EFFECT OF pH AND IMPROVEMENT IN VIOLET REMAZOL DYE REMOVAL FROM WATER BY *Eichhornia Crassipes* (water hyacinth)

EFEITO DO pH E MELHORIA NA REMOÇÃO DO CORANTE VIOLETA REMAZOL EM ÁGUA POR *Eichhornia Crassipes* (aguapé)

EFECTO DEL pH Y LA MEJORA EN REMOVAL TINTE DE REMAZOL VIOLET DEL AGUA POR *Eichhornia Crassipes* (jacinto de agua)

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Abstract: In this work water hyacinth (Eichhornia crassipes) was used as adsorbent for aqueous Violet Remazol 5R (VR 5) solutions. In order to obtain the best conditions for the removal, the pH influence, time of contact and concentration were verified. The adsorption showed itself highly dependent of the pH, being the maximum adsorption was observed pH 1.0. In the kinetic experiments, the batchwise method was used with concentrations of 250 and 1000 mg dm^{-3}, and after the time of contact the concentration was determined by spectrophotometry. An adequation of the models of pseudo-first and second order was made based on the results that were found. The second order model was the most suitable in both concentrations, with R^2= 0.999. The Langmuir, Freundlich, Sips and Temkin models were used to analyze the experimental data in its linearized mode. The Langmuir model was the most appropriate. In the conditions studied, the removal of textile dye Violet Remazol 5R by water hyacinth showed good results, making it possible to use this material as an alternative way to remove dyes from textile effluents.
Keywords: Water hyacinth (*Eichhornia crassipes*). Textile dye. Adsorption.

Resumo: Neste trabalho foi utilizado o aguapé (Eichhornia crassipes) como adsorvente de soluções aquosas de Violeta Remazol 5R (VR 5). De modo a obter as melhores condições para a remoção foram verificados, a influência do pH, tempo de contato e a concentração. A adsorção mostrou-se altamente dependente do pH, sendo que a adsorção máxima foi observada pH 1,0. Nas experiências de cinética, o método de batelada foi utilizado nas concentrações de 250 e 1000 mg dm^{-3}, e após o tempo de contato com a concentração foi determinada por espectrofotometria. Uma adequação dos modelos de pseudo-primeira ordem e segunda foi feita com base nos resultados que foram encontrados. O modelo de segunda ordem foi o mais adequado em ambas às concentrações, com R ² = 0,999. Os modelos de Langmuir, Freundlich, Sips e Temkin foram utilizados para analisar os dados experimentais em seu modo linear. O modelo de Langmuir foi o mais adequado. Nas condições estudadas, a remoção de corante têxtil Violeta Remazol 5R por aguapé mostrou bons resultados, tornando possível a utilização deste material como uma forma alternativa para remover corantes de efluentes têxteis.
Palavras-chave: Aguapé (*Eichhornia crassipes*). Corante têxtil. Adsorção.

Resumen: En este trabajo fue utilizado el jacinto de agua (Eichhornia crassipes) como adsorvente para la remoción del colorante textil aniónico Violeta de Remazol 5R de soluciones acuosas. Con el objetivo de encontrar condiciones mas adecuadas para la remoción, se verificó la influencia del pH, tiempo de contacto y concentración. La adsorción se mostró fuertemente dependiente del pH, presentando una adsorción máxima a pH 1,0. Para los experimentos de cinética se utilizou el método de lote en las concentraciones de 250 y 1000 mg dm^{-3}, y después del tiempo de contacto se realizó la determinación de la concentración atraves de espectrofotometria. Con los resultados conseguidos se hizo una adaptación a los modelos de pseudo-primera orden y segunda orden, con R^2 = 0,999. Los modelos de Langmuir, Freundlich e Temkin, fueron utilizados para analizar los datos experimentales en su forma linealizada. El modelo que major se adaptó fue el de Langmuir. Bajo las condiciones de estudio, la remoción del colorante textil aniónico Violeta de Remazol 5R por jacinto de agua mostró buenos resultados, lo que permitiria que pueda ser utilizado como una alternativa para la remoción de colorantes en efluentes textiles.
Palabras clave: Jacinto de agua (*Eichhornia crassipes*). Colorante textil. Adsorción.

