REMOVAL OF BASIC FUCHSINE FROM AQUEOUS SOLUTION BY ADSORPTION ON AN ALTERNATIVE ZSM-5 ZEOLITE.

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ABSTRACT: Dyes are the major compound of the textile effluents. This work aimed to analyze the Basic Fuchsine (BF) adsorption onto ZSM–5 zeolite synthetized using chitin as template. The zeolite synthesis was performed by means of a nucleating gel as structure–directing agent and the obtained zeolite was characterized. The potential of ZSM–5 to adsorb BF was evaluated in batch mode, by studies of adsorbent dosage, pH, kinetic, equilibrium and thermodynamic. The chitin provided some attractive characteristics on the ZSM–5, like mesopores volume of 0.142 cm³ g⁻¹ and pore diameter of 22.49 nm. The adsorption was favored with adsorbent dosage of 2.0 g L⁻¹ and the optimum pH was 9.0. The kinetic profile was represented by homogenous surface diffusion model (HSDM) and the equilibrium was in accordance with the Sips model. The adsorption was spontaneous, favorable and endothermic.

KEYWORDS: Basic Fuchsine; Adsorption; Zeolite.

1. INTRODUCTION

Dye removal is the motive of many studies because water pollution from textiles activities is a persistent problem. The color of the textile wastewater is usually due the dyeing step. To color acrylic fibres is applied cationic dyes, whose Basic Fucsine is an example. BF is a triphenylmethane dye, it is widely used as coloring agent for textile and leather materials (Gupta et al., 2008).

Diverse technologies can be applied to treat textile effluents and the adsorption is one of these. Zeolites have favorable characteristics to adsorption application like: high specific surface area, negatively charged lattice, selectivity, and also their relatively cheap prices (Brião et al., 2017), but zeolites also display diffusion limitations for transport of reagents with size similar to the size of the micropores (Perez-Ramirez et al., 2008). In order to enjoy the advantages of zeolites and overcoming the above mentioned limitations regarding dye adsorption, it is necessary a change in the porosity of this material, so the use of chitin (a low cost and available natural biopolymer) as template can be an alternative.

Therefore, this work aimed to improve the structural characteristics and BF dye adsorption potential ZSM–5 zeolite, through the insertion of chitin as template agent in the synthesis route.

2. MATERIALS AND METHODS

2.1. Reagents

The analytical grade reagents H₂SO₄ (95.0 %), SiO₂ (Aerosil) (0.2–0.3 μm), Al₂(SO₄)₃, NaOH (99.0%) and TPAOH (20% v/v) were purchased from Sigma Aldrich (Germany) and VETEC (Brazil). Chitin (deacetylation degree of 44±1%, particle size of 75 μm, crystallinity index of 86±1%) was obtained from shrimp wastes (Penaeus brasiliensis) by demineralization, deproteinization, deodorization, drying and milling steps, according Dotto (2016).

Basic Fuchsine (color index 42500, molar weight of 337.85 g mol⁻¹, λmax=540 nm, purity of 99.0%) was purchased from INLAB Ltda. (Brazil). The structural formula of BF is presented in Figure 1. All other reagents utilized were of analytical grade. Deionized water was used to prepare all solutions.
2.2 Synthesis and characterization of chitin/ZSM-5 zeolite

