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Adsorption study with NaOH chemically treated soybean hull for textile dye removal

The activities of the textile industry generate effluents containing high chemical load, due to the presence of toxic dyes, causing water contamination. Adsorption is a promising technique for the removal of effluent dyes, however, studies are needed to look for alternative adsorbent materials, aiming at low cost and removal efficiency. The soybean hull (Glycine max) is an agroindustrial residue widely generated in Brazil, but its application is only for animal feed. The objective of this work was to evaluate the adsorption potential of chemically treated soybean hull with sodium hydroxide (NaOH) in the removal of 5G reactive blue dye. The biosorbent was subjected to NaOH treatment at different concentrations (0.01, 0.1 and 0.5 mol L-1), influence of temperature (30, 40 and 50 ºC), rotation speed (30, 60 and 90 rpm) and pH (1 to 11) was verified. The kinetics and equilibrium isotherms were performed using the best conditions obtained in the preliminary tests, and the experimental data adjusted to the theoretical models previously described in the literature. The studied variables indicated that the highest removal (about 88%) occurred under the conditions of 0.01 mol L-1 NaOH concentration, 50 ºC, 90 rpm and pH 2. In the kinetics, it was observed that the equilibrium was achieved in about 240 min, with the best fit of the pseudo-second order model. In isotherms, the Langmuir model better predicts experimental data, predicting a maximum adsorption capacity of 16.46 mg g-1. The soybean hull was effective in removing dyes in aqueous solution, however, NaOH treatment showed no relevant improvement in adsorptive capacity.

Keywords: Biosorbent; Agroindustrial Waste; Reactive Dye; Chemical Treatment.

Estudo da adsorção com casca de soja tratada quimicamente com NaOH para remoção de corantes têxteis

As atividades da indústria têxtil geram efluentes contendo alta carga química, devido à presença de corantes tóxicos, causando contaminação da água. A adsorção é uma técnica promissora para a remoção de corantes efluentes, no entanto, são necessários estudos para procurar materiais adsorventes alternativos, visando baixo custo e eficiência na remoção. O casco da soja (Glycine max) é um resíduo agroindustrial amplamente gerado no Brasil, mas sua aplicação é apenas para ração animal. O objetivo deste trabalho foi avaliar o potencial de adsorção do casco de soja tratado quimicamente com hidróxido de sódio (NaOH) na remoção do corante azul reativo 5G. O biossorvente foi submetido ao tratamento com NaOH em diferentes concentrações (0,01, 0,1 e 0,5 mol L-1), influência da temperatura (30, 40 e 50 ºC), velocidade de rotação (30, 60 e 90 rpm) e pH (1 a 11) foi verificado. As isotérmicas de cinética e equilíbrio foram realizadas utilizando as melhores condições obtidas nos testes preliminares, e os dados experimentais ajustados aos modelos teóricos descritos anteriormente na literatura. As variáveis ??estudadas indicaram que a maior remoção (cerca de 88%) ocorreu nas condições de 0,01 mol L-1 de NaOH, 50 ºC, 90 rpm e pH 2. Na cinética, observou-se que o equilíbrio foi alcançado em cerca de 240 min, com o melhor ajuste do modelo de pseudo-segunda ordem. Nas isotermas, o modelo de Langmuir prediz melhor os dados experimentais, prevendo uma capacidade máxima de adsorção de 16,46 mg g-1. O casco de soja foi eficaz na remoção de corantes em solução aquosa; no entanto, o tratamento com NaOH não apresentou melhora relevante na capacidade adsortiva.

Palavras-chave: Biossorvente; Resíduos Agroindustriais; Corante Reativo; Tratamento químico.

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INTRODUCTION

The disordered growth of the population and industrial activities has contributed to the increasingly of environmental contamination, causing changes in soil, air and water quality (PIZATO et al., 2017). The textile industries are major consumers of water and synthetic dyes, generating complex effluents, due to the high content of inorganic salts and organic load (ZHOU et al., 2014). The resistance of these compounds to conventional treatments and the products generated by incomplete degradation result in new molecules with potential mutagenic or harmful effects (GAYLARDE et al., 2005). Synthetic dyes are included in the category of emerging pollutants, which are defined as any chemical substance that has not been attached to monitoring programs or legislation regarding environmental quality but are constantly being introduced into the environment due to anthropic activities (HORVAT et al., 2012).