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1 INTRODUCTION

The textile industries produce a lot of wastewater, which contains a number of contaminants, including acidic or caustic dissolved solids, toxic compounds, and many different dyes. The extensive use of dyes represents a concern in public health and serious environmental problems due to their persistence in nature and characteristics of non biodegradable (HU et al., 2010).

Conventional methods used for dye removal from industrial effluents include coagulation/flocculation, anaerobic degradation, photocatalysis, advanced oxidation processes (AOPs), filtration and adsorption. In addition, most of these methods are often expensive or ineffective, especially for dye removal from dilute solutions (GUPTA; SUHAS, 2009).

The utilization of adsorbents for dye removal from wastewater is an efficient treatment processes, which can be used to remove different types of materials, especially non degradable. The most commonly used adsorbent is activated carbon, however, its use has several drawbacks such as low retention capacity of bulky adsorbates and operational cost dearly (GUPTA; SUHAS, 2009).

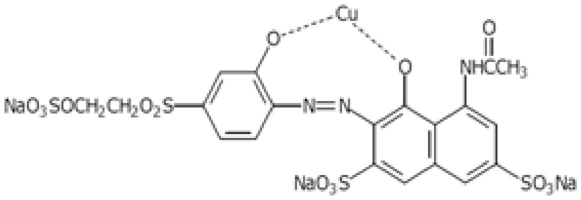
Some low-cost lignocellulosic materials have been used as biosorbents for the adsorption of cationic and anionic dye (SALLEH et al., 2011). The exploration and appropriate use of these biomaterials bring great economic and social benefits to mankind, helping to minimize environmental and energy issues. Brazil has a high potential for the production of lignocellulosic fiber that have shown many potential applications in removing pollutants (SANTANA et al., 2010; VIEIRA et al., 2010; VIEIRA et al., 2011).

Water hyacinth (Eichhornia crassipes) is one of the most abundant species in the Amazon and its distribution extends to all tropical regions of the planet (SCULTHORPE, 1985). Water hyacinth grows rapidly on the surface of waterways, forming a dense mat which depletes the surrounding environment of essential nutrients. These properties, rarely encountered in other plant systems, are features of an ideal feedstock for renewable biomass (HRONICH et al., 2008). The same plant has demonstrated an amazing ability to absorb and concentrate many toxic metals from aquatic environments. Consequently, research activity on utilization of the plant has been registered over the last few decades (ABDEL-SABOUR, 2010; CHEN et al., 2010; MAHAMADI, 2011). In recent years, was studied the utilization of water hyacinth in the removal of color from wastewater of textile dyeing processes, for cationic dye (TARAWOU; HORSFALL; VICENTE, 2007; EL-KHAIARY, 2008; RENGANATHAN et. al., 2008). Thus, the purpose of this paper is to characterize by FTIR spectral analysis and to investigate the efficiency of water hyacinth as adsorvent in removal of anionic textile dye Violet Remazol 5R in aqueous solution, contributing to the use of this material useful for the removal of pollutants in aquatic environments.

2 MATERIALS AND METHODS

The dye Violet Remazol 5R (VR 5) was provided by Indústria Toalhas São Carlos, state of São Paulo, Brazil. The chemical structure is shown in figure 1. The dye was chemical product of analytical grade and was used without further purification.

Figure 1 - Chemical structure of dye Violet Remazol 5R (VR 5)



Source: Costa et al. (2009)

The water hyacinth was acquired in the city of São Luís, Maranhão state, Brazil. The material was washed thoroughly with water to for several times to remove matter and all dirt particles. It was then dried at 50 ºC for 24 hours. The dried material was then crushed, sieved in the 0.088 to 0.177 mm range.

Infrared spectrum was obtained in the 4000 to 400 cm^{-1} range by accumulating 32 scans by Fourier transform infrared spectroscopy (FTIR), on a MB-Bomem spectrophotometer, using KBr pellets with a resolution of 4 cm^{-1}. Spectroscopy in the visible region was obtained in a quartz cell with path length of 1.0 mm. The spectra were recorded on a Cary 50 Varian spectrophotometer apparatus at 540 nm, maximum wavelength for VR 5.