Chitin/ZSM–5 was synthesized with based procedure on a previous work (Stamires, 2006). Firstly, the nucleating gel was prepared containing the molar composition as follows: 1 SiO$_2$: 0.3 Na$_2$O: 0.05 TPA$_2$O: 24 H$_2$O: 0.3 OH$^-$ . After, the mixture was charged into a PTFE lined stainless autoclave, and aged for 7 days at 60 °C resulting in a gel solution (solution A). Secondly, the precursor gel (solution B) for the synthesis of chitin/ZSM–5 zeolite was prepared using the molar composition as follows: 1 SiO$_2$: 0.033 Al$_2$O$_3$: 0.6 Na$_2$O: 0.001 TPA$_2$O: 25 H$_2$O: 0.2 OH$^-$ . Then, an amount of 1% wt. of solution A was placed into the solution B under magnetic agitation for some minutes, resulting in a mixture with TPAOH/SiO$_2$ molar ratio of 0.001. The proportions of chemical compounds above mentioned were used in order to obtain a ZSM-5 zeolite containing SiO$_2$/Al$_2$O$_3$ ratio of 30 (Oliveira, 2016). In order to increase the porous size, 4.0% wt. of chitin was added on the mixture. The resulting mixture was homogenized during 30 min using an ultrasound equipment (UP200S, Hielscher, Germany) of 200 W equipped with a titanium sonotrode during 1 h at 24 kHz. The respective mixture was charged into PTFE–lined stainless autoclaves and submitted to a hydrothermal treatment at 170 °C for 24 h. After, the powders were separated using filtration, washed with distilled water and dried at 110 °C for 12 h. Then, the material was calcinated at 600 °C for 5 h in a muffle furnace under oxidizing atmosphere, in order to remove the chitin by combustion reaction, generating cavities on the chitin/ZSM–5 structure.

Chitin/ZSM–5 zeolite was characterized according to the following methods: FT–IR (Prestige, 21210045, Japan) to identify the functional groups; XRD (Rigaku, Miniflex 300, Japan) to determine the amorphous/crystalline structure and also the crystallinity index; volumetric adsorption analyzer (Quantachrome Instruments, New Win 2, USA) to determine the specific surface area, pore volume, average pore radius and porosity using the BET and BJH methods; SEM (Jeol, JSM–6610LV, Japan) to visualize the surface modifications caused by the addition of chitin.

2.3 Batch adsorption experiments

Typical batch adsorption experiments were performed to evaluate the BF removal capacity of chitin/ZSM-5 zeolite. A stock solution (1.0 g L$^{-1}$) was prepared with deionized water and all subsequent experiments were made by diluting this solution. The experiments were carried out in a thermostated agitator (Marconi, MA 093, Brazil) using Erlenmeyer flasks and, the experimental conditions were determined by preliminary tests. For all experiments the volume of solution was 50.0 mL.

Firstly, the adsorbent dosage effect was investigated from 0.5 to 2.0 g L$^{-1}$, at the original solution pH, temperature of 298K, agitation of 200 rpm, contact time of 2 h and initial concentration of 50.0 mg L$^{-1}$. After, to evaluate the pH effect on the dye removal, the same conditions were maintained, but the pH was adjusted from 2.0 to 10.0 with 0.1 mol L$^{-1}$ NaOH or HNO$_3$ and, the zeolite dosage was obtained in the mass effect test. Then, kinetic curves were constructed using the more adequate values of adsorbent dosage and pH (above determined), with contact time from 0 to 120 minutes at initial dye concentration of 50.0 mg L$^{-1}$, 298 K and 200 rpm. Finally, equilibrium isotherms were obtained at different temperatures (298, 308, 318 and 328 K) with dye concentration range from 0.0 to 300.0 mg L$^{-1}$, the best zeolite dosage, ideal pH and the optimum time.

For all experiments, samples were collected, centrifuged (Centribio, 80–2B, Brazil) at 4000 rpm for 20 min, and the remaining BF concentration in liquid phase was determined by spectrophotometry at the maximum wavelength of absorption (540 nm) using a spectrophotometer Biospectro SP–22 (Brazil). The experiments were carried out in triplicates and blanks were performed. The dye removal percentage (%R), equilibrium adsorption capacity ($q_e$) and adsorption capacity at any time ($q_t$) were determined by equations 1–3, respectively:
where, $C_0$ is the initial dye concentration in liquid (mg L$^{-1}$), $C_e$ is the equilibrium dye concentration in liquid (mg L$^{-1}$), $C_i$ is the dye concentration in liquid at any time (mg L$^{-1}$) $m$ is the amount of adsorbent (g) and $V$ is the volume of solution (L).

