INFLUENCE OF INITIAL WATER pH AND CONCENTRATION OF REGENERATING AGENT ON THE DYE DESORPTION EFFICIENCY OF BONE CHAR

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ABSTRACT: The aim of this was to evaluate the influence of the initial water pH and the concentration of regenerating agent in desorption of Reactive Blue BF-5G dye previously adsorbed in bone char. The inorganic solvents water and sodium hydroxide, and the organic solvents methyl, ethyl, and isopropyl alcohols were used as regenerating agents in batch systems. Water pH had no significant influence on desorption efficiency, and sodium hydroxide gave negligible results as well. The best desorption efficiencies, 20.88% and 19.49%, were obtained using isopropyl and ethyl alcohols, respectively. These results indicate the strong interactions between the dye and bone char surface are not able to be broken by the regenerating agents we studied, preventing from high dye desorption efficiency. Thus, desorption efficiencies may be related only to the regeneration of weaker interactions on bone char surfaces for these systems in batch conditions.

KEYWORDS: Desorption efficiency, bone char, reactive dye, regenerating agent, batch system.

1. INTRODUÇÃO

Generally, cationic, acid, reactive, direct, disperse, and sulfur dyes are used in the textile industry (Mahmoud *et al*., 2012). Among these, the use of reactive dyes has grown rapidly due to its increasing application in cellulosic fibers. However, reactive dyes have low rates of fixation to the fiber, which leads to a high concentration in textile effluents.

Among the methods used to remove dyes from textile effluents, the adsorption has proven to be an excellent process, offering significant economical and environmental advantages as compared to conventional methods, and include low cost, availability, profitability, efficiency, and ease of operation (Demirbas, 2009; Gautam *et al.*, 2013). Of the various existing adsorbents, activated carbon is the most common for color removal from textile effluent (Ahmed and Dhedan, 2012; Ip *et al.*, 2010; Belaid *et al.*, 2013). On the other hand, activated carbon has a finite capacity for adsorption. Thus, the regeneration of saturated carbons becomes one of the most important issues, since the reuse of the activated carbons could determine the economic viability of the adsorption process (Ofomaja and Ho, 2007).

The method of chemical regeneration offers a number of significant advantages: (1) it may be performed *in situ*, thus eliminating unloading, transporting and reconditioning of the adsorbent; (2) the loss of carbon resulting from thermal regeneration is eliminated; (3) the recovery of the adsorbate is possible; and (4) with the appropriate treatments, chemical regenerant can be reused (Cooney *et al.*, 1983).

The regenerating agents that can be used in chemical regeneration can be classified into two groups: inorganic chemicals and organic solvents. Among the inorganic regenerating agents, sodium hydroxide is one of the most effective agents in regenerating granular activated carbon (Leng and Pinto, 1996). On the other hand the organic solvents with different polarities can compete with the dye for the adsorption sites of activated carbons, leading to a dye desorption (Mahmoodi *et al.*, 2011).

Therefore, the aim of this work was to evaluate the influence of the initial water pH and concentration of regenerating agents on the desorption efficiency of Reactive Blue BF-5G dye in bone char.

2. MATERIALS AND METHODS

2.1. Materials

The bone char utilized in this work was generously donated by the company Bonechar Carbon do Brazil LTDA. The average particle diameter of the sample was of 0.725 mm. The main characteristics of this bone char sample were a surface area of 103 m^2/g, average pore diameter of 98 Å, and total pore volume of 0.2836 cm^3/g. These values are in accordance with literature (Ip *et al.*, 2010; Leyva-ramos *et al.*, 2010). The pH of zero charge (pH_{PZC}) is 7.5, as measured by the method described previously (Regabulto and Robles, 2004).

The Reactive Blue BF-5G dye (C.I. Reactive Blue 203; MW=1052 g/mol) was provided by Texpal Chemical Industry S/A.