2.1 Point of zero charge

The point of zero charge of water hyacinth was determined by the solid addition method (SANTANA et al., 2010). To a series of 100 cm^3 of conical flasks were transferred 25.0 cm^3 of 0.1 mol dm^{-3} KCl aqueous solution, with pH varying of 1 to 12. The initial pH value (pHi) of each solution was adjusted by adding either 0.10 mol dm^{-3} of hydrochloric acid or sodium hydroxide. The pH measurements were obtained using a DM-21 Digimed instrument. The pHi's of the solutions were then accurately measured and 100.0 mg of water hyacinth was added to each flask. The suspensions were shaken for 24 h and then the pH values of the supernatant were measured. The difference between the initial and final pH value, (pHi − pHf), was plotted against pHi and the point of intersection of the resulting for null ∆pH corresponds to point zero charge, pHpzc.

2.2 Effect of pH

The effect of pH on the sorption was performed by an using identical series of flasks containing 100.0 mg of biomaterial with 25.0 cm^3 of the 250 mg dm^{-3} VR 5 dye solution, maintaining stirring for 24 h at 25 ºC. pH in the range from 1.0 to 6.0 was adjusted when necessary with 0.10 mol dm^{-3} hydrochloric acid or sodium hydroxide solutions. At the end of this process, the solid was separated by centrifugation and the dye concentration found in the supernatant was determined by using a Varian AA 50 spectrophotometer at a wavelength of 540 nm.

The amount of dye adsorbed, qe (mg g^{-1}), was obtained with equation 1:

(1) $$q_e = \frac{(C_i - C_f)}{W} \times V$$

Where Ci and Cf are the initial and final dye concentrations at equilibrium in the aqueous phase (mg dm^{-3}), respectively, V is volume of dye solution (dm^3) and W (g) is the amount of water hyacinth employed.

2.3 Kinetics and isotherms

For the kinetics studies 100.0 mg of adsorbent were mixed with 25.0 cm^3 aqueous dye solutions of known concentration (250 and 1000 mg dm^{-3}) at 25 ± 1 °C. The flasks were capped and the system was stirred for variable times. For all experiments the aqueous solution pH was adjusted at 2.0, using hydrochloric acid. After centrifugation, the dye was determined as previously described. To investigate the possible mechanism of sorption, some kinetic models were used: pseudo-first order, pseudo-second order kinetics and intraparticle diffusion (ACHARYA et al., 2009), represented by linear equations (2) to (4), respectively:

(2) $$\ln\,(q_e - q_t) = \ln q_e - k_1 t$$

(3) $$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e}$$

(4) $$q_t = k_{dif} t^{1/2} + I$$

where qe and qt are the amounts of dye sorbed (mg g^{-1}) on water hyacinth at equilibrium and at time t (min), and k1 (min^{-1}) and k2 (g mg^{-1} min^{-1}) are pseudo-first order and pseudo-second order rate constants, respectively; kdif (mg g^{-1} $min^{-0.5}$) is the intraparticle diffusion rate constant and I, constant related to the diffusion resistance of the adsorbed species due to the effect of interface.

The isotherms were obtained using a batchwise method, which consisted in suspending a series of 100.0 mg of water hyacinth in 25.0 cm^3 of aqueous dye solutions having different concentrations, varying from 50 to 1000 mg dm-3 under equilibrium time and optimal pH conditions, previously established. At the end of this process, the solid was separated by centrifugation and the dye amount adsorbed was determined as previously described. The isotherms of the Langmuir, Freundlich , Temkin and Sips (CHOWDHURY; SAHA, 2011; GILES; D'SILVA; EASTON, 1974; FEBRIANTO et al., 2009) models in their linear forms, equations (5) to (8) respectively, were applied to the equilibrium data of sorption of Violet Remazol 5R on water hyacinth.

