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APPLICATION OF RESPONSE SURFACE METHOD FOR OPTIMIZATION OF ERIOCHROME BLACK T REMOVAL BY ADSORPTION

BY

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Abstract. This study used Response Surface Methodology (RSM) to optimize Eriochrome Black T adsorption on activated carbon. Based on the Design of Experiments approach, static and static-stirred experiments were planned and conducted. Each set had 15 experiments and three replications at the center point. Experiments were conducted at room temperature with no pH adjustments. In static conditions, the maximum removal efficiency was 63.01%, while in static-stirred conditions, it was 78.18%. MINITAB 17.1.0 was used to analyze and interpret the experimental results. Linear, factorial, quadratic, and cubic statistical models were developed to describe static and static-stirred process parameter interactions. In addition, 3-D response surface plots and 2-D contour plots were used to show parameter efficiency. The RSM analysis of process parameters and their influence on retention yield increased removal efficiencies up to 85.20% in the static regime and to complete removal in the static-stirred regime, demonstrating optimization's ability to improve process performances.

Keywords: adsorption, activated carbon, Eriochrome Black T, optimization, Response Surface Methodology.

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1. Introduction

Eriochrome Black T (EBT) is a typical component of the azo-dye class of colorants, which is well known as a complexometric indicator for some divalent cations Ca^{2+}, Mg^{2+}, Zn^{2+} (Dave *et al*., 2011) and some rare earth metals (Lyle and Rahman, 1963). EBT is also a dye and has commercial applications in the textile, rubber, ink, cosmetic and pharmaceutical industries (Ben Arfi *et al*., 2017; Onu *et al*., 2021; Veerakumar *et al*., 2019). EBT is a naphthol azo dye known for its toxicity and resilience to light, heat, chemical attack, microbiological attack, and oxidative biodegradation (Mittal and Gupta, 2010; Veerakumar *et al*., 2019). Furthermore, its decomposition may produce carcinogenic aromatic compounds, and this is another reason for its use as a model pollutant (Mittal and Gupta, 2010; Bandari *et al*., 2015; Veerakumar *et al*., 2019). As a result, numerous methods were developed for removing EBT from aqueous solutions, such as chemical coagulation (Oyewo *et al*., 2022), electrochemical coagulation (Cañizares *et al*., 2006), flocculation (Abbas *et al*., 2020), adsorption (Manzar *et al*., 2021; Altaher *et al*., 2014; Dave *et al*., 2011; de Luna *et al*., 2013; Mittal and Gupta, 2010; Raveendra *et al*., 2015; Saxena *et al*., 2020; Pintilie *et al*., 2022a), photocatalysis (Abbas and Trari, 2020; Zhu and Jiang, 2021; Pintilie *et al*., 2022b; Pintilie *et al*., 2022a; Suditu *et al*., 2022) and various advanced oxidation processes (Gogoi *et al*., 2019; Zhu and Jiang, 2021; Bedoui *et al*., 2009).

The advantages of adsorption over other methods include its simplicity and efficiency (Yagub *et al*., 2014; Corda and Kini, 2018; Laskar and Kumar, 2022). However, the biggest drawback being its high cost, which is usually related to the high price of efficient adsorbents (Altaher *et al*., 2014; Corda and Kini, 2018; Karaca *et al*., 2008; Laskar and Kumar, 2022). Currently, lignocellulosic biomass (LCB) is used as raw material for activated carbon (AC) preparations (Mohamad Nor *et al*., 2013; Gayathiri *et al*., 2022). However, this involves various thermal and physicochemical treatments that increase the production costs (Gayathiri *et al*., 2022; Yogalakshmi *et al*., 2022). Besides that, the LCB quality differs from season to season depending on the amount of annual precipitation, transport, and storage condition (Gayathiri *et al*., 2022; Othmani *et al*., 2021). As a result, each batch of freshly produced AC requires complete physicochemical characterization, which is both costly and time-consuming. On the other hand, the commercial AC is at least partially characterized by producers, with uniform composition and reliable properties. Therefore, for this study, a commercial form of AC was used.

The mechanism for dye retention by adsorption commonly involves bulk diffusion, film diffusion, pore diffusion or intra-particle diffusion, and chemical adsorption/reaction (Crini, 2005; Deb *et al*., 2020) or physical adsorption (Secula *et al*., 2014). In addition, the mixing conditions can have a significant effect on the adsorption mechanism, influencing both the solute distribution in the bulk solution and the formation of an external boundary film over the adsorbent particles (Dotto and Pinto, 2011; Deb *et al.*, 2020; Saxena *et al.*, 2020; Karaca *et al*., 2008). However, few literature reports consider the hydrodynamic conditions (overhead stirring, orbital shaking, magnetic stirring, or ultrasonic mixing) among the process parameters during optimization studies on EBT adsorption on AC (Altaher *et al*., 2014; Secula *et al*., 2014).

The Response Surface Method (RSM), proposed by Box and Hunter in the late ’50s (Box and Hunter, 1957; Box and Hunter, 1961), is now regarded as a classic optimization procedure (Gunst, 1996; Myers *et al*., 2016). Most modern optimization software programs include response surface methodology (*e.g*., Minitab, Design Expert, Stat-Ease, IBM SPSS). RSM minimizes the number of experiments, covers the variation range of the selected parameters, and provides information about the interactions among the parameters. The primary goal of RSM is to find the optimum response. RSM also shows how changing design variables affect the response in a particular direction, and the response surface can be graphically visualized.

In this work, the absorption of EBT on activated carbon was studied experimentally by using the Design of Experiments approach (DOE) and modeled using RSM. In order to study the hydrodynamic conditions, two sets of experiments were planned. For each set, three process variables were considered: adsorbent concentration (Z1, g/L), time (Z2, min.), and dye concentration (Z3, mg/L). The first set was performed in static conditions (without stirring), while the second was performed under constant and continuous stirring (100 r.p.m.). After that, the experimental results were analyzed and interpreted using the MINITAB 17.1.0 software suite. Statistical models (linear, factorial, quadratic, and cubic) were developed to describe the interaction of the selected process parameters in static and static-stirred conditions. In addition, 3-D response surface plots and two-dimensional contour plots were used to highlight the parameters' influence on process efficiency.

2. Materials and methods

2.1. Materials and equipment

The EBT solution was made by dissolving the appropriate amount of dye powder , as presented in Table 2 (supplied by S.C. ChimReactiv S.A., analytical purity) in distilled water; any other concentrations required were obtained by diluting the initial solution.

The activated carbon was supplied by S.C. Romcarbon S.A. The particles were washed several times with bi-distilled water to remove surface impurities before being dried at 120°C for 24 hours and sieved, with the average diameter ranging between 2.5 and 3.15 mm. The main characteristics of the commercial activated carbon were extensively investigated and reported by Secula *et al*.

(Secula *et al*., 2011; Secula *et al*., 2014). Additionally, for this study, SEM images were registered using a Vega/Tescan scanning electron microscope (Fig. 1). The particle form, size, pore size, and layout were identified. The parallel alignment of the pores, as well as their size equivalence, were highlighted by the captured images.

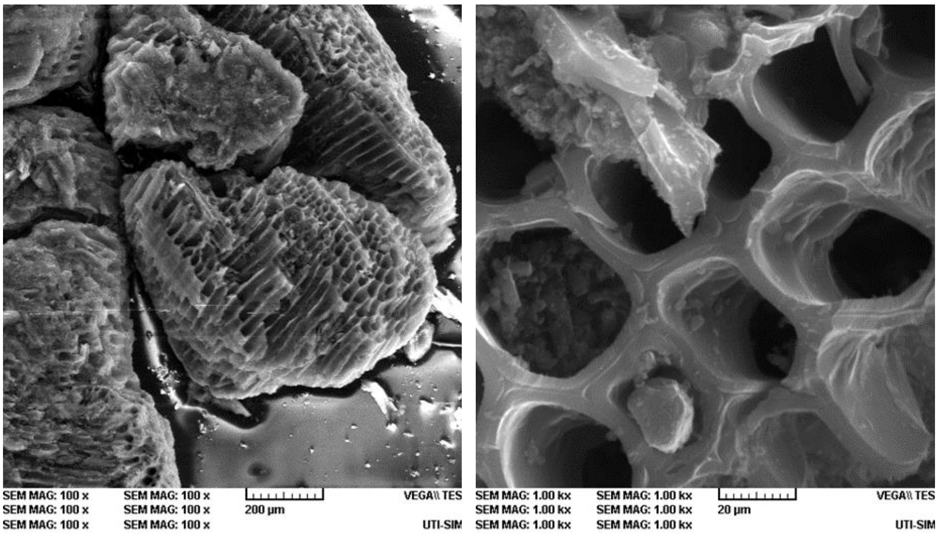


Fig. 1 – SEM images of the commercial AC: particle size, pore shape, and pore alignment.

The UV-Vis spectra and the absorbance values to determine the EBT concentrations were recorded by using a HITACHI U5100 UV-VIS spectrophotometer. Dye retention was evaluated by measuring the absorbance of the Eriochrome Black T solution at 535 nm. A calibration curve (linear, with R^2 = 0.99781) was used to make the absorbance - concentration conversion (Pintilie *et al*., 2022b; Sava *et al*., 2022).

2.2. Experimental design

The RSM approach was used in this study to optimize Eriochrome Black T adsorption on commercial activated carbon. The process parameters considered for optimization were the adsorbent concentration, contact time, and dye concentration. These independent variables and their limits (Table 1) were selected based on the data provided by literature (Manzar *et al*., 2021).

Based on the DOE methodology, two sets of experiments, one static and one static-stirred, were planned and carried out (Table 2). The design included a total of 15 experiments for each set and three replications at the center point.

Table 1
The designated variables and their variation range

Independent variables/	Symbol	Range		Measurement units
		from	to	
AC concentration	Z1	0.8	8	g/L
Contact time	Z2	10	60	min
EBT concentration	Z3	1	10	mg/L

Table 2
Central composite design and experimental results

No.	Input variables							Response	
	Type[1]	Z1		Z2		Z3		η_S, (%)	η_D, (%)
		real	*coded*[2]	*real*	*coded*[2]	*real*	*coded*[2]		
1	O1	8	1	60	1	10	1	**43.98**	**57.92**
2	O2	0.8	−1	60	1	10	1	**10.79**	**20.26**
3	O3	8	1	10	−1	10	1	**23.66**	**47.32**
4	O4	0.8	−1	10	−1	10	1	**10.03**	**28.99**
5	O5	8	1	60	1	1	−1	**63.01**	**78.18**
6	O6	0.8	−1	60	1	1	−1	**30.39**	**68.83**
7	O7	8	1	10	−1	1	−1	**6.91**	**53.25**
8	O8	0.8	−1	10	−1	1	−1	**9.04**	**13.77**
9	S1	8.774	α	35	0	5.5	0	**43.78**	**63.16**
10	S2	0.026	−α	35	0	5.5	0	**12.09**	**29.73**
11	S3	4.4	0	65.375	α	5.5	0	**42.85**	**62.98**
12	S4	4.4	0	4.625	−α	5.5	0	**10.93**	**42.86**
13	S5	4.4	0	35	0	10.9675	α	**49.37**	**53.30**
14	S6	4.4	0	35	0	0.0325	−α	**4.10**	**5.00**
15	C1	4.4	0	35	0	5.5	0	**6.43**	**54.00**
16	C2	4.4	0	35	0	5.5	0	**10.79**	**59.67**
17	C3	4.4	0	35	0	5.5	0	**10.37**	**61.46**
18	C4	4.4	0	35	0	5.5	0	**11.04**	**56.84**

[1]: O = orthogonal design points, S = axial or star points, C = centre points;
[2]: -1 = low value, 1 = high value, 0 = centre value, -α, α = star point value; η_S = removal efficiency (%), static; η_D = removal efficiency (%), static-stirred

2.3. Adsorption studies

The batch adsorption experiments were conducted at room temperature, with no pH adjustments. 100 mL samples of specifically diluted EBT solutions were mixed with an adequate amount of activated carbon, according to the data presented in Table 2. The first experimental set was performed in static conditions (without stirring), while the second one was performed under constant and continuous stirring (100 rpm). Disposable disc filters 0.22 µm were used for particle separation during solution sampling. The efficacy of EBT decolorization was calculated using Eq. (1), where $[Z_3]_i$ and $[Z_3]_f$ are the concentrations recorded before and after each experiment:

$$\eta\ (\%) = \frac{[Z_3]_i - [Z_3]_f}{[Z_3]_i} \cdot 100 \quad (1)$$

3. Results and discussions

3.1. Response surface modeling

As it can be observed from Table 2, the experimental studies considered both static and static-stirred conditions for analyzing removal efficiencies. Experimental run no. 8 (Table 2), corresponding to the highest AC amount, longest contact time, and lowest dye concentration, yielded the best results in both cases (63.01% static and 78.18% static-stirred). As a general rule, stirring (static conditions, at equilibrium or static-stirred) has a beneficial impact on removal efficiencies. The turbulence produced in static-stirred conditions favors the transfer of dye molecules through the surface of the adsorbent, facilitating the adsorption process. This proves once more that stirring is an essential factor in adsorption, since it influences both the solute distribution in the bulk solution and the creation of the exterior boundary film. Furthermore, the switch to static-stirred conditions triggered an increase in both film and intraparticle diffusivity that increased adsorption capacity.

The regression equations were obtained by fitting linear, quadratic, cubic, and 2-factor interaction models to the experimental data to determine the relevance of the selected variables. Some of the statistical parameters of the polynomial models are presented in Table 3.

Table 3
The statistical parameters of the polynomial models developed for EBT retention on AC

Model	**Static**			**Static-stirred**		
	Std. dev.	R^2	*Adj.* R^2	*Std. dev.*	R^2	*Adj.* R^2
linear	13.71	0.58	0.47	16.34	0.44	0.32
2-factor interaction	13.35	0.73	0.50	16.28	0.61	0.33
quadratic	14.80	0.75	0.38	16.40	0.68	0.32
cubic	11.95	0.95	0.60	9.16	0.95	0.79
cubic simplified	7.64	0.91	0.83	7.1	0.94	0.87

Among the tested models, the cubic simplified approach had the best fitting performance. It was obtained by using the Backward elimination of terms, and its regression equation is presented in Eqs. 2 (static) and 3 (static-stirred). In addition, the analysis of variance is presented in Table 4 for static conditions and Table 5 for static-stirred conditions.

Table 4
Analysis of variance for the best model obtained in static conditions

Source	DF	Seq-SS	Contribution	Adj-SS	Adj-MS	F-Value	P-Value
Regression	7	4838.94	91.19%	4838.94	691.28	11.82	0.001
Z1	1	1224.7	23.08%	3.4	3.40	0.06	0.815
Z2	1	1721.62	32.44%	355.39	355.39	6.08	0.039
Z3	1	106.26	2.00%	977.17	977.17	16.71	0.003
Z3·Z3	1	47.47	0.89%	929.2	929.20	15.89	0.004
Z1·Z2	1	368.72	6.95%	368.72	368.72	6.31	0.036
Z2·Z3	1	397.18	7.48%	397.18	397.18	6.79	0.031
Z3·Z3·Z3	1	972.98	18.34%	972.98	972.98	16.64	0.004
Error	8	467.7	8.81%	467.7	58.46		

Table 5
Analysis of variance for the best model obtained in static-stirred conditions

Source	DF	Seq SS	Contribution	Adj SS	Adj MS	F-Value	P-Value
Regression	9	6301.31	93.99%	6301.31	700.15	13.89	0.001
Z1	1	1931.74	28.81%	683.70	683.70	13.57	0.006
Z2	1	1031.97	15.39%	1422.77	1422.77	28.23	0.001
Z3	1	1.21	0.02%	2244.06	2244.06	44.52	0.000
Z3·Z3	1	746.58	11.14%	1663.35	1663.35	33.00	0.000
Z1·Z2	1	14.59	0.22%	254.45	254.45	5.05	0.055
Z1·Z3	1	6.42	0.1%	162.68	162.68	3.23	0.110
Z2·Z3	1	763.03	11.38%	962.52	962.52	19.01	0.002
Z3·Z3·Z3	1	1500.04	22.37%	1500.04	1500.04	29.76	0.001
Z1·Z2·Z3	1	305.72	4.56%	305.72	305.72	6.07	0.039
Error	8	403.21	6.01%	403.21	50.40		

In Tables 4 and 5, DF represents the degrees of freedom and is used to estimate the values of unknown population parameters. Adj-SS represents the adjusted sum of squares and indicates the variations for different elements in the model. Adj-MS represents the adjusted mean squares. It is based on DF, and indicates how much variation a specific term of the model explains. F value indicates if the terms of the models are associated with the response.

$$Y = -28.2 + 0.30 \cdot Z1 + 0.514 \cdot Z2 + 34.42 \cdot Z3 - 7.92 \cdot Z3 \cdot Z3 + 0.0754 \cdot Z1 \cdot Z2 - 0.0626 \cdot Z2 \cdot Z3 + 0.489 \cdot Z3 \cdot Z3 \cdot Z3 \quad (2)$$

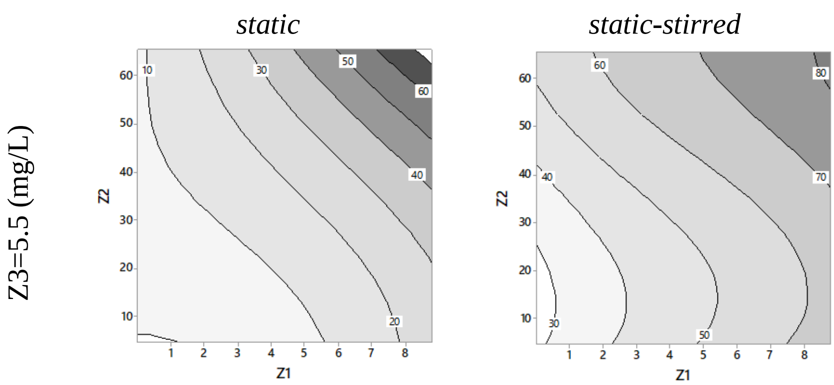
$$Y = -47.1 + 6.85 \cdot Z1 + 1.301 \cdot Z2 + 52.56 \cdot Z3 - 10.59 \cdot Z3 \cdot Z3 - 0.099 \cdot Z1 \cdot Z2 - 0.479 \cdot Z1 \cdot Z3 - 0.154 \cdot Z2 \cdot Z3 + 0.608 \cdot Z3 \cdot Z3 \cdot Z3 + 0.01526 \cdot Z1 \cdot Z2 \cdot Z3 \quad (3)$$

As it can be observed from Table 4, in the case of static conditions, only Z1 has a p-value higher than 0.05, indicating that the parameter has a statistically insignificant contribution to the specific model. On the other hand, in the case of static-stirred conditions, the terms Z1·Z3 and Z1·Z2 have p-values higher than 0.05, pointing out that these interactions between the terms are not significant to the model selected.

3.2. Effects of experimental variables on EBT removal

The study of the contour plots (Fig. 2) and the 3-D plots (Fig. 3) allows the visualization of maximum and/or minimum points, allowing the precise identification of the optimal values and revealing the impact of the selected parameters on the dye removal efficiency.

The analysis of Fig. 2 reveals the benefits of the static-stirred vs. static conditions in terms of decolorization efficiency. In similar experimental conditions, the EBT retention is up to 40% higher in static-stirred conditions.



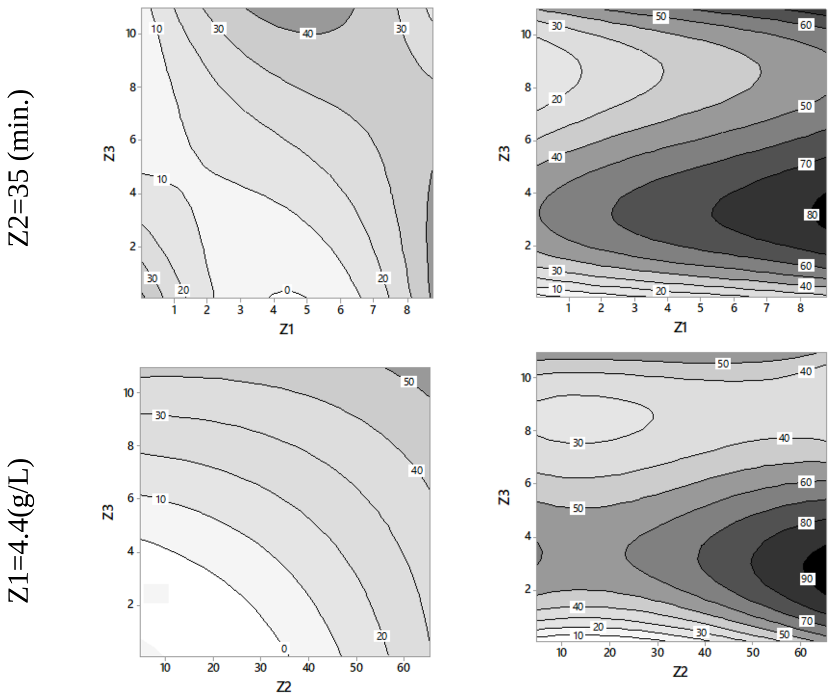
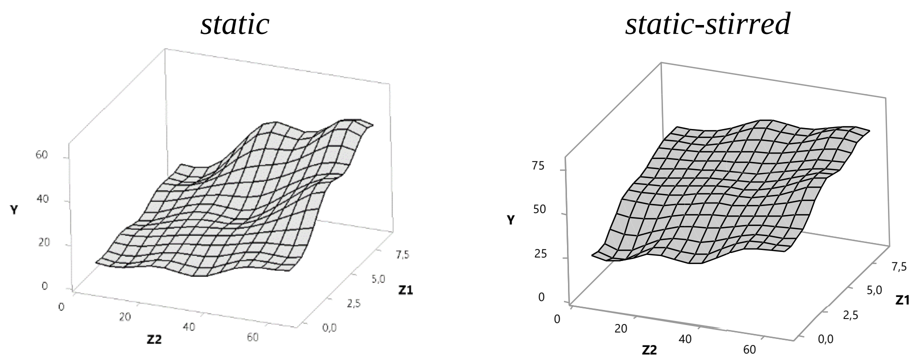


Fig. 2 – Contour plots static vs. static-stirred.

The 3-D charts were constructed by keeping one parameter constant (typically the median value of the variation interval) (Fig. 3). The analysis of Fig. 3 shows equivalent behavior towards process parameters variation – the shape of the 3D plots is remarkably alike in static and static-stirred conditions, which means that the adsorption mechanism is analogous. Consequently, stirring affects the absorption rate's limiting steps (transfer through the boundary layer and/or intraparticle diffusion) but has no effect on the adsorption mechanism.



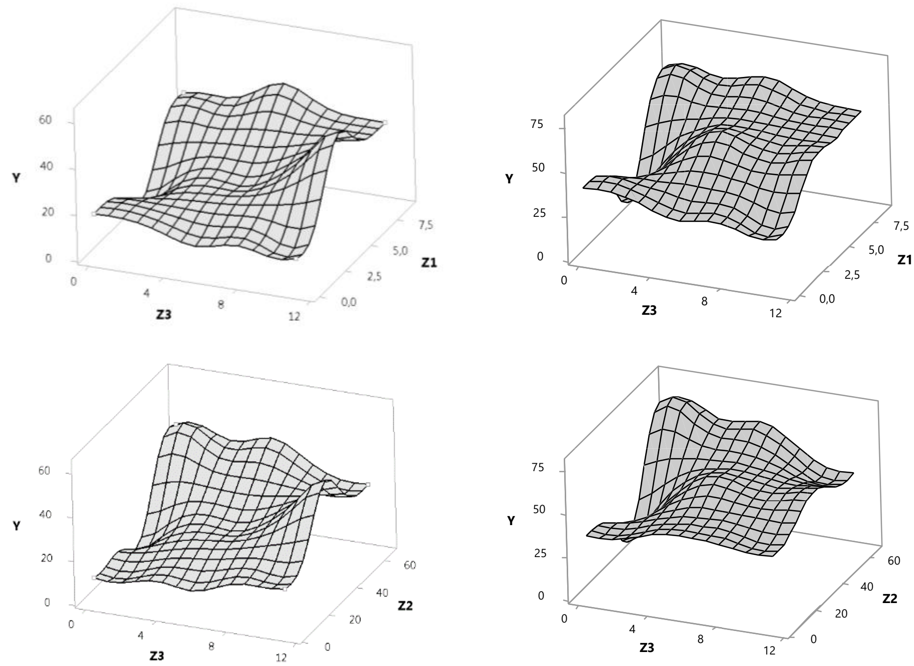


Fig. 3 – Surface plot static vs. static-stirred.

3.3. Optimization results

Based on the models previously determined, the Minitab software was used to determine the conditions that lead to a maximization of efficiency in both cases. The best results are presented in Table 6.

Table 6
Optimization results

	Solution	Z1	Z2	Z3	Fit (%)
Static	1	8.77	65.38	2.46	85.20
	2	8.77	65.38	0.44	63.01
	3	8.77	37.38	10.97	63.00
	4	4.4	35	2.71	41.58
	5	0.35	65.38	2.49	41.15
Static-stirred	1	8.77	65.38	3.01	103.46
	2	8.77	34.48	10.97	78.18
	3	8.77	34.48	10.97	78.18
	4	8.77	65.38	0.10	78.18
	5	0.04	65.38	1.39	78.18

As it can be observed from Table 6, the maximum efficiency in both cases (solution 1) was obtained when Z1 and Z2 had the same values. However, Z3 had a higher value in the static-stirred case, indicating that stirring could perform better at higher EBT concentrations. Moreover, for solution 1, the efficiency in static-stirred conditions was higher (103.46%). Although the model indicates an efficiency higher than 100%, this is due to the model's error, which has an R^2 of 0.94.

Overall, the optimization results provided solutions better than the experimental data, demonstrating that the applied strategy can improve the efficiency of the process.

4. Conclusions

This work focused on modeling and optimizing the absorption of EBT on activated carbon. Based on a series of experimental data planned through a DOE approach, an RSM strategy was used to determine the best model that suits the process studied in two cases: static and static-stirred conditions. The experimental study showed that the maximum removal efficiency in the static-stirred case is approximately 15% better than in the static conditions. In addition, the change from static to static-stirred settings resulted in an increase in film and intraparticle diffusivity that amplifies the adsorption capacity.

After that, using the best models obtained, the Minitab software was used to determine the conditions that lead to the highest efficiency. The results showed that the optimization strategy led to better solutions in both cases compared with the experimental data.

Thus, it was shown that, even for somewhat classical known processes, the modeling and optimization strategies could improve the process and identify optimum conditions that can be further used in various set-ups.

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METODA SUPRAFEȚEI DE RĂSPUNS PENTRU OPTIMIZAREA ELIMINĂRII ERIOCROM NEGRU T PRIN ADSORBȚIE

(Rezumat)

În acest studiu, metodologia suprafeței de răspuns (RSM) a fost aplicată pentru a optimiza adsorbția Eriocrom negru T pe cărbune activ. Folosind metoda planificării experimentale, au fost realizate o serie de experimente în condiții statice și statice cu agitare. Fiecare set a avut 15 experimente și trei replicări în punctul central.

Experimentele au fost efectuate la temperatura camerei, fără ajustări ale pH-ului. În condiții statice, eficiența maximă de îndepărtare a fost de 63.01%, în timp ce în condiții statice cu agitare a fost de 78.18%. Softul MINITAB a fost utilizat pentru a analiza și interpreta rezultatele experimentale. Modele statistice (liniar, factorial, pătratic și cubic) au fost dezvoltate pentru a descrie interacțiunile dintre parametrii de proces. Grafice suprafață de răspuns 3-D și de contur 2-D au fost utilizate pentru a arăta eficiența parametrilor. Prin analiza RSM a parametrilor de proces și a influenței lor asupra randamentului de retenție s-a crescut eficiența de eliminare la 85.20% în condiții statice și la 100% în condiții statice cu agitare, demonstrând capacitatea optimizării de a îmbunătăți performanța procesului.