The chemicals used as regenerating agents were deionized water, sodium hydroxide, methyl alcohol, ethyl alcohol, and isopropyl alcohol and were all of analytical grade. These reactants were used as obtained.

2.2. Preparation Of Saturated Activated Carbon

Before starting the desorption tests, the saturation of the bone char was performed by adding 1.5 g of adsorbent into Erlenmeyer flasks containing 150 mL of 4000 mg/L dye solution. All the flasks were shaken in a Dubnoff bath with controlled temperature (Nova Ética, model 304 DE) at 80 rpm and 30 ºC for 36 hours to reach adsorption equilibrium. After that, the saturated bone char was separated from solution by filtration and dried at 40 ºC until at constant weight. The dye solution absorbance was measured by UV/vis spectrophotometer (Shimadzu, model UV-1203) at predetermined wavelengths (600 nm for dye diluted in NaOH solution, 620 nm for dye diluted in water, and 625 nm for dye diluted in organic solvents). Then, the concentrations of the dye solutions were calculated by the Lambert-Beer law and the amount of dye removed from the solution was calculated using the relation:

$$q_{ea} = [(C_0 - C_{ea}) \times V] / M \qquad (1)$$

where q_{ea} is the amount of dye removed (mg/g) at equilibrium, C_0 is the initial concentration (mg/L) of dye in liquid phase, C_{ea} is the concentration of dye (mg/L) in the liquid phase at equilibrium, V is the volume of dye solution (L), and M is the mass of bone char sample (g).

The amount of dye removed at equilibrium by bone char was 105.5 mg/g.

2.3. Kinetics of desorption

To evaluate the kinetics of desorption deionized water (without pH adjustment), sodium hydroxide (0.1 mol/L), methyl alcohol 50% (v/v) ethyl alcohol 50% (v/v), and isopropyl alcohol 50% (v/v) were used as regenerating agents. These solutions were chosen due to the significant results reported in previous papers (Gupta *et al.*, 2009; Ip *et al.*, 2009; Lu *et al.*, 2011; Mahmoodi *et al.*, 2011; Mittal *et al.*, 2010; Robinson *et al.*, 2002).

The desorption kinetics experiments were performed by adding 0.2 g of saturated bone char sample to Erlenmeyer flasks and then adding 20 mL of the regenerating solution. All of the flasks were shaken in a Dubnoff bath with controlled temperature (Nova Ética, model 304 TPA) at 80 rpm and 30 ºC for 12 hours. Each flask was picked up from the shaker at predetermined intervals of time and its content was immediately filtered. The dye concentration in each filtered solution was determined as described above. Then the amount of dye desorbed as a function of time (t) was determined by:

$$q_{td} = (C_{td} \times V_r) / M_s \qquad (2)$$

where q_{td} is the amount of dye (mg/g) desorbed at time t, C_{td} is the desorbed dye concentration (mg/L) in the fluid phase at time t, V_r is the volume (L) of regenerating agent solution, and M_s is the mass (g) of bone char saturated sample.

Therefore, the desorption efficiency as a function of time t was determined by:

$$E_{td}\,(\%) = (q_{td} / q_{ea}) \times 100 \qquad (3)$$

where E_{td} is the desorption efficiency (%) at a time t, q_{td} is the amount of dye desorbed (mg/g) at a time t, and q_{ea} is the amount of adsorbed dye (mg/g) at equilibrium.

2.4. Influence Of The Initial Water pH

In order to analyze the effect of the pH on the dye desorption efficiency, the initial water pH was adjusted from 2 to 12 using 0.1 mol/L solutions of HCl or NaOH. Thus, 20 mL of deionized water with different initial values of pH were added to Erlenmeyer flasks containing 0.2 g of saturated bone char sample. All flasks were shaken in the Dubnoff bath using the same experimental conditions described in the previous section. The concentration of dye in each filtered solution was determined as described above. The amount of desorbed dye was determined according to:

$$q_{ed} = (C_{ed} \times V_r) / M_s \qquad (4)$$

where q_{ed} is the amount of desorbed dye (mg/g) at equilibrium, C_{ed} is the desorbed dye concentration (mg/L) in the fluid phase at equilibrium, V_r is the solution volume (L), and M_s is the mass (g) of bone char saturated sample.