(5) $$\frac{C_e}{q_e} = \frac{1}{q_m K_L} + \frac{1}{q_m} C_e$$

(6) $$\log q_e = \frac{1}{n_F} \log C_e + \log K_F$$

(7) $$q_e = \left(\frac{RT}{b_T}\right) \ln K_T + \left(\frac{RT}{b_T}\right) \ln C_e$$

$$\frac{C_e^{1/n_s}}{q_e} = \frac{1}{q_m} C_e^{1/n_s} + \frac{1}{q_m K_S}$$

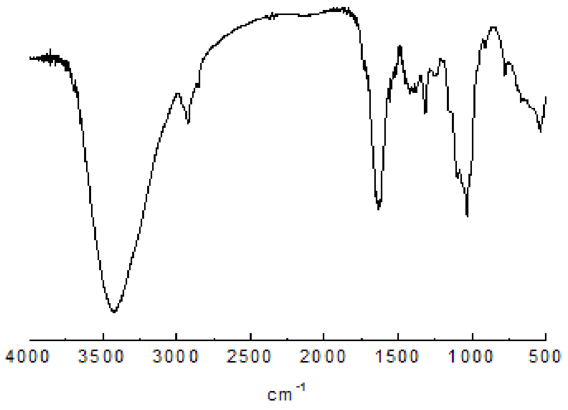
where q_e (mg g^{-1}) is the amount of dye sorbed per unit mass of sorbent, q_m is the maximum Langmuir sorption capacity (mg g^{-1}), Ce is the equilibrium concentration (mg dm^{-3}), n_F is interpreted as a factor of heterogeneity, ns is a parameter related to the characteristics of adsorption sites, K_L (dm^3 mg^{-1}), K_F [mg g^{-1} (dm^3 mg^{-1})$^{1/n}$], K^T (L g^{-1}) and K_S (L mg^{-1})$^{-1/ns}$ are the Langmuir, the Freundlich, the Temkin and the Sips constants, respectively.

3 RESULTS AND DISCUSSION

3.1 Characterization of the sorbent

Water hyacinth, as shown in Figure 2, presented bands attributed to O–H stretching at 3200 to 3600 cm^{-1}. The stretching bands of C–H and CH_2 appear in the 2900 to 3000 cm^{-1} interval and the corresponding deformation is at 1453 and 1373 cm^{-1}. The band at 1639 cm^{-1} corresponds to H–O–H bending of absorbed water while that the band at 1316 cm^{-1} is assigned to CH_2 rocking vibration. Band at 1248 cm^{-1} is attributed to C–O stretching in cellulose and hemicelluloses. The spectra show also the prominent peak with maximum near 1030 cm^{-1} due to C–O vibrations in the cellulose pyranoside units. The bands associated with tetra- and trisubstituted benzene out of plane vibrations are assigned at 913 and 774 cm^{-1}, respectively (TSERKI et al., 2005).

Figure 2 - FTIR spectrum of the water hyacinth



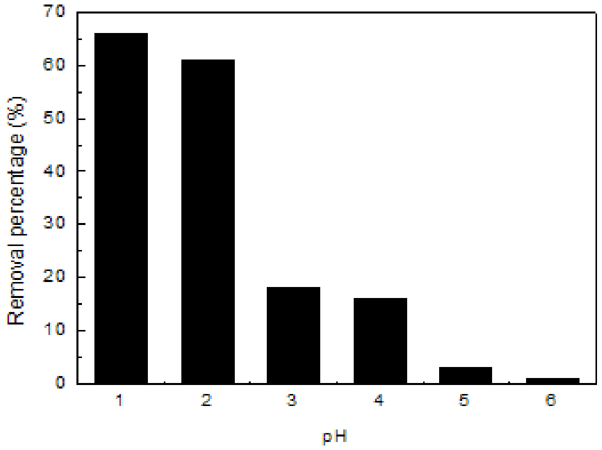
Source: developed by the authors.

3.2 pH effect

The pH of the solution affects the surface charge of the adsorbents as well as the degree of ionization and speciation of different pollutants (EL-SAID; BADAWY; GARAMON, 2010). Since the efficiency of sorption processes is strongly dependent on the pH, comparative experiments were performed at pH between 1 and 6 (Figure 3). The adsorption of the VR 5 on water hyacinth increases with the decrease in pH value and reaches maximum values at pH 1.0 (66 %) and 2.0 (61 %). Kinetic studies and adsorption isotherms were performed at pH 2.0, less drastic condition, with slight variation in results when compared to pH 1.0.

