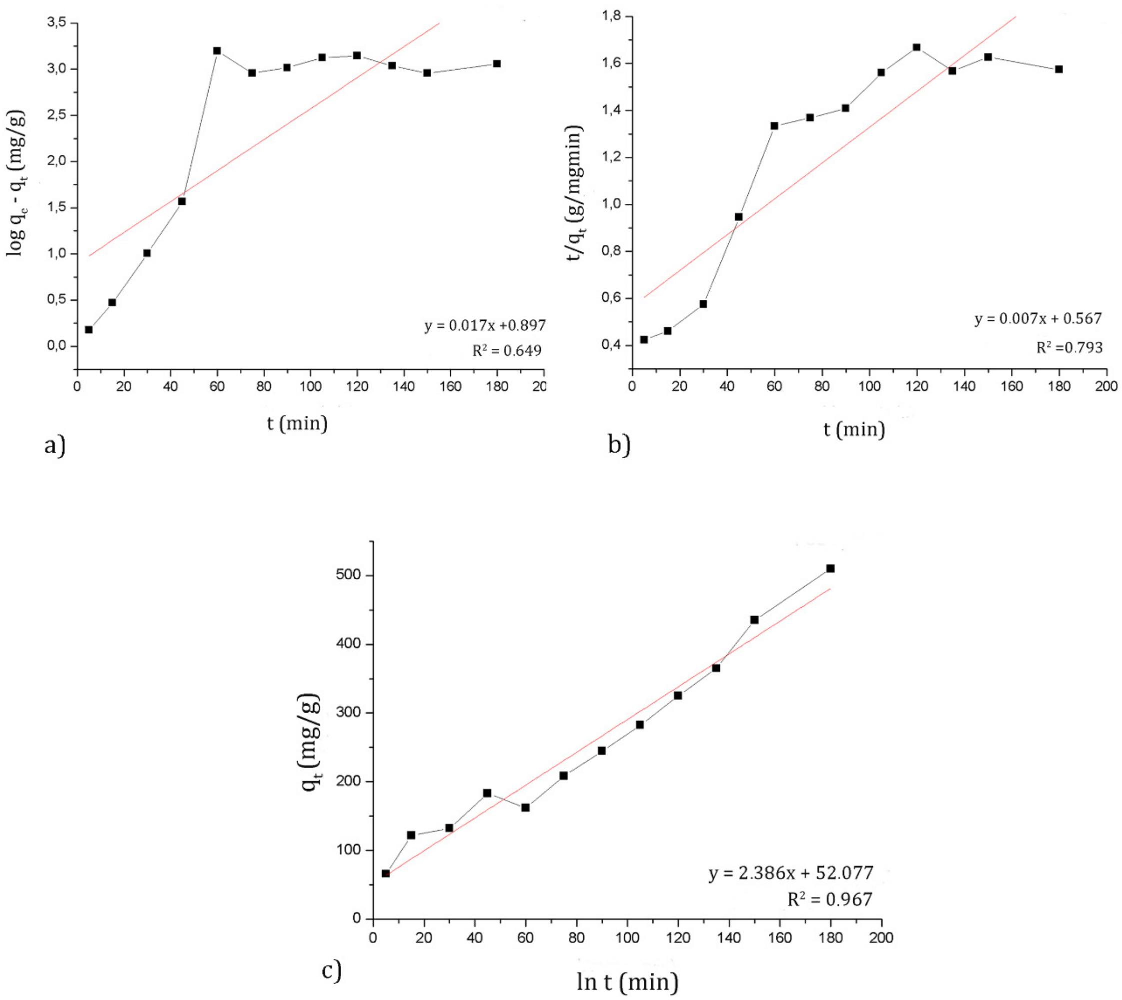

Figure 3: The plots of modelling experimental data: a) pseudo-first order, b) pseudo-second order, c) Elovich model

Table 3: Parameters of kinetic models for the adsorption of Cyan dye on activated carbon

Pseudo-first order			Pseudo-second order			Elovich		
K_1 (h^{-1})	q_e (μg/g)	R^2	K_2 (g/μg h)	h (μg/g h)	R^2	α (μg/gh)	β (μg/g)	R^2
1.33	409.7	0.649	0.0036	689.69	0.793	$2.28E^{+04}$	0.024	0.967

The obtained values of the coefficients of determination (R^2) leads to the conclusion that Elovich model ($R^2 = 0.967$) is best suited to describe the adsorption kinetics. The Elovich model is based on the assumption that the surface of the adsorbent is energetically heterogeneous an that chemical interactions occur in the activated carbon/prinitng dye system, which is primarily a consequence of the presence of numerous functional groups on the surface of the activated carbon that have a high affinity to the dye (Tang et al., 2021; You et al., 2021; Atugoda et al., 2020). The adsorption mechanism of Cyan dye onto the activated carbon surface is probably accomplished by a variety of mechanisms including van der Waals interactions, hydrogen bonding and halogen bonding interactions (Tubić et al., 2013). Furthermore, Elovich model is related to the kinetic principle which assumes that adsorption decreases with time, due to the coverage of the active sites by adsorbed dye molecules (Ayawe et al., 2017), which is in accordance with the optimization diagram (Figure 2).

4. CONCLUSIONS

Commercial activated carbon was applied as adsorbent to remove water-based phthalocyanine printing dye (Cyan) form aqueous solution. In order to quantify and elucidate the effects of the influencing factors (initial dye concentrations, adsorbent mass, pH and reaction time on the removal rate of Cyan dye) definitive screening design was used to estimate the linearity of the effects. 32.8 mgL^{-1} of the initial dye concentration, 0.09 g of the adsorbent mass, pH 6 and 20 minutes of the reaction time led to the best Cyan dye removal (88.55 %), according to the desirability function. Cyan adsorption kinetics and equilibrium adsorption data show that the process is chemisorption onto a heterogeneous surface. Three kinetic models (the pseudo-first-order, the pseudo-second-order and Elovich) were evaluated in order to validate experimental facts about sorption mechanisms and identify potential reaction processes that control the adsorption mechanism. The adsorption mechanism implies chemical binding with several binding forces, such as hydrogen bonding, halogen bonding and van der Waals interactions, that may be involved in the adsorption of printing dye on activated carbon.

5. ACKNOWLEDGMENTS

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