### 2.4. Mass Transfer and Equilibrium Models

Generally, in solid–liquid adsorption systems it is accepted that the mass transfer can occur in three steps: external mass transfer, intraparticle diffusion and adsorption on active sites. The intraparticle diffusion may be controlled by pore volume diffusion, surface diffusion or a combination of both mechanisms (Ruthven, 1984 and Qiu et al., 2009). When mass transfer resistance is internal, intraparticle diffusion controls the process. In this case considering diffusivity constant, particle amorphous, homogeneous and spherical, and the external resistance being negligible, the adsorption process can be represented by homogenous surface diffusion model (HSDM), Equation 4:

$$\frac{\partial q}{\partial t} = \left(D_p \frac{\partial^2 q}{\partial r^2} + \frac{2 \partial q}{\partial r} \right)$$

(4)

Using appropriate boundary and initial conditions, and considering a linear isotherm between initial and final concentration, for finite volume process (Crank, 1975) developed a solution that can be approximated to the first term of series when the Fourier number is higher than 0.2, Equation 5:

$$\frac{q}{q_e} = 1 - \left[ 6a(\alpha + I) \exp \left( - \frac{q^2D_pl}{R_p^2} \right) \right]$$

(5)

where $D_p$ is the intraparticle diffusivity (m$^2$ min$^{-1}$), $\alpha$ is the effective volume ratio, expressed as a function of the equilibrium partition coefficient (solid/liquid concentration ratio) and is obtained by the ratio ($C_i/C_0 - C_e$) and $q_n$ represents the non-zero solutions of Equation 6.

$$\tan q_n = \frac{3q_n}{3 + aq_n^2}$$

(6)

Freundlich, Langmuir and Sips isotherms were used to fit the equilibrium data. The choice of these models was based on the type of equilibrium curves obtained for the systems BF/ZSM-5. The Freundlich isotherm (Equation 7) assumes that the adsorption occurs on a heterogeneous surface. The Langmuir isotherm model (Equation 8) adopts a monolayer adsorption onto a homogeneous surface. The Sips isotherm (Equation 9) is a combination of the Langmuir and Freundlich isotherms.

$$q_e = k_F C_e^{1/n_F}$$

(7)

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

(8)

$$q_e = \frac{q_m S (k_s C_e)^m}{1 + (k_s C_e)^m}$$

(9)

where, $k_F$ is the Freundlich constant (mg g$^{-1}$) (mg L$^{-1})^{-1/n_F}$, $1/n_F$ is the heterogeneity factor, $q_m$ and $q_{mS}$ are the maximum adsorption capacities (mg g$^{-1}$) and $k_s$ and $k_S$ is the Langmuir constant (L mg$^{-1}$) and Sips equilibrium constant, respectively.

The kinetic and equilibrium parameters were determined by adjust of the models with the experimental data through nonlinear regression. The estimation was based on the minimization of the least squares function by means of the Statistic 9.1 software (Statsoft, USA). The fit quality was measured through determination coefficient ($R^2$) and average relative error (ARE).

### 2.5. Thermodynamic parameters estimation

The estimation of thermodynamic parameters assists to understand the temperature effect on the adsorption operation. The standard values of Gibbs free energy ($\Delta G^0$, kJ mol$^{-1}$), enthalpy ($\Delta H^0$, kJ mol$^{-1}$) and entropy ($\Delta S^0$, kJ mol$^{-1}$ K$^{-1}$) changes were
estimated from the parameters obtained in best isotherm model according to the equations 10–12. (Liu, 2009):

\[ \Delta G^0 = -R T \ln(K_c) \]  
\[ \Delta G^0 = \Delta H^0 - T \Delta S^0 \]  
\[ \ln(K_c) = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{RT} \]

where, \( K_c \) is the equilibrium constant (L g\(^{-1}\)), \( T \) is the temperature (K), \( R \) is 8.31×10\(^{-3}\) (kJ mol\(^{-1}\) K\(^{-1}\)) and \( \rho \) is the solution density (g L\(^{-1}\)).

3. RESULTS AND DISCUSSION

3.1. Characteristics of Chitin/ZSM-5 zeolite

Figure 2 presents the FT–IR spectra of Chitin/ZSM–5 zeolite.

The absorbance bands at 780 cm\(^{-1}\) and 465 cm\(^{-1}\) are assigned to the symmetrical stretching and T–O (T = Si, Al) bending, respectively, indicating the formation of T–O tetrahedrons (Luo et al., 2017). The absorbance band at around 1090 cm\(^{-1}\) is assigned to the T–O internal asymmetrical stretching vibration. A shoulder band around 1220 cm\(^{-1}\) can be attributed to the external T–O asymmetrical stretching vibration. The absorbance bands at 3480 cm\(^{-1}\) and 1630 cm\(^{-1}\) are belonged to the stretching and bending vibrations of silanol groups (Si – OH). In Figure 2 it was not found the main intense bands of the functional groups of chitin, like: O–H and N–H stretching (3300 cm\(^{-1}\)), CH\(_2\) and CH\(_3\) stretching (3000 and 2800 cm\(^{-1}\)), C=O secondary amide stretch (1650 cm\(^{-1}\)), N–H bend and C–N stretch of amide II (1550 cm\(^{-1}\)), C–O asymmetric stretch in phase ring (1020 cm\(^{-1}\)) (Dotto et al., 2015). Since chitin bands were not found it can be stated that it was completely eliminate in the calcination step.

In Figure 3 are illustrated the X–ray patterns (XRD) of the chitin/ZSM–5 sample. It can be observed that the sample shows characteristic peaks of MFI (Mobil Five) type structure that represents the orthorhombic end member of the pentasil family of zeolites (Treacy and Higgins, 2001), confirming the formation of the ZSM–5 zeolite. Nevertheless, possibly, the addition of a template can lead to a crystallinity decrease (Li et al., 2016).

Figure 3. X–ray patterns of chitin/ZSM–5.

N\(_2\) adsorption–desorption isotherm of chitin/ZSM–5 zeolite is shown in Figure 4.

Figure 4. N\(_2\) adsorption–desorption isotherm for chitin/ZSM–5 zeolite.

The isotherm presents adsorption volume at relatively low pressures (P/P\(_0\) < 0.1), indicating that
the microporosity of sample chitin/ZSM–5 is small. The chitin/ZSM–5 presented a loop that starts at P/P₀=0.6 and has greatly increased at partial pressures P/P₀>0.8, indicating the presence of mesopores (Yang et al., 2004).

The textural properties of chitin/ZSM–5 zeolite are summarized in Table 1, with information about surface, volume and pore diameter. The mesopore volume was 2.6 times larger than the micropore volume. The mean pore diameter, determined by the BJH method, was 22.49 nm, which in the mesopore range.

Table 1. Textural properties of chitin/ZSM–5.

<table>
<thead>
<tr>
<th>Surface (m² g⁻¹)</th>
<th>Volume(cm³ g⁻¹)</th>
<th>Pore Diameter</th>
</tr>
</thead>
<tbody>
<tr>
<td>𝑆_{BET} 164</td>
<td>𝑆_{Micro} 118</td>
<td>𝑆_{Ext} 46</td>
</tr>
</tbody>
</table>

* BJH Adsorption average pore diameter (4V/A)

Figure 5 shows the pore size distribution curve of the chitin/ZSM–5 sample obtained by BJH method. It can be verified that the pores of chitin/ZSM–5 were predominantly in the region from 15 to 80 nm, the mesopore range.

![Figure 5](image)

Figure 5. Pore size distribution for ZSM–5 zeolite and chitin/ZSM–5 zeolite.

The SEM images of chitin/ZSM–5 zeolite, with magnifications from 1,000 to 100,000x are presented in Figure 6. Chitin/ZSM–5 was in the form of large agglomerates composed by fine particles (Figure 6 (a)). In Figure 6 (b), the crystals exhibited a hexagonal sheet structure with smooth surface. The chitin/ZSM–5 presented particle size lower than 0.5 µm (Figure 6 (b)). At 100,000 times of magnification (Figure 6 (c)), a mesopores network can be visualized.

![Figure 6](image)

Figure 6. SEM images of chitin/ZSM–5 zeolite: (a) ×1,000, (b)×10,000 and (c)×100,000.
Based on the FT–IR, XRD, BET, BJH and SEM techniques, it can be concluded that the insertion of chitin as template in the synthesis route of ZSM–5 provided the following characteristics for the material, regarding the dye adsorption: i) the functional groups are the same of the pattern ZSM–5; ii) the solid structure is characterized like MFI; iii) the mesopore pore volume was 2.6 times larger than the micropore volume; iv) the mean pore diameter was in the mesopore range. All these characteristics become the mesoporous ZSM–5 a potential adsorbent to removal dye molecules.

3.2 Adsorbent dosage effect on dye adsorption

The adsorbent dosage study provides information on the feasibility of the operation. In the Figure 7, the adsorbent dosage effect on the removal percentage for BF dye was presented.

![Figure 7. Adsorbent dosage to dye removal.](image)

In Figure 7 is possible to note that dye removal percentage was greatest at 2.0 mg L⁻¹. This occurs because more adsorbent increases the total number of adsorption sites. The maximal dye removal was 81.2%.

3.3 pH effect on dye adsorption

pH affects the surface charge of the adsorbent and also in the ionization degree of the adsorbate. So, Figure 8 indicates that the pH increase from 2.0 to 10.0 caused an increase in the dye removal percentage.

![Figure 8. pH effect on dye removal percentage.](image)

From the Figure 8 is possible to find optimum pH. According Lan et al. (2013) pH above 9.0, the structure of BF changes and results in fading and red sediment. Then, the best pH was 9.0. The %R was higher in alkaline conditions, possibly because cationic dyes are positively charged and there is a little competition with the H⁺ ions in solution.

3.5 Kinetic studies

The time required for equilibrium attainment of BF dye adsorption onto chitin/ZSM-5 zeolite was investigated in the range of 0 – 2h. (Figure 9).

![Figure 9. Kinetic curves of BF adsorption.](image)

In Figure 9 was observed an initial fast step and subsequent adsorption rate decrease, being the equilibrium attained at around 20 min, but in order to ensure the system equilibrium, the contact time for the equilibrium test was 120 min. The kinetic profiles revealed that the adsorption sites were
progressively occupied by dye molecules. The maximum \( q_t \) was 23.5 mg g\(^{-1}\).

In the Table 2 is possible to observe the intraparticle diffusivity for each system.

**Table 2.** Kinetic parameters for FB adsorption on chitin/ZSM-5 zeolite.

<table>
<thead>
<tr>
<th>Dye</th>
<th>( D_p ) (m(^2) min(^{-1}))</th>
<th>( R^2 )</th>
<th>ARE</th>
</tr>
</thead>
<tbody>
<tr>
<td>BF</td>
<td>1.37x10(^{-13})</td>
<td>0.9996</td>
<td>0.55</td>
</tr>
</tbody>
</table>

It can be perceived, in Table 2, that \( D_p \) is in the range of intraparticle diffusion (Feliczak-Guzik, 2018) that is typical of the mesoporous materials. The \( R^2 \) values are near to 1.00 that means the dates are adjusted satisfactory. The fit was great also because of the ARE was near zero.

### 3.6 Equilibrium results

Equilibrium isotherms (Figure 10) were obtained at different temperatures (298, 308, 318 and 328 K) with concentration range from 0 to 300 mg L\(^{-1}\) and maintained the best parameters above studied.

![Figure 10](image_url) **Figure 10.** Isotherm curves for the BF adsorption on chitin/ZSM-5 zeolite.

Figure 10 shows that the isotherm curves were favored by the temperature increase. The quantity of dye adsorbed increase with the intensification of \( C_e \) (favorable adsorption). The slope of curve indicates that the saturation occurs at very higher concentrations and that the chitin/ZSM–5 contains several binding sites for BF adsorption.

The equilibrium curves were fitted with the Langmuir, Freundlich and Sips models, being the results presented in Table 3.

**Table 3.** Equilibrium parameters for the BF adsorption on chitin/ZSM–5 zeolite.

<table>
<thead>
<tr>
<th>Isotherm Model</th>
<th>Temperature(K)</th>
<th>( q_m ) (mg g(^{-1}))</th>
<th>( k_L ) (L mg(^{-1}))</th>
<th>( R^2 )</th>
<th>ARE (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>298</td>
<td>308</td>
<td>318</td>
<td>328</td>
<td></td>
</tr>
<tr>
<td>Freundlich</td>
<td></td>
<td>2.08</td>
<td>6.17</td>
<td>5.52</td>
<td>7.30</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.14</td>
<td>0.91</td>
<td>1.02</td>
<td>1.04</td>
</tr>
<tr>
<td>Langmuir</td>
<td></td>
<td>8291.9</td>
<td>789.1</td>
<td>3881.9</td>
<td>6863.6</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.0042</td>
<td>0.0071</td>
<td>0.0016</td>
<td>0.0012</td>
</tr>
<tr>
<td>Sips</td>
<td></td>
<td>215.66</td>
<td>216.40</td>
<td>237.18</td>
<td>234.55</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.035</td>
<td>0.050</td>
<td>0.056</td>
<td>0.079</td>
</tr>
</tbody>
</table>

*(mg g\(^{-1}\)) (mg L\(^{-1}\) \(^{-1}\) n\(^{-1}\))

In order to attest the efficiency of chitin/ZSM–5 zeolite for BF adsorption, a comparison amid maximum adsorbent capacities \( (q) \) of several adsorbents was performed (Table 4).

**Table 4.** Comparison of chitin/ZSM–5 zeolite with other adsorbents for BF dye adsorption.

<table>
<thead>
<tr>
<th>Adsorbent</th>
<th>( q ) (mg g(^{-1}))</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chitin/ZSM–5 zeolite</td>
<td>234.4</td>
<td>This work</td>
</tr>
<tr>
<td>Mesoporous Al-MCM-41 from</td>
<td>54.4</td>
<td>Guan et al. (2016)</td>
</tr>
<tr>
<td>natural palygorskite</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Graphene/β-cyclodextrin</td>
<td>425.8</td>
<td>Tan and Hu (2017)</td>
</tr>
<tr>
<td>composite</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**3.7 Thermodynamics results**

From the thermodynamic viewpoint, the adsorption was evaluated by the standard values of Gibbs free energy \( (\Delta G^0) \), enthalpy \( (\Delta H^0) \) and
entropy ($\Delta S^0$) changes. The thermodynamic parameters are shown in Table 5.

**Table 5.** Thermodynamics parameters.

<table>
<thead>
<tr>
<th>$\Delta G^0$ (kJ mol$^{-1}$)</th>
<th>$\Delta H^0$ (kJ mol$^{-1}$)</th>
<th>$\Delta S^0$ (kJ mol$^{-1}$ K$^{-1}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>-22.13</td>
<td>2.29 x 10$^{-01}$</td>
<td>1.51 x 10$^{-01}$</td>
</tr>
<tr>
<td>-23.76</td>
<td></td>
<td></td>
</tr>
<tr>
<td>-25.07</td>
<td></td>
<td></td>
</tr>
<tr>
<td>-26.73</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The negative $\Delta G^0$ values presented in Table 5 indicated that the adsorption was a spontaneous and favorable operation. The temperature increase led to a more negative $\Delta G^0$ values, indicating that the adsorption was most spontaneous and favorable at 328 K. Based on the positive $\Delta H^0$ values and magnitude, it was verified that the BF dye adsorption onto the zeolites was an endothermic operation and there was a physisorption.

4. CONCLUSION

In the present study, a mesoporous ZSM-5 zeolite was synthesized using chitin as template, in order to form a zeolite more attractive for the adsorption of large dye molecules. The synthesized zeolite was employed for the adsorption of Basic Fuchsin dye. It was found that chitin/ZSM-5 zeolite presented adequate characteristics for adsorption purposes, as follows: the volume of mesopores 2.6 times higher than the micropores volume and the mean pore diameter of 22.49 nm.

The results of adsorption studies showed that the operation was favored using adsorbent dosage of 2.0 g L$^{-1}$ and pH 9.0. The HSDM kinetic model was tested and the diffusion coefficients obtained were $1.37 \times 10^{-13}$ m$^2$ min$^{-1}$ to BF adsorption. The equilibrium was in accordance with the Sips model. The adsorption was spontaneous, favorable and endothermic. The maximum adsorption capacity was 234.45 mg g$^{-1}$.

5. REFERENCES