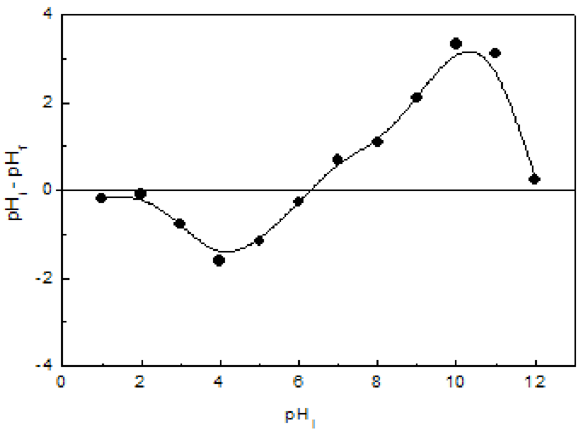
It must be noted that the surface of the adsorbent changes its polarization according to the value of the pH of the solution and to the pHpzc of the solid. At pH lower than pHpzc the surface becomes positively charged and it is the opposite for pH higher than pHpzc. As noted in figure 4, the surface of water hyacinth is protonated in values pH smaller than 6.3, favoring the adsorption of compounds with negative charge. For values of pH greater than this, the surface of the water hyacinth becomes deprotonated, favoring the attraction of positive charges. The studied dye had several functional groups such as OH- or expose negatively sulfonate SO_3^{2}- charged groups, then, it is expected that at pH lower than 6.3 to adsorption of anionic dyes is favored. The sorption profile of VR 5 on water hyacinth as a function of the initial pH could also be explained on the basis of surface charge density of functional groups and the decrease in adsorption with increase in initial pH can be attributed to the fact that the negatively charged anionic dyes are repulsed at higher pH values, which results are in agreement with earlier reports (SANTANA et al., 2010).

Figure 3 - Effect of pH on Violet Remazol sorption by water hyacinth of aqueous solution (24 hours at 25 ± 1 °C, [VR 5R] = 250 mg dm^{-3})



Source: developed by the authors.

Figure 4 - Point of zero charge (pHpzc) of water hyacinth



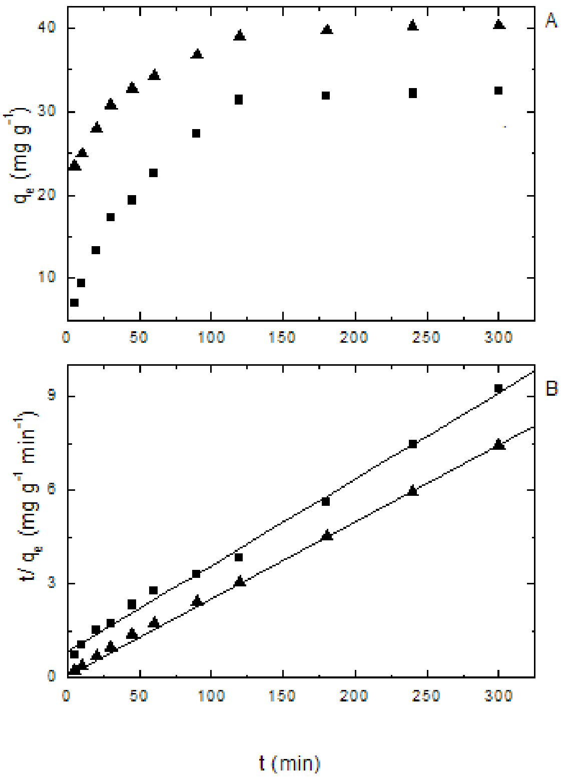
Source: developed by the authors.

3.3 Kinetics of adsorption

The adsorption capacity versus adsorption time plot is shown in figure 5(a). Equilibrium is achieved in about 120 min at 25 ± 1 °C with a dose of 100 mg of adsorbent in 25 dm^{-3} of dye solution in concentrations of 250 and 1000 mg dm^{-3}, indicating that the equilibration time is not influenced by the concentration of the dye.