In this sense, adsorption is a successfully used dye removal technique, relating high efficiency, simple operation and low cost depending on the adsorbent used (DOTTO et al., 2011). Still, in some cases it allows the recovery of the dye without the loss of its chemical identity, because it is a non-destructive method. Agricultural residues are being widely studied for adsorption, because they are cheaper than the commercial adsorbents available as activated carbon and resins, and yet they can be used on a large scale (TANYILDIZI, 2011). In this sense, Brazilian soybean production between 2018 and 2019 was approximately 115.1 million tons, with a soybean hulls generation of approximately 9.5 million tons (CONAB, 2019). Among these residues, the soybean hulls (*Glycine max*) stand out, which have in its composition about 52% of cellulose, favoring the adsorption process, since, in contaminants such as dyes, they react with cellulose and the fibers present in the biosorbent, however, this low cost agro-industrial residue has been limited to use in animal feed (KARUPPUCHAMY et al., 2009; AL-DEGS et al., 2007; CASSALES et al., 2011).

Pretreatments can still be employed to improve the adsorption capacity of soybean hulls such as alkaline (with NaOH or KOH), for example, because of their ability to break lignin and xylan crosslinked ester bonds, providing cellulose and hemicellulose enriched fractions, as well as remove alkaline soluble substances such as lignin and pectin, and other soluble impurities that cover the outer surface of the fiber cell wall (MCINTOSH et al., 2011; MAHECHA et al., 2015; HORN et al., 2012; NG et al., 2015).

Therefore, the present study aimed to evaluate the potential of soybean hulls chemically treated with sodium hydroxide (NaOH) in the removal of textile dye. The effect of NaOH concentration on soybean hull treatment on adsorption capacity was verified. Adsorption was performed under different conditions of pH, agitation and temperature. In addition, adsorption isotherms and adsorption kinetics were evaluated by theoretical models.

MATERIALS AND METHODS

Obtaining and preparation of biosorbent

The soybean hulls that were used as biosorbent came from the city of Rondonópolis, Mato Grosso, Brazil. After collection, the biosorbent was subjected to convective drying (CIENLAB, CE-220) with forced air

circulation at 60°C for 24 hours and crushed in a hammer mill (SPLabor, SP-33), with a particle size of 20 mesh. The ground soybean hulls were also submitted to chemical treatment with sodium hydroxide (NaOH) in concentrations of 0.1, 0.5 and 0.01 mol.L^{-1} for 30 min under agitation, washed with distilled water and dried again at 60°C for 24 hours.

Preparation of 5G reactive blue dye solutions

Initially, a 500 mg L^{-1} stock solution of 5G reactive blue dye with 98% purity was prepared to be used in the dilutions of the other concentrations studied. In addition, a 100 mg L^{-1} solution was used to determine the maximum absorption length in a UV/VIS spectrophotometer (Varian, Cary 50 Scan), which was obtained at 610 nm. In order to obtain the calibration of the curve, dye solutions were prepared with concentrations from 0 to 50 mg L^{-1} with an interval of 5 mg L^{-1}. The pH of the dye solutions was adjusted by adding solutions of 0.1 mol L^{-1} HCl or NaOH, when necessary.

