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RESEARCH ARTICLE

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ADSORPTION STUDY FOR REMOVAL OF REMAZOL BLACK B DYE IN AQUEOUS SOLUTION USING ACTIVATED CARBON

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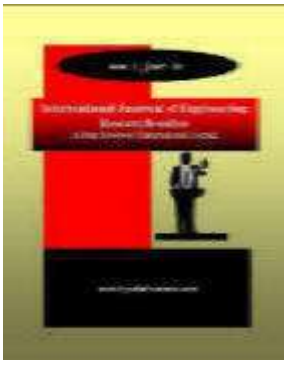

ABSTRACT
It is of great environmental importance that effluents from textile industries are treated before being discarded, since they contain dyes that are polluting agents when in contact with nature. A widely used process to remove these substances from the medium is adsorption, due to the simplicity of operation, as it is economically viable in industrial proportions, allowing the use of low cost materials and high solute removal capacity. This work aimed to evaluate the removal of the Remazol black B dye through this process using activated carbon as an adsorbent, in order to determine process equilibrium isotherms, the adsorption kinetic curves and to evaluate the dye removal capacity in a synthetic effluent. The experiments were carried out in a glass reactor in a mud bed in a batch process at a constant stirring speed of 300 rpm at 30°C where the carbon and the synthetic dye solution were placed. Samples were taken with a porous filter and read on a spectrophotometer at a wavelength of 595 nm. In the kinetics experiment, identified as 1st order, equilibrium was reached after 240 min with 84% removal, stabilizing at 330 min by removing 90% of the dye. The Langmuir isotherm model was the one that best adjusted to the RB adsorption behavior on the adsorbent surface and the maximum adsorptive capacity obtained was