The rate constant for the first-order equation was determined from the slope of the plot ln (q_e-q_t) versus time. If first-order kinetics is applicable to the system under study, the plot of ln (q^e-q_t) versus time, as represented by Eq. (2), should give a linear relationship. Further, the q_e obtained from the plot should also be close to the q_e experimentally obtained from the dye solution. Even though the correlation coefficient of the first-order equation was reasonable (R_2 = 0.980), the calculated value of qe from the first order kinetic plot was far too small (q_e = 27.6 mg g^{-1}), compared to the experimental values ($q_{e,exp}$ ≈ 40 mg g^{-1}). The second-order sorption rate constant and q_e were determined from the slope and intercept of t/q_t versus time plot (Figure 5 b) and the correlation coefficients for the linear plot are better than 0.99. The calculated q_e value from the pseudo-second-order model (q_e = 41.6 mg g^{-1}) is in good agreement with experimental q_e values. This suggests that the sorption system followed the pseudo second-order model. The applicability of the pseudo second-order model suggests that chemisorption might be the rate-limiting step that controls the adsorption processes. In general, this model has the following advantage: the adsorption capacity, the pseudo second-order rate constant and the initial adsorption rate can be determined (HO; MCKAY, 2006).

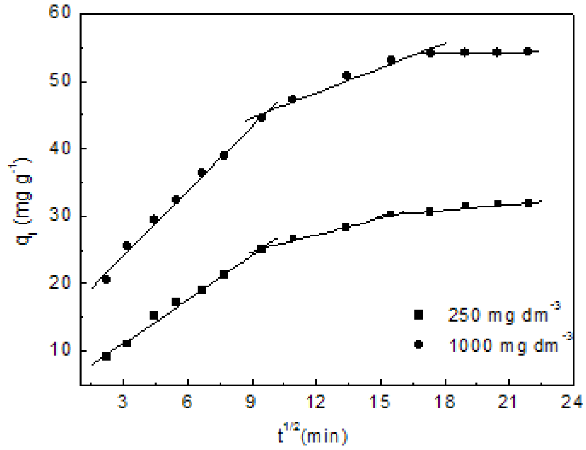
Figure 5 - Kinetics for Violet Remazol 5R (VR 5) adsorptions by water hyacinth (A) and fit the pseudo-second order models (B). (■) 250 mg dm-3; (▲) 1000 mg dm^{-3}



Source: developed by the authors.

Figure 6 shows that intraparticle diffusion adsorption VR 5 occurs in three characteristic stages. The first stage is characterized by the onset of a rapid diffusion of the adsorbate toward the outer surface of the adsorbent. The second stage is represented by a straight linear profile that presents a gradual adsorption, where intraparticle diffusion begins to decay due to the low concentration gradient of the solute, as well as a reduced availability of sites for adsorption. The third stage is characterized by a line indicating the remaining balance, as observed at both concentrations studied.

Figure 6 - Intraparticle diffusion model for Violet Remazol sorption from aqueous solution by water hyacinth



Source: developed by the authors.

By analyzing the values of intraparticle diffusion constants shows that there is a significant decrease with changing steps, the first stage> second stage> third stage (ranging from 2.18 to 0.8 and 2.19 to 0.05 for concentrations of 250 and 1000 mg dm^{-3}, respectively), indicating a gradual increase of the diffusion resistance in the interface provided between the dye and the surface of the adsorbent, which is assigned to the saturation of the adsorbents surface, the system reaching equilibrium.

Despite the high values of the linear regression coefficients for the intraparticle diffusion (R_2 ≈ 0.99), it contributes only at the beginning of the adsorption process, and the overall mechanism of the adsorption is controlled by the second-order chemical process.