Adsorption experiments

The experimental adsorption conditions were obtained through preliminary tests to investigate the variables temperature (30, 40 and 50 °C), rotation speed (30, 60 and 90 rpm), pH (adjusted solutions from 1 to 11). These were performed in triplicate, using 50 mL of 5G blue reactive dye solution at a concentration of 100 mg L^{-1}, with approximately 0.3 g of chemically treated NaOH soybean hulls deposited in 125 mL conical flasks, remaining in a Shaker incubator (SPLabor, SP-222) under constant agitation for approximately 120 min. At the end of the assays, the samples were submitted to the centrifugation process, followed by spectrophotometric reading (Bel, SP-2000), and the dye concentration was determined by Equation 1:

$$q = \frac{V(C_0 - C)}{m} \quad (1)$$

Where q the amount of dye adsorbed per gram of water bath root (mg g^{-1}), C_0 and C dye concentration in the initial and final aqueous solution respectively (mg L^{-1}), V the volume of dye solution (L) and m the mass of biosorbent (g).

Adsorption isotherm and kinetic

The isotherms and adsorption kinetics were conducted under the conditions previously defined in the preliminary batch tests and conducted in triplicate. The kinetics were performed with 50 mL of 5G blue reactive dye solution at a concentration of 100 mg L^{-1} and 0.3 g of biosorbent in 125 mL conical flasks at 50°C, 90 rpm and pH=2. The equilibrium isotherms were constructed using 50 mL blue reactive dye solution of 5G with 300 mg L^{-1} at 50°C, 90 rpm and pH=2, varying the biosorbent mass from 0.02g to 0.80g. The equilibrium was defined when the concentration of the dye showed standard error between its measurements of less than 5% in 3 measurements at 1 hour intervals.

Kinetic and isotherm analysis

The equilibrium kinetics and isotherms were analyzed according to the models shown in Table 1. The Pseudo-primary order and Pseudo-second order models were applied to represent the adsorption kinetics,

according to equations 2 and 3. The adsorption isotherms were evaluated by the Langmuir, Freundlich, Redlich-Peterson and Sips models, according to equations 4 to 7. The parameters of the models were calculated using the software Origin® version 15, by minimizing the objective function sum of the squares (PICCIN et al., 2016).

Table 1: Equations of kinetic and equilibrium models.

Model	Equation		Parameters
Kinetic models			
Pseudo-first order	$q(t) = q_1(1 - e^{-k_1 t})$	(2)	k_1 (min^{-1}) rate constant of pseudo–first order. q_1 (mg g^{-1}) theoretical value of adsorption capacity.
Pseudo-second order	$q(t) = \frac{t}{(1/k_2 q_2{}^2) + (t/q_2)}$	(3)	k_2 (g mg^{-1} min^{-1}) rate constant of pseudo–second order q_2 (mg g^{-1}) theoretical value of adsorption capacity
Isotherm models			
Langmuir	$q_e = \frac{q_m K_L C_{eq}}{1 + K_L C_{eq}}$	(4)	q_m (mg g^{-1}) maximum adsorption capacity K_L (L mg^{-1}) Langmuir constant
Freundlich	$q_e = K_F C_e^{1/n}$	(5)	K_F (mg g^{-1}) (mg L^{-1})$^{-1/n}$ Freundlich constant $1/n$ (-) heterogeneity factor
Redlich-Peterson	$q_e = \frac{K_{RP} C_e}{1 + \alpha C_e^{\beta}}$	(6)	K_R (L g^{-1}) Redlich-Peterson constant α (mg L^{-1})$^{-\beta}$ Redlich-Peterson constant β (-) heterogeneity factor
Sips	$q_e = \frac{q_m K_S C_e^m}{1 + K C_e^m}$	(7)	q_m (mg g^{-1}) maximum adsorption capacity K_S (L mg^{-1})$^{-m}$ Sips constant m (-) heterogeneity factor

RESULTS AND DISCUSSION

Adsorption Experiments

Figures 1 (a), (b), (c) and (d) show the influence on the adsorption process of the variable's chemical treatment, temperature, rotation speed and pH, respectively. Analyzing Figure 1 (a) it is observed that the increase in NaOH concentration caused a reduction in the adsorption potential, evidenced in the concentrations of 0.1 and 0.5 mol L^{-1}, only concentration of 0.01 mol L^{-1}, provided a slight increase in dye removal (87.80%) compared to the control sample. However, the use of 0.01 mol L^{-1} NaOH concentration for the treatment of biosorbent was chosen.