The desorption efficiency was calculated by:

$$E_{eq}\,(\%) = (q_{ed} / q_{ea}) \times 100 \qquad (5)$$

where E_{eq} is the desorption efficiency (%) at equilibrium, q_{ed} is the amount of desorbed dye (mg/g) at equilibrium, and q_{ea} is the amount of adsorbed dye (mg/g) at equilibrium.

2.5. Influence Of Regenerating Agent Concentration

In order to verify the best concentration of each the inorganic or organic regenerating agents, 20 mL of each solution with different concentrations of each agent were added to Erlenmeyer flasks containing 0.2 g of saturated bone char sample. All of the flasks were shaken in the Dubnoff bath using the same experimental conditions described in the previous section. The concentration of dye in each filtered solution was determined as described above and the desorption efficiency was calculated by:

$$E_{ed}\,(\%) = (q_{ed}/q_{ea}) \times 100 \qquad (6)$$

where E_{ed} is the desorption efficiency (%) at equilibrium, q_{ed} is the amount of desorbed dye (mg/g) at equilibrium, and q_{ea} is the amount of adsorbed dye (mg/g) at equilibrium.

2.6. Fourier Transform Infrared Spectroscopy

Fourier transform infrared spectroscopy (FTIR) provided information on the chemical structure of the adsorbent material. The parent bone char sample was characterized by FTIR as received. Samples saturated with dye and desorbed with different regenerating agents were also analyzed.

In all cases the bone char samples were dried at 100 ºC for 12 hours, then crushed and mixed with KBr (5% bone char; 95% KBr).

3. RESULTS AND DISCUSSION

3.1. Equilibrium Time For Desorption

At first, time required for the equilibrium between saturated bone char and the regenerating agent solution should be obtained. The results obtained are shown in Figure 1.

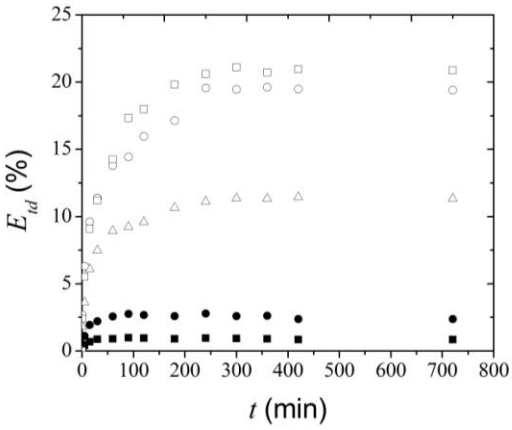

Figure 1 – Desorption kinetics of Reactive Blue BF-5G dye in bone char at 30 °C: (■) deionized water; (●) sodium hydroxide (0.1 mol/L); (Δ) methyl alcohol 50% (v/v); (○) ethyl alcohol 50% (v/v); and (□) isopropyl alcohol 50% (v/v).

As shown in Figure 1, the time to accomplish the desorption equilibrium was short: approximately 2 hours for the water and the sodium hydroxide solutions, and close to 4 hours for the other regenerating agents solutions.

These low desorption equilibrium times, especially for the water and sodium hydroxide solutions, may be important for processes to be applicable on an industrial scale, since it could lead to a reduction in the adsorbent regeneration stage time, as well as a decrease in operating costs.

We also observed that the ethyl alcohol led to a 19.49% increase in the desorption efficiency and isopropyl lead to a 20.88% increase, which corresponded to 20.55 mg/g and 22.02 mg/g, respectively. Methyl alcohol showed intermediate behavior, with a desorption efficiency of 11.34% (11.96 mg/g), whereas water and sodium hydroxide led to very low desorption efficiencies of 0.89% and 2.59%, which correspond to 0.99 mg/g and 2.73 mg/g, respectively. Thus, it may be concluded that organic regenerating agents are far more efficient in dye desorption than inorganic agents.