3.4 Sorption isotherms

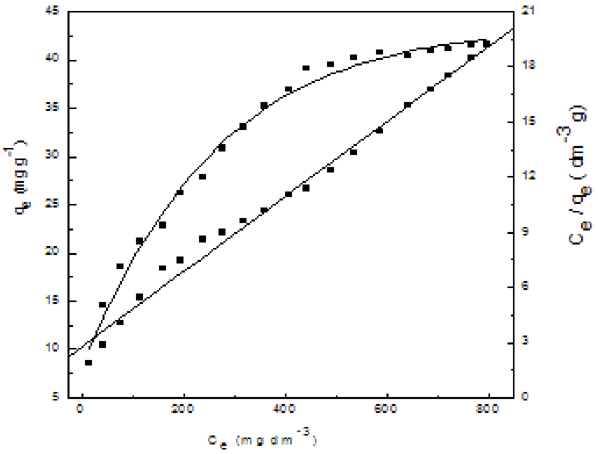
The adsorption isotherm represents the concentrations of dye sorbed at equilibrium (q_e) according to the amount adsorbed per gram of sorbent. Profiles of the isotherm of VR 5 adsorption from aqueous solution on water hyacinth is shown in figure 7. From the adsorption curves the plateau was progressively approached as the concentration investigated increased. Analyses of correlation coefficients showed that the Langmuir isotherm provided the best fit for experimental data, shown in figure 7. The Langmuir isotherm parameter qm indicates the maximum adsorption capacity of the material, in other words, the adsorption of dye at high concentration (GILES; D'SILVA; EASTON, 1974). From the correlation coefficients, R_2, we can compare the applicability of the isotherm equation. The Langmuir isotherm was obeyed better than the Freundlich, Sips and Temkin isotherms, as is evident from the values of the regression coefficients (Table 1). The Langmuir model is an indication of surface homogeneity of the adsorbent. Most of the reported dyes systems suggest the applicability of the Langmuir equation, which assumes there are monolayer coverage and constant sorption energy on the sorbent surface (GILES, D'SILVA; EASTON, 1974). The adsorption results for VR 5 on water hyacinth (48.9 mg g^{-1}) were higher than those reported for anionic dyes adsorbed on peanut hulls (15.6 mg g^{-1}) (GONG et al., 2005), wheat bran (10.5 mg g^{-1}) (ÇICEK et al., 2007), almond hulls (20.5 mg g^{-1}) (ARDEJANI et al., 2008) and Brazil nuts (7.81 mg g^{-1}) (BRITO et al., 2010), which suggests that this biomaterial have high performance for dye removal at the solid/liquid interface.

The results from the Temkin model presented a poor linear fit for VR 5 dye adsorption on water hyacinth. A summary of the parameters calculated from the models are listed in table 1. Based on the correlation coefficients, the Langmuir, Freundlich and Sips models presented the values very close, in adsorption dye on water hyacinth. But when comparing the amount adsorbed obtained experimentally and calculated, we can identify a relative closeness between the values. Furthermore, Langmuir isotherm model was observed for adsorption of dyes in various biosorbent, such as palm kernel fiber (OFOMAJA; HO, 2007) thuja (AKAR et al., 2008) and cottonseed hulls (TUNÇ; TANACI; AKSU, 2009).

4 CONCLUSION

Our study showed that the amount of dye adsorbed by the water hyacinth, an aquatic plant macrophyte, was found to vary with initial Violet Remazol 5R concentration, pH and contact time. Also, the decrease in pH favored the adsorption process, reaching a maximum value of 66 %, that is justified by the point of zero charge (pHpzc = 6.3). By adsorption data was found to follow the pseudo second order kinetics at room temperature and the Langmuir model adsorption showed maximum adsorption 48.9 mg g-1 at pH 2.0. FTIR analysis revealed structural features such as O–H and C–H stretching that are characteristic of the chemical fractions of the materials, which can sorb Violet Remazol 5R dye in a process that depends on pH. In view of these results, which indicate the potential of this material for removing dye in water, we suggest their application to adsorption of other dyes, as well as actual samples, and changes in surface for the incorporation of new active sites for specific applications.

Figure 7 - Isotherms and Langmuir adsorption for Violet Remazol 5R onto water hyacinth in aqueous solutions at 25 ± 1 °C and pH 2



Source: developed by the authors.

Table 1 - Isotherm data for Violet Remazol 5R, using water hyacinth as sorbent at 25 ± 1 °C and pH 2. qm (mg g^{-1}) = maximum Langmuir and Sips sorption capacity; K_L (dm^3 mg^{-1}), K_F [mg g^{-1} (dm^3 mg^{-1})$^{1/n}$], KS (L mg-1)-1/ns = Langmuir, Freundlich, Sips and Temkin constants, respectively; n_F and n_s (dimensionless) = terms used to estimate the heterogeneity for the Freundlich and Sips models, respectively, where a value that deviates from unity indicates heterogeneity of a material; R_2 = correlation coefficient.

Parameter	Langmuir	Freundlich	Sips	Temkin
q_m	48.9	-	51.5	-
K_L	0.007	-	-	-
n_F	-	2.49	-	-
K_F	-	3.12	-	-
n_S	-	-	120.2	-
K_S	-	-	0.011	-
A	-	-	-	1.55
b	-	-	-	259.7
R^2	0.989	0.982	0.986	0.964

Source: developed by the authors.

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