Marshall et al. (1996) demonstrated that 0.1 mol L^{-1} NaOH-treated soybean hulls adsorbed 26% more zinc (II) ions than untreated hulls. This difference in the improvement of the adsorptive process compared to the present study may occur because the chemical composition of soybean hulls varies according to processing technology, plant genetics and growing conditions (CASSALES et al., 2011). Thus, suggesting that the soybean hull employed has low interfering content, such as lignin, for example, showing no significant improvements in the adsorption process using NaOH.

Figure 1 (b) and 1 (c) show that the higher temperature (50ºC) and rotation speed (90 rpm) influenced the adsorptive process, with an increase in the dye removal rate under study, around 94%. Lee et al. (1997) explain that this occurs because the increase in agitation causes greater transfer of convective mass of the solute present in the solution, besides reducing the thickness of the film around the adsorbent surface area, promoting a higher adsorption rate. In the pH effect test of the 5G blue reactive dye solution on the adsorption by the soybean hulls chemically treated with NaOH, as shown in Figure 1 (d), pH 2 showed higher adsorption capacity, with a removal percentage close to 93%. Módenes et al. (2015) explain that the

structural stability of the blue reactive dye 5G can be affected in acid conditions, since the dye can be deprotonated in the solution medium, thus originating a polar molecule (R-SO3), with a high density of negative charge. Thus, the electrostatic repulsion between the adsorbent site and the negatively charged dye ions was reduced at low pH.