3.2. Influence Of Initial Water pH

Figure 2 shows the influence of initial water pH on the desorption efficiency of the bone char sample. The initial water pH could influence the amount of dye desorbed from the bone char surface. In fact, according to Mahmoodi *et al*. (2011), as the pH of the system increases, the number of sites with negative charge increases, favoring the anionic dye desorption resulting from electrostatic repulsion.

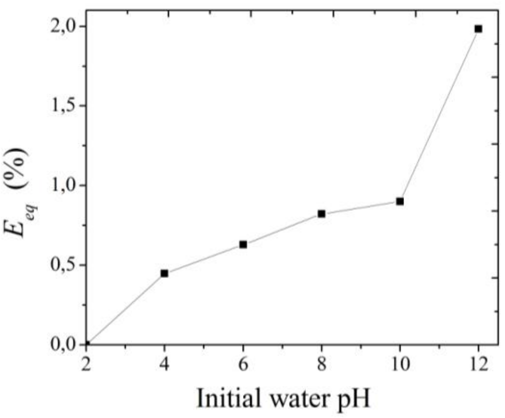

Figure 2 – Influence of initial water pH on the Reactive Blue BF-5G desorption efficiency in bone char at 30 ºC.

The curve presented in Figure 2 shows that the dye desorption efficiency increases with the initial water pH. Thereby, while there was no dye desorption in water at pH 2, the desorption efficiency increased continuously until pH 12, reaching a value of 1.98%, which corresponds to 2.09 mg/g. The desorption mechanism is schematically represented in Figure 3.

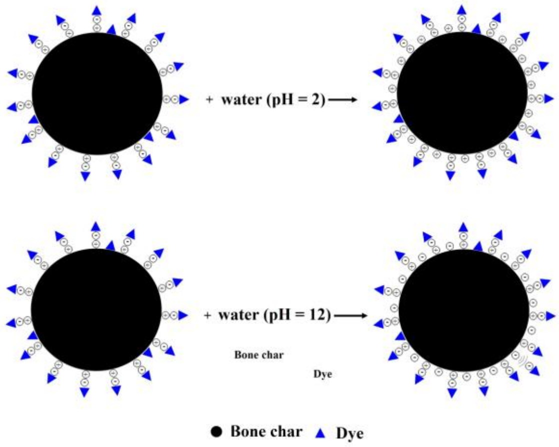

Figure 3: Squematic representation of the influence of water pH in the desorption mechanism of the Reactive Blue BF-5G molecule (in blue) adsorbed in the bone char sample (black)

In strongly acidic solutions, significantly lower than pH_{PZC} 7.5, the anionic dye adsorption is favored by the increasing eletrostatic interaction between positive charges generated on the bone char surface (Salleh *et al.*, 2011) and the anionic group of the dye, which does not favor dye desorption. On the other hand, when the pH is higher than pH_{PZC} 7.5, the alkalinity favors the appearance of negative charges on the bone char surface, increasing the anionic dye repulsion and, consequently, favoring desorption. Although the desorption efficiency increased with increasing pH, the increase was very small, showing that changing the initial water pH has a very small effect on the desorption efficiency. This indicates that changing from an acid to basic medium is not sufficient to break the interactions formed between the dye and bone char. These results emphasize the importance of investigating more efficient regenerating agents.

3.3. Influence Of Regenerating Agent Initial Concentration

According to Lu *et al*. (2011), the initial concentration of the regenerating agent may influence the desorption efficiency. Therefore, we performed experiments with different initial concentrations of inorganic and organic regenerating agents in order to verify their influence on the dye desorption. Results obtained with sodium hydroxide as an inorganic regenerating agent are shown in Figure 4.

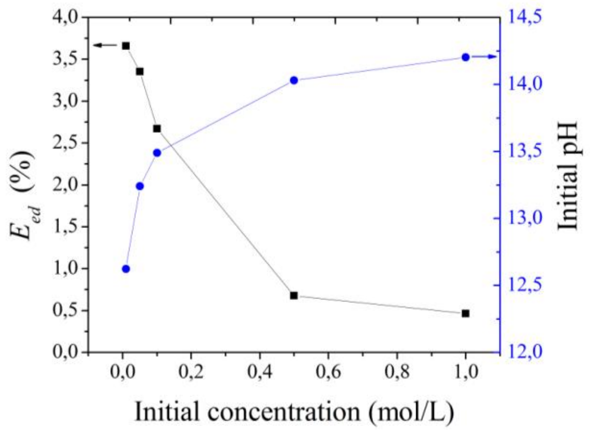

Figure 4 – Desorption efficiency of Reactive Blue BF-5G dye as a function of the initial concentration of the sodium hydroxide solution at 30 ºC.

As observed in Figure 1, the dye desorption efficiency using a 0.1 mol/L solution of NaOH was very low (2%). As the sodium hydroxide concentration increases the medium pH also increases. Thus, it was expected that solutions with higher basicity would lead to an effective desorption of the dye on activated carbon. As the solid surface becomes negatively charged, there would be an increase in the electrostatic dye repulsion, as discussed earlier.

According to Mall *et al*. (2006), strong acids, such as HCl or HNO_3, or strong bases, such as NaOH, can promote dye desorption. Indeed, Namasivayam *et al*. (1998) performed the adsorption of the Direct Red and Acid Brilliant Blue dyes, both of which are anionic in nature. It was observed that adsorption increased in acidic solutions and desorption in alkaline solutions. According to the authors, this shows that these dyes are most likely retained by a strong interaction of ion exchange. Similar results were found by Sivaraj *et al*. (1984). However, the dissociation of the molecules present in strong acids and bases, such as NaOH and HCl, occurs in aqueous solution, and the ions Na^+, OH^-, H^+, and Cl^- may compete for positively or negatively charged adsorption sites on the bone char surface, affecting the nature of the activated carbon surface (Martin and NG, 1984). Leng and Pinto (1996) studied the chemical regeneration of activated carbon saturated with different adsorbates. It was observed that for the activated carbon saturated with phenol, aniline, and benzoic acid, the increase on the initial concentration of NaOH resulted in a decrease in desorption efficiency. This NaOH dependence can be attributed to the higher adsorption of OH^- at higher concentrations of NaOH, which inhibits the desorption process.

As shown in Figure 4, the desorption efficiency of the dye decreases with increasing sodium hydroxide concentration. At higher concentrations of sodium hydroxide, the initial pH is higher (for 1 mol/L NaOH solution, pH=14.2). This increase in pH generates negative charges on the bone char surface, which would increase the repulsion between the dye and adsorbent agent. Thus, it is believed that the Na^+ ions present in the solution would neutralize these negative surface charges. This reduces the electrostatic repulsion between the dye and the bone char. As a consequence, dye molecules remain adsorbed onto the bone char surface. Therefore, sodium hydroxide was not effective in promoting the dye desorption, since the higher desorption efficiency observed was only 3.66%, obtained using a very low initial concentration of 0.01 mol/L. Figure 5 presents a schematic representation of the desorption mechanism at low NaOH concentration (say 0.01 mol/L) and high NaOH concentration (say 1 mol/L).

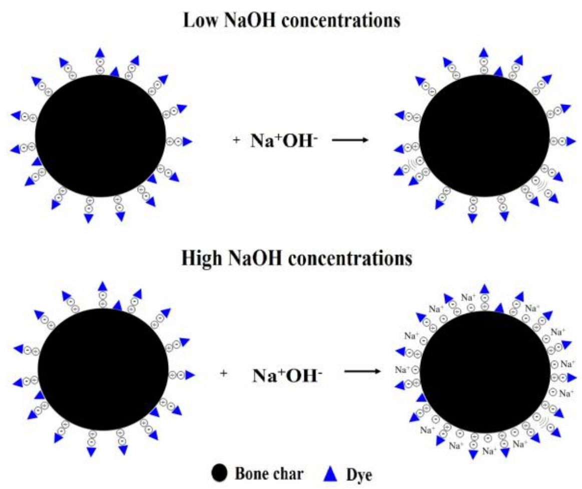

Figure 5 – Schematic representation of desorption mechanism of Reactive Blue BF-5G in bone char with the regenerating agent NaOH

As already shown in Figure 1, organic regenerating agents have a higher ability to promote dye desorption from the bone char surface. A more accurate analysis is shown in Figure 6, which depicts the dye desorption efficiency as a function of the initial concentration of the organic regenerating agents.

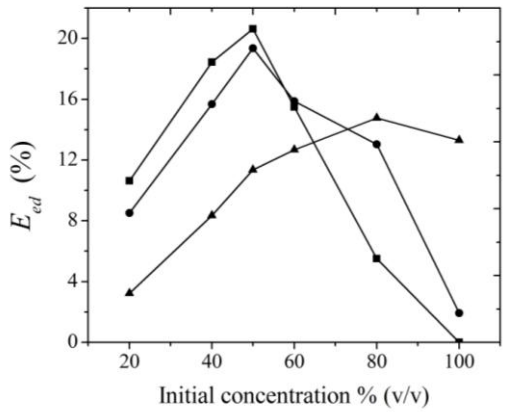

Figure 6 – Influence of initial concentration of organic solvents on dye desorption efficiency at 30 ºC: (▲) methyl alcohol; (●) ethyl alcohol; and (■) isopropyl alcohol.

It was observed that the most efficient desorption process was achieved by alcohol-water solutions. The optimal concentrations for each regenerating agent were 50% for isopropyl alcohol, 50% for ethyl alcohol, and 80% for methyl alcohol, with dye desorption efficiencies of 20.63%, 19.35%, and 14.75%, respectively. These results are in agreement with previously published results. Lu *et al*. (2011) studied the desorption of anionic dyes in coconut shell activated carbon using isopropyl alcohol and acetone as regenerating agents. The best desorption capacities were obtained with 40% (v/v) isopropyl alcohol for Mustard Yellow dye and 60% (v/v) acetone for Peach Red dye. Thus, the results indicate that the solvents have higher efficiency in alcoholic solutions and lower efficiency when highly concentrated alcohols are used.

One of the characteristics of organic regenerating agents that may influence desorption efficiency is their hydrophobicity (Lu *et al*., 2011). The partition coefficients of methanol, ethanol, and isopropanol are 0.15, 0.48, and 2.19, respectively. A regenerating agent with higher partition coefficient is more hydrophobic. Therefore, it is expected that a competition occurs between the alcohol and the dye molecule for adsorption sites on the bone char surface. As the isopropyl and ethyl alcohols are more hydrophobic than methyl alcohol, it is believed that these two compounds cause a higher competition between the dye molecules and the regenerating agent for the adsorption sites, leading to a higher desorption efficiency.

According to results shown in Figure 6, we concluded that the highest desorption efficiencies were obtained using ethanol (19.49%) and isopropanol (20.88%). However, it is necessary to emphasize that the values of desorption efficiency reached were still low. Figure 7 presents a schematic representation of the desorption mechanism using organic regenerating agents.

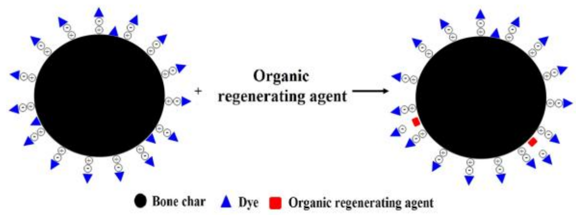

Figure 7 – Schematic representation of Reactive Blue BF-5G desorption in bone char with the organic regenerating agents.

According to Çelekli *et al*. (2011), the reversibility of the adsorption process depends on the existence of strong interactions (e.g. ionic or covalent) or weak interactions (e.g. Van der Waals forces or dipole-dipole interactions) that are formed between the dye molecules and the adsorbent surface. Therefore, the values of desorption efficiency obtained indicate that the irreversibility of adsorption was associated with the fact that they show strong interactions between the dye and the bone char surface, which is characteristic of chemisorption. Thus, the regenerating agents studied in this work were not able to break all these strong interactions. These solvents probably regenerated only the weak interactions on the bone char surface.

Samples saturated with dye and desorbed with the regenerating agents ethanol and isopropanol were analyzed by Fourier transform Infrared Spectroscopy (FTIR) and the spectra obtained are shown in Figure 8.

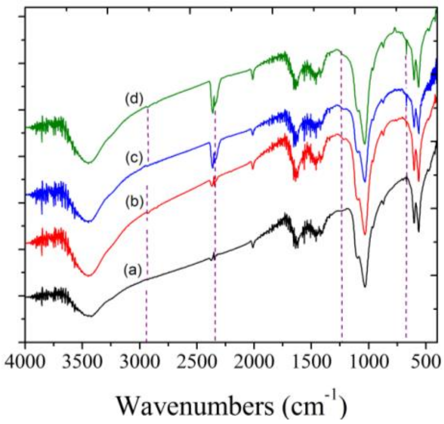

Figure 8 – Infrared spectroscopy of bone char (a) parent sample, (b) saturated with Reactive Blue BF-5G dye, (c) desorbed with ethyl alcohol, and (d) desorbed with isopropyl alcohol.

Some common peaks were observed in Figure 8, such as those at 3414 and 665 cm^{-1}, which can be assigned to the stretching vibrations of –OH. The peak at 2358 cm^{-1} is related to C≡C, and that at 1616 cm^{-1} is related to C=O, while the molecular vibration of CO_3^{2-} can be observed at 1446 and 873 cm^{-1}. There were also characteristic peaks for PO_4^{-3} at 1046, 608, and 550 cm^{-1} (Brum *et al.*, 2010; Pavia *et al.*, 2001; Silverstein *et al.*, 2005; Socrates, 2001;).

After adsorption of the dye molecules (spectrum b), three different peaks were found in comparison with the parent bone char sample (spectrum a). One at 2931 cm^{-1}, which is related to C–H, another at 1219 cm^{-1} corresponding to SO_3^-, and a final one at 726 cm^{-1}, which corresponds to –OH (Silverstein *et al.*, 2005). These bands may represent strong adsorption of the dye molecule that contains these groups and the surface of the bone char.

The most characteristic change was the increase of the peaks at 2358 cm^{-1} and, to a lesser extent, at 665 cm^{-1}. Changes in intensity may suggest that the characteristic groups (–OH and C≡C, respectively) had relevance in the desorption process. We also believed that such changes are related to the –OH group in both peaks. This is in agreement with the quantitative desorption results and supports the mechanism proposed in Figure 7, where desorption occurs through replacement of the dye by the alcohol molecules.

Finally, it is important to note that the bands at 2931 and 1219 cm^{-1} that surged after the saturation remained during the regeneration process. Therefore, it may be concluded that some irreversible changes occurred when the bone char was submitted to adsorption with Reactive Blue BF-5G.

4. CONCLUSION

The results obtained in this work allowed us to conclude that initial water pH has no significant influence on desorption process and sodium hydroxide give negligible results as well. The highest desorption efficiencies were obtained using isopropyl (20.88%) and ethyl (19.49%) alcohols, respectively. The results also show that the adsorption mechanism occurs predominantly through strong interactions, although weak interactions also occur. Only weak interactions between dye and bone char surface can be regenerated.

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