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ADSORPTION STUDY OF BASIC DYES IN AQUEOUS SOLUTION ON CHITOSAN USING RESPONSE SURFACE METHODOLOGY

AVALIAÇÃO DA ADSORÇÃO EM SOLUÇÃO AQUOSA DE CORANTES BÁSICOS EM QUITOSANA UTILIZANDO METODOLOGIA DE SUPERFÍCIE DE RESPOSTA

CRYSTIANO MATHEUS DIAS CARVALHO
UEG – Universidade Estadual de Goiás, Anápolis / GO
crystianomatheus@gmail.com

ANA CLÁUDIA NOGUEIRA SOARES
UEG – Universidade Estadual de Goiás, Anápolis / GO
ananogueira4553@gmail.com

BEATRIZ FERREIRA DA CUNHA PUDO
UEG – Universidade Estadual de Goiás, Anápolis / GO
beatrizfcpudo@gmail.com

ROBERTA SIGNINI
UEG – Universidade Estadual de Goiás, Anápolis / GO
roberta.signini@ueg.br

Resumo: A Quitosana foi utilizada como adsorvente na remoção dos corantes catiônicos azul de metileno e cristal violeta em soluções aquosas utilizando como ferramenta a metodologia de superfície de resposta (MSR) para otimizar o planejamento experimental. A quitosana foi caracterizada pela espectroscopia de infravermelho com transformada de Fourier (FTIR) e pH_{PCZ}, que obteve valor de 6,9. O planejamento experimental completo 3^2 foi utilizado tendo como variáveis independentes os fatores pH e tempo de contato e como variável dependente a capacidade de adsorção, variando nos níveis 7,5-8,5-9,5 para pH e 60-120-180 minutos para o tempo de contato. Os resultados indicaram que 77% do corante azul de metileno pode ser removido em pH 9,5 e tempo de contato de 60 minutos, sendo que o pH é o principal fator que influencia na capacidade de adsorção. Os valores de remoção foram melhores ajustados ao modelo de regressão quadrática, com desejabilidade de 0,99 e q_e de 1,84 mg g^{-1}. Já para o corante cristal violeta, é possível remover até 70% do corante em pH 7,5 e tempo de contato de 120 minutos, com capacidade de adsorção (q_e) de 1,2 mg g^{-1}. O modelo de regressão quadrática se ajusta melhor para descrever o processo de adsorção, obtendo desejabilidade de 0,99 em pH 7,5 e tempo de contato de 120 minutos e q_e de 1,2 mg g^{-1}, indicando que tanto o pH como o tempo de contato influenciam na variação dos valores de q_e, com maior significância ao pH. Em geral, essa pesquisa mostra que a quitosana galena possui potencial de adsorção de corantes básicos em baixas concentrações de corantes catiônicos, com mais de 70% de capacidade de remoção.
Palavras-chave: cristal violeta. desejabilidade. planejamento fatorial. azul de metileno.

Abstract: Chitosan was used as adsorbent in the removal of the cationic dyes methylene blue and crystal violet in aqueous solution. Response surface methodology was applied as a tool to optimize experimental conditions. Chitosan was characterized by Fourier-transform infrared spectroscopy and point-of-zero charge analysis. The point of zero charge was found to be 6.9. A 3^2 full factorial design was used, with pH (7.5, 8.5, and 9.5) and contact time (60, 120, and 180 min) as independent variables and adsorption capacity (q_e) as the dependent variable. The results indicated that 77% methylene blue can be removed at pH 9.5 with a contact time of 60 min and that pH is the main factor influencing q_e. Removal values were better fitted by a quadratic regression model, with a desirability of 0.99 and a q_e of 1.84 mg g^{-1}. Results for crystal violet showed that it is possible to achieve up to

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70% removal at pH 7.5 and with a contact time of 120 min, resulting in a q_e of 1.2 mg g^{-1}. A quadratic regression model provided the best fit to the adsorption data, with a desirability of 0.99 at pH 7.5 in 120 min of contact and a q_e of 1.2 mg g^{-1}. These findings indicated that both pH and contact time influence q_e values, with pH having a greater significance. Overall, this research showed that Galena chitosan is able to adsorb basic dyes at low concentrations, with a removal capacity of more than 70%.
Keywords: crystal violet**.** desirability**.** factorial design**.** methylene blue.

Introduction

Contamination of water bodies is a major problem faced by both developed and developing countries (Freire et al., 2018). Industrial processes contribute significantly to water contamination, as several industries produce large amounts of wastewaters containing a high load of pollutants. Particularly worrisome are wastewaters containing high concentrations of non-biodegradable organic compounds, such as industrial dyes (Orts et al., 2018).

Inadequate disposal of industrial wastewater negatively impacts water resources and aquatic fauna and flora, exposing ecosystems to the risk of contamination by toxic compounds (Sanmuga Priya & Senthamil Selvan, 2017). Water contamination with industrial dyes can inhibit photosynthesis, given that it prevents sunlight from reaching the bottom of ponds, rivers, and lakes (Ali et al., 2016).

Although contact with industrial dyes and their byproducts is harmful to several life forms, these compounds are of paramount importance to several fields, being used in the glass, wood, textile, food, paper, and plastic industries. Two important examples of such dyes are methylene blue and crystal violet (Nayak et al., 2020).

Methylene blue is a cationic thiazine dye with applications in various fields, such as in biology, chemistry, and medicine (Vutskits et al., 2008). Furthermore, it can be used to dye cotton, wool, and silk (El-Kousy et al., 2020). This basic dye is a heterocyclic aromatic compound with chemical formula $C_{16}H_{18}N_3SCl$ and molar weight of 319.8 g mol^{-1} (JORGE et al., 2015). Figure 1a illustrates the chemical structure of methylene blue.

Crystal violet is another cationic dye used in various chemical and industrial processes, such as textile industries, which generate large amounts of colored wastewater (Muthukumaran et al., 2016; Vyavahare et al., 2019). As shown in Figure 1b, crystal violet contains three amino groups, which can bind to a large variety of compounds to form different products. This dye can exert mutagenic, carcinogenic, and mitogenic effects on several organisms (Kulkarni et al., 2017; Puri & Sumana, 2018).

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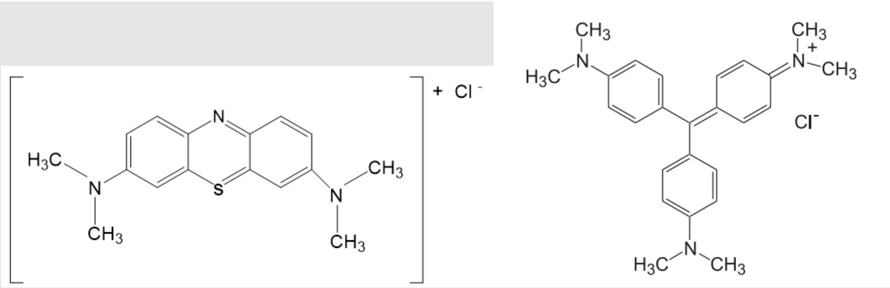
Figure 1: Chemical structure of (a) methylene blue and (b) crystal violet.



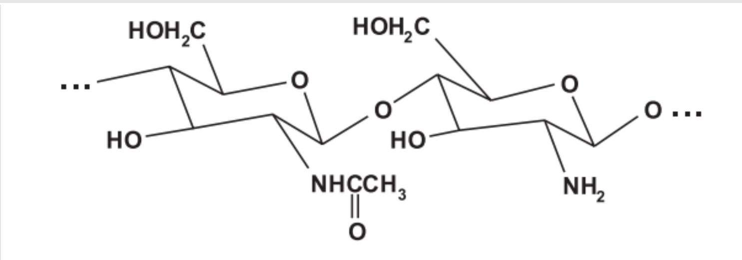
(a) (b

Source: CARVALHO C. M. D. et al.

Removing or reducing dyes from contaminated wastewaters is a great challenge for industries. Adequate treatment of aquatic pollutants is crucial to avoid negative impacts on organisms that inhabit aquatic ecosystems (Nayak et al., 2020). Several physicochemical methods can be used to eliminate or reduce the concentration of dyes in water supplies, such as coagulation and flocculation (Szyguła et al., 2008), membrane separation (Ahmad et al., 2002), oxidation or ozonation (Wijannarong et al., 2013), electrocoagulation (Alinsafi et al., 2005), and adsorption (Kaykhaii et al., 2018). Adsorption is a particularly interesting technique, as it eliminates trace elements and enables adsorbent reuse over several cycles (Tan et al., 2015; Wang et al., 2008).

A wide range of adsorbents can be used for wastewater treatment, such as activated carbon (Lin & Liu, 2000), sugarcane bagasse (Xavier et al., 2021), silica-based xerogels (Buzato et al., 2021), and chitosan (Wong et al., 2003). Chitosan is a natural polysaccharide obtained by deacetylation of chitin (Choi et al., 2016; Ngo et al., 2015). The great effectiveness of chitosan as an adsorbent is due to the presence of amino and hydroxyl groups in its chemical structure (Figure 2).

Figure 2: Chemical structure of the elementary unit of chitosan.



Source: CARVALHO C. M. D. et al.

In this study, the adsorption capacity of chitosan for methylene blue and crystal violet was evaluated and optimized using response surface methodology. For this, adsorption tests were carried out according to a full factorial design. Statistical methods were used to compare the results and construct graphs to assist in the identification of optimal adsorption conditions for each factor, namely pH and contact time. The mass of adsorbent and temperature were maintained constant, allowing us to assess the behavior of the adsorbent in the presence of cationic dyes.

Material and methods

Material

Chitosan (average diameter of less than 150 µm, degree of deacetylation of 90.7%, molecular weight of 5.7×10^3 g mol^{-1}) was acquired from Galena Chemicals & Pharmaceuticals. Ltd. Methylene blue and crystal violet were purchased from Sigma–Aldrich. Hydrochloric acid (HCl), sodium hydroxide (NaOH), and all other compounds were of analytical grade.

Fourier-transform infrared spectroscopy (FTIR)

A small sample of adsorbent was dried in a vacuum oven at 80 °C for 12 h and then sprayed with KBR with a spectroscopic degree in the ratio of 1:100 (w/w). Then, this mixture was pressed into pellets and dried again in a vacuum oven at 80 °C for 12 h. FTIR analyses were performed on a PerkinElmer Frontier FTIR/NIR spectrometer (PerkinElmer Inc., Norwalk, CT, USA) in the spectral range of 4000 to 400 cm^{-1} with a resolution of 4 cm^{-1}.

Point of zero charge (pH_{pzc})

For pH_{pzc} determination, suspensions containing 20 mg of chitosan were prepared in 20 mL of 0.1 mol L^{-1} NaCl solution. The pH was adjusted to values between 1 and 11 with 0.1 mol L^{-1} HCl and 0.1 mol L^{-1} NaOH. The initial pH ($pH_{initial}$) was measured using a Gehaka PG1800 pH meter. Samples were incubated in a thermostatic bath at 100 rpm and 25 °C for 24

h. After this period, solutions were filtered and the pH was measured again (pH_{final}). The pH_{pzc} was determined by plotting a graph of pH_{final} (ordinate axis) as a function of $pH_{initial}$ (abscissa axis). The pH_{pzc} was identified as the point at which pH_{final} remained constant, regardless of $pH_{initial}$; that is, the point at which the surface behaves as a buffer.

Experimental design and adsorption study

Factorial design

A full factorial design with two factors and three levels (3^2) was used. The factors were pH and contact time. Each run was performed in duplicate, totaling 18 experiments per dye. The response variable was adsorption capacity (q_e). Table 1 shows the levels and factors of the factorial design.

Table 1: Real and coded values of independent variables in the full factorial design used to study the adsorption of methylene blue and crystal violet on chitosan.

Factor	Level		
	−1	0	+1
pH	7.5	8.5	9.5
Contact time (min)	60	120	180

Source: CARVALHO C. M. D. et al.

Adsorption study

The conditions for the adsorption study were chosen based on the results of the experimental design. A 25 mg aliquot of adsorbent was weighed on an analytical scale and mixed with 25 mL of 2.5 mg L^{-1} dye solution in a beaker. The pH was adjusted to the pre-established pH_{pzc} by using a Gehaka PG1800 pH meter and 0.1 mol L^{-1} HCl or 0.1 mol L^{-1} NaOH. After pH adjustment, the dye + adsorbent system was incubated in a thermostatic bath at 100 rpm and 25 °C for a predetermined amount of time. After incubation, an aliquot (about 5 mL) was collected and centrifuged at 2500–3000 rpm for 3–5 min. Then, a 2 mL aliquot of the supernatant was analyzed spectrophotometrically on a PerkinElmer Lambda 35 UV-Visible spectrometer at the maximum absorption wavelength of each dye (λ_{max} = 664 nm for methylene blue and λ_{max} = 589 nm for crystal violet). This procedure was repeated separately for all conditions of the factorial design for both dyes.

Adsorption capacity (q_e, mg g^{-1}) was calculated by using Equation 1.

$$q_e = \frac{(C_0 - C_e)V}{w} \quad (1)$$

where C_0 (mg L^{-1}) is the initial concentration of the dye solution (before adsorption), C_e (mg L^{-1}) is the concentration of the solution at equilibrium (after adsorption), w (g) is the weight of the solution, and V (L) is the volume of the solution.

Experimental design and statistical analysis

Analysis of variance (ANOVA) was applied at the 95% confidence interval to test the significance of the curve, as measured by the t-test. The predictive model describing the adsorption process was second-order, according to the following equation (Montgomery, 2017):

$$y_i = b_0 + \sum_{i=1}^{k} b_i X_i + \sum_{i=1}^{k} b_{ii} X_i^2 + \sum_{i<1}^{k} b_{ij} X_i X_j + \varepsilon \quad (2)$$

where y is the response variable, X_i and X_j are the independent variables, b represents the coefficients estimated by the least-squares method, with a confidence interval of 95% measured by the t-test, and ε is the experimental error interval.

The second-order polynomial model was defined using the direct selection method, which considers the smallest constant value for the sum of squared errors. Once an adjusted polynomial model was obtained, the best process conditions were defined using the optimization algorithm proposed by Derringer and Suich (1980). For this, a desirability (D) function was defined, restricted to the interval [0,1]. The lower, middle, and upper limits were 0, 0.5, and 1.0, respectively. When the response is the expected value, D = 1, and when the response is outside the acceptable range, D = 0. Thus, independent variables are chosen so as to maximize the overall desirability. All statistical analyses, calculations, and graphs were performed using Statistica version 10 (StatSoft, Tulsa, USA).

Results and discussion

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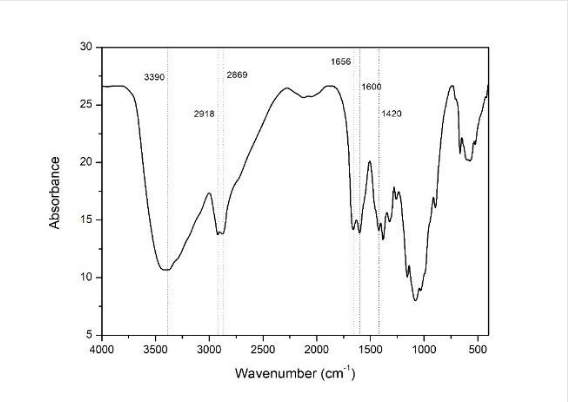
Figure 3 shows the absorption spectrum of chitosan in the infrared region. The following characteristic bands were observed: –OH axial stretching band at 3500–3400 cm^{-1}, which is superimposed on the –NH axial stretching band; angular deformation band of –NH near 1600–1550 cm^{-1} (amide II band); C=O axial deformation band at 1670–1650 cm^{-1} (amide I band); angular deformation band of the $–CH_3$ group near 1380 cm^{-1}; axial deformation band of –CH and $–CH_2$ at 2950–2850 cm^{-1}; axial deformation band near 1420 cm^{-1} of the C–N group of amides; and axial deformation band of the C–N group of amines at 1350–1000 cm^{-1}.

Figure 3: Infrared spectrum of chitosan.

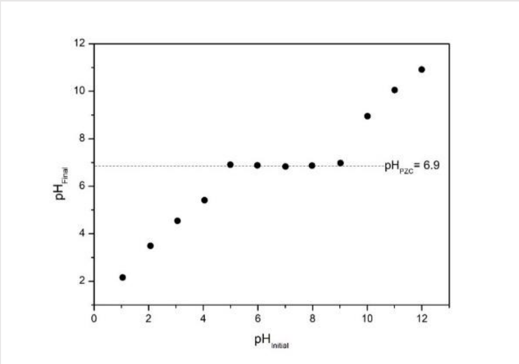


Source: CARVALHO C. M. D. et al.

pH_{pzc} represents the pH at which a solid has null electrical charge on its surface. This parameter allows predicting the surface charge of the adsorbent as a function of pH. When pH < pH_{pzc}, the adsorbent predominantly has positively charged sites and will tend to adsorb anions; when pH > pH_{pzc}, the adsorbent predominantly has negatively charged sites and will tend to adsorb cations, as depicted in Figure 4. The pH_{pzc} was found to be 6.9, as calculated using the arithmetic base of pH values. Given that methylene blue and crystal violet are basic dyes (cationic), the pH of the adsorption suspension would have to be greater than 6.9.

Methylene blue and crystal violet removal was assessed using a 3^2 full factorial design (Table 1), with pH and contact time as independent variables and adsorption capacity as the dependent variable. Table 2 shows the experimental results, values predicted by the second-order quadratic regression model, and removal percentages for both cationic dyes. The findings show that the predicted adsorption capacity had variations of less than 0.1 mg g^{-1} in relation to experimental values. Thus, predictions can be used for optimization of the adsorption process by response surface methodology.

Figure 4: Point of zero charge (pH_{pzc}) of chitosan.



Source: CARVALHO C. M. D. et al.

Table 2: 3^2 Full factorial design and experimental values for chitosan adsorption capacity (q_e) and removal of methylene blue and crystal violet.

Run	pH	Adsorption time (min)	Methylene blue		Crystal violet	
			q_e (mg g^{-1})	Removal (%)	q_e (mg g^{-1})	Removal (%)
1	9.5	120	1.26 ± 0.04	51.3 ± 1.75	0.25 ± 0.04	11.72 ± 1.70
2	7.5	180	0.96 ± 0.06	40.1 ± 2.14	1.03 ± 0.16	62.99 ± 8.91
3	8.5	120	1.24 ± 0.08	46.0 ± 3.46	0.69 ± 0.07	27.59 ± 2.94
4	9.5	60	1.84 ± 0.02	75.8 ± 2.39	0.70 ± 0.02	29.45 ± 0.94
5	8.5	180	0.53 ± 0.05	25.1 ± 2.48	0.81 ± 0.16	45.95 ± 9.24
6	7.5	60	0.82 ± 0.06	31.3 ± 2.74	0.68 ± 0.10	31.63 ± 4.80
7	7.5	120	0.79 ± 0.02	32.2 ± 0.95	1.19 ± 0.04	69.03 ± 2.12
8	8.5	60	0.47 ± 0.05	19.6 ± 1.92	0.54 ± 0.10	22.09 ± 4.16
9	9.5	180	1.27 ± 0.03	52.8 ± 1.40	0.49 ± 0.15	23.05 ± 7.10

Source: CARVALHO C. M. D. et al.

For methylene blue, the highest adsorption capacity coincided with the highest removal percentage, obtained with the +1 level of pH (pH 9.5) and the −1 level of adsorption time (60 min). The lowest adsorption capacity and removal percentage were obtained by using the 0 level of pH (pH 8.5) and the −1 level of adsorption time (60 min). Interestingly, the highest and lowest adsorption capacities were achieved in a reaction time of 60 min. Thus, differences in the results can be attributed to pH, whereby pH 9.5 afforded the highest adsorption capacity and pH 8.5 afforded the lowest, proving that the dye is cationic.

Table 3 shows the results of ANOVA for linear and quadratic models of methylene blue and crystal violet removal, indicating which effects are significant or not in the adsorption process.

Table 3: ANOVA table for linear and quadratic regression models of adsorption of methylene blue and crystal violet on chitosan as a function of pH and contact time.

Factor	Methylene blue				Crystal violet			
	LR		QR		LR		QR	
	DF	SS	DF	SS	DF	SS	DF	SS
Curvature	1	0.0	-	-	1	0.3*	-	-
pH	1	0.9*	2	0.3*	1	0.4*	2	0.9*
pH^2	-	-	2	0.3*	-	-	2	0.9*
Time	1	0.1*	2	0.7*	1	0.1*	2	0.3*
$Time^2$	-	-	2	0.7*	-	-	2	0.3*
pH × Time	1	0.3*	4	1.1*	1	0.0	4	0.1*
Lack of fit	-	-	-	-	5	0.0	-	-
Pure error	5	0.0	9	0.0	5	0.0	9	0.0
Adjusted R^2	0.979		0.986		0.974		0.969	

Note: Values highlighted with * are significant. LR, linear regression; QR, quadratic regression; DF, degrees of freedom; SS, mean sum of squares.

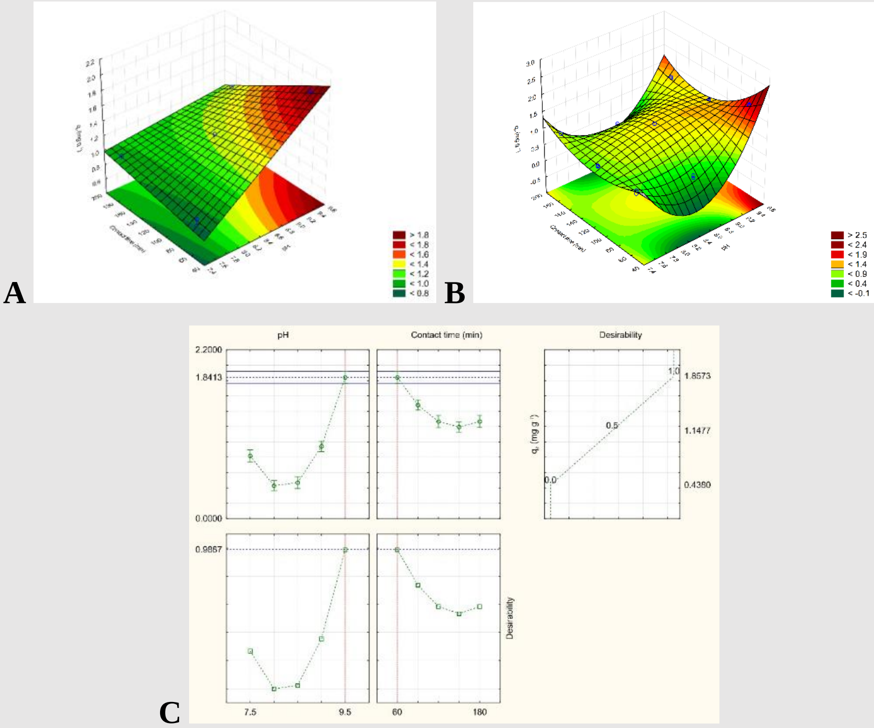
Source: CARVALHO C. M. D. et al.

It was observed that, although the curvature of methylene blue removal was not significant according to ANOVA (Table 3), the linear response surface plot shows a small curve at extreme values (Figure 5A). According to the quadratic regression model, however, all factors and their linear and quadratic interactions were significant, as depicted in the curves of Figure 5B. Table 3 shows that the effect with the greatest influence on adsorption capacity was the linear term of pH, with an adjusted R^2 of 0.986.

Experimental data were subjected to response surface methodology. Equation 3 presents the second-order regression model for methylene blue adsorption capacity as a function of pH (x) and contact time (y).

$$q_e = 263.30421391027 - 63.344808199614x + 3.7835030005927x^2 - 4.5134686256945y + 0.018234245692184y^2 + 1.0829649013353xy - 0.0043648052825903xy^2 - 0.064250707222058x^2y + 0.00025829598307936x^2y^2 + 0 \quad (3)$$

Figure 5. Response surface plot based on (A) linear and (B) quadratic regression models and (C) desirability function for methylene blue removal by Galena chitosan as a function of pH and contact time.



Source: CARVALHO C. M. D. et al.

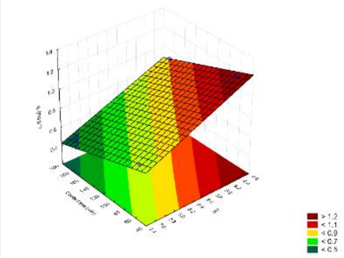
When applying the adsorption desirability function for methylene blue (Figure 5C), it was observed that the ideal adsorption pH is 9.5 for a contact time of 60 min. The desirability function was approximately 0.99, with a removal of about 76%.

For crystal violet, it was found that the best pH level was −1 (pH 7.5) and the best contact time was 120 min. The curvature was significant, indicating the need to use the quadratic regression model. The model had an adjusted R^2 of 0.969, being lower than that of the linear regression model (0.974). All factors were significant in the quadratic model, and pH had a major influence on adsorption capacity. Response surface methodology was applied using the quadratic regression model (Equation 4) to predict the desirability of the system.

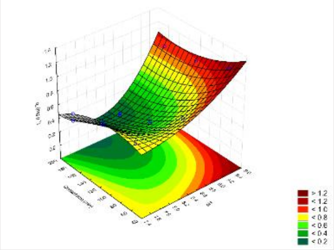
$$q_e = -11.478662523046 + 2.507400005467x - 0.11935000032124x^2 + 0.52415375045327y - 0.001564977432421y^2 - 0.11975541677419xy + 0.00034961805599806xy^2 + 0.006707500006318x^2y - 0.000019187500026001x^2y^2 + 0 \quad (4)$$

The desirability of crystal violet adsorption (Figure 6C) was close to 1, with a value of 0.99 at pH 7.5 and a contact time of 120 min. Crystal violet removal was found to be 70% with an adsorption capacity of 1.2 mg g^{-1}.

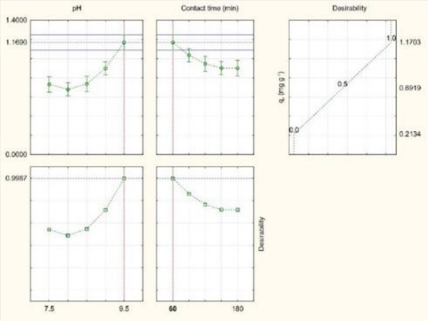
Figure 6. Response surface plot based on (A) linear and (B) quadratic regression models and (C) the desirability function for crystal violet removal by Galena chitosan as a function of pH and contact time.



A



B



C

Source: CARVALHO C. M. D. et al.

Conclusion

Chitosan was characterized by FTIR and its pH_{pzc} was found to be 6.9. An experimental design was applied, and the adsorption process was optimized by response surface methodology, as supported by ANOVA. Methylene blue removal reached 77.5% in 60 min of reaction at pH 9.5. pH had a greater influence on the variation of adsorption capacity than contact time. The desirability function was approximately 0.99, with an adsorption capacity of 1.86 mg g^{-1}. For crystal violet, the results showed that both pH and contact time influenced adsorption; 69% dye removal was achieved at pH 7.5 and 120 min of reaction. The highest desirability value (0.99) was achieved at pH 9.5 and 60 min of reaction, with a removal of 69.03% and an adsorption capacity of 1.2 mg g^{-1}. Chitosan demonstrated good adsorption capacity, removing up to 70% methylene blue and crystal violet.

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