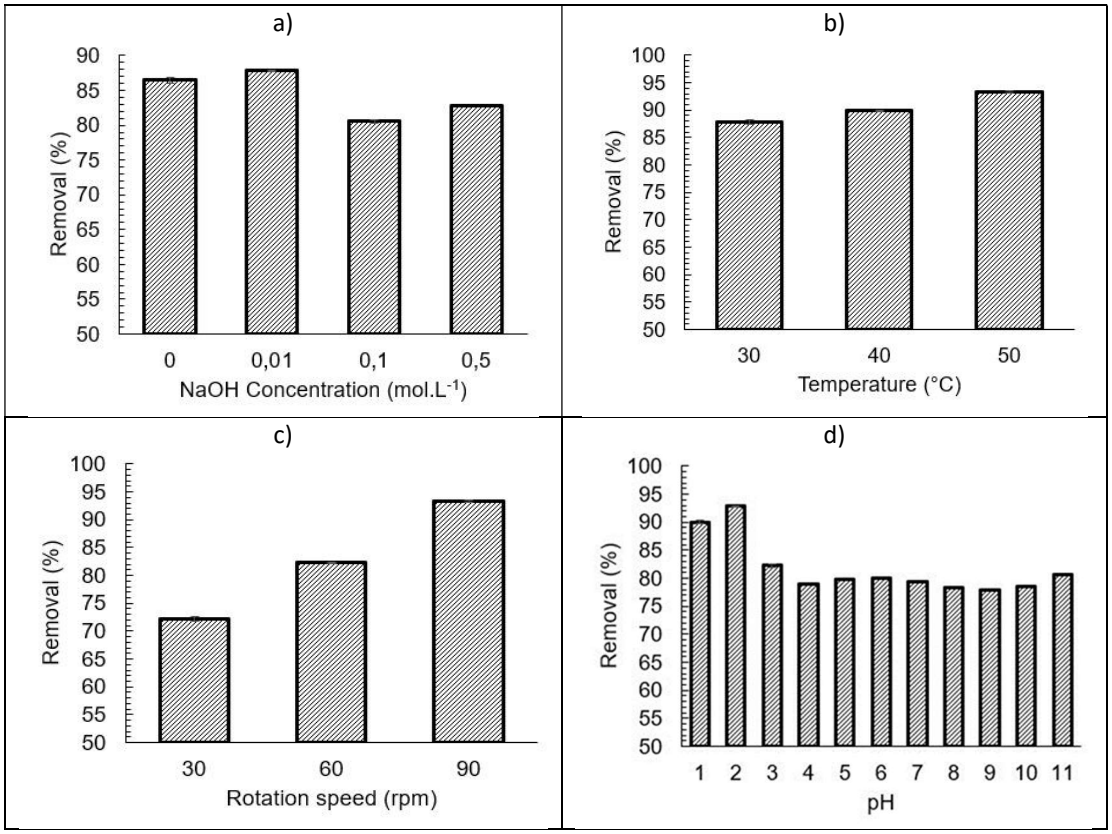

Figure 1: Influence of variables on the adsorption process (a) NaOH concentration, (b) temperature, (c) rotation speed and (d) pH.

Adsorption kinetics

Adsorption kinetics are shown in Figure 2. In this test, after 240 min the equilibrium was observed, and about 88% of the 5G blue reactive dye was removed from the aqueous solution. The kinetic parameters of the proposed models (Equations 2 and 3) are presented in Table 2**.**

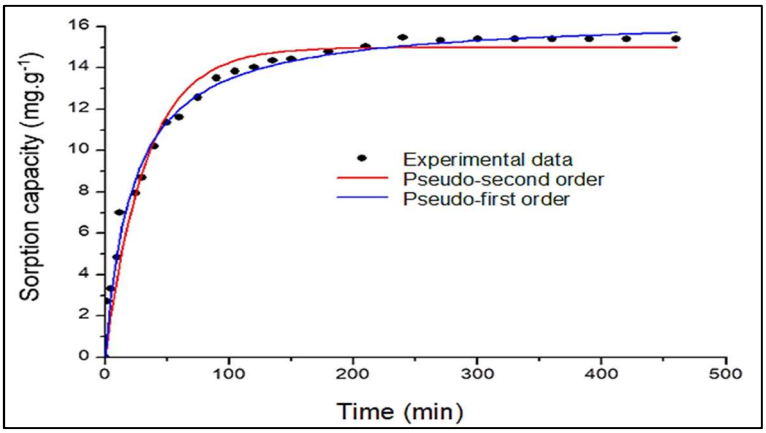

Figure 2: Kinetic profile and mathematical modeling of adsorption of 5G blue reactive dye by chemically treated soybean hulls with NaOH (50 °C, 90 rpm and pH=2).

From the analysis of the parameters of the kinetic models under study it was noted that both can be used to describe the adsorption kinetics of the present study with R^2= 0.96 and 0.98 for the Pseudo-primary and second order models, respectively. However, the Pseudo-second order model presented a better adjustment to the experimental data, as well as a higher value for solute adsorbed in the equilibrium, which in this case was 16.46 mg g^{-1} (Table 2).

Table 2: Parameters of kinetic models for 5G blue reactive dye adsorption by chemically treated soybean hulls with NaOH (50 °C, 90 rpm and pH=2).

Models	**Parameters**	**Valeu**
Pseudo-first ordem	q_1 (mg.g^{-1})	14,98
	k_1 (min^{-1})	0,03
	R^2	0,96
Pseudo-second ordem	q_2 (mg.g^{-1})	16,46
	k_2 (g.mg^{-1}.min^{-1});	0,002
	R^2	0,99

Using the soybean hulls treated with NaOH to adsorb the 5G dye, the kinetic equilibrium was reached in 240 min and the best adjusted model was the Pseudo-second order, with a quantity of solute adsorbed in the equilibrium of 16.46 mg g^{-1}. In the study by Gong et al. (2008) using soybean hulls esterified with urea to adsorb acridine orange and aniline green colorants, the kinetic data of adsorption were given in the first 120 min and were also better represented by the Pseudo-second order model. The adsorbed amount of acridine orange was 238.1 mg g^{-1} and aniline green was 178.57 mg g^{-1}, values relatively higher than in the present study.

Adsorption isotherms

The adsorption isotherm of the 5G blue reactive dye using chemically treated soybean hulls with NaOH are shown in Figure 3. It is observed that the isotherm has a concave characteristic, indicating that the adsorption process is favorable, providing high dye removal capacity type L2 or H2, according to the classification suggested by Giles et al. (1960). This type of isotherm further suggests that the adsorbent has several finite sites, which when filled no additional adsorption can occur, leading to the formation of a plateau. In this case, the plateau was observed around 43 mg g^{-1}.

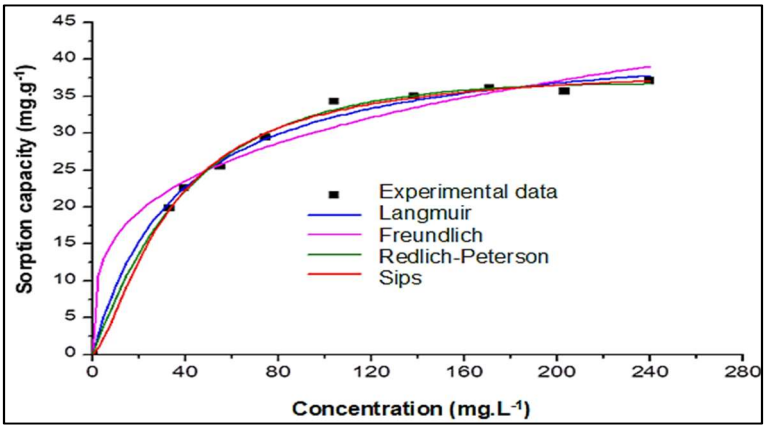

Figure 3: Equilibrium isotherm and mathematical modeling of 5G blue reactive dye adsorption by chemically treated soybean hulls with NaOH (50 °C, 90 rpm and pH=2).

Table 3 shows the adjustment of the equilibrium data to the adsorption isothermal models represented by Equations 4 to 7. The coefficients of determination (R^2) of Langmuir, Freundlich, Redlich-Peterson and Sips models equal to or near to 0.99 show that these models are suitable for representing the experimental data of adsorption of 5G blue reactive dye adsorption by chemically treated soybean hulls with NaOH.

Table 3: Equilibrium model parameters for adsorption of 5G blue reactive dye adsorption by chemically treated soybean hulls with NaOH (50 °C, 90 rpm and pH=2).

Models	**Parameters**	**Valeu±SE (95%)**
Langmuir	q_m (mg.g^{-1})	43,5447
	b (L.mg^{-1})	0,02738
	R^2	0,99262
Freundlich	k_F (L.g^{-1})	8,35480
	n_F	3,55320
	R^2	0,97144
Redlich-Peterson	K_{RP} (L.g^{-1})	0,84347
	α_{RP} (L.mg^{-1})	0,00592
	n_{RP}	1,21129
	R^2	0,99649
Sips	q_m (mg.g^{-1})	39,1567
	k_S (L.mg^{-1})	0,03048
	n_S	1,44976
	R^2	0,99551

However, the empirical models of Redlich-Peterson and Sips incorporate in their equation's parameters of the Langmuir and Freundlich models, in which the closer the value of n is to 1 or 0, the closer it is to the Langmuir or Freundlich models, respectively. Thus, it is noted that the values of n of these models are closer to 1, tending to the Langmuir model. This can be evidenced in Figure 3, since the curves of the empirical models overlap Langmuir's, and only the Freundlich curve presents a different profile. Thus, the Langmuir model was chosen to represent the balance data of this study. Langmuir's model assumes monolayer adsorption, with all bonding sites limited with equivalent energy and no interactions, where each site can adsorb only one molecule of adsorbate (CHUNG et al., 2015).

CONCLUSIONS

Under the conditions studied in the present study, alkaline pretreatment employing sodium hydroxide in soybean hulls showed no significant improvement in textile dye removal. Moreover, the results obtained in the kinetic and equilibrium assays under the studied conditions showed that the adsorption equilibrium was obtained in about 240 min, with 88% dye removal, being the Pseudo-Second order model and the Langmuir isotherm, the mathematical models that best represented the kinetic and equilibrium data, respectively. In general, the biosorbent studied was effective to remove reactive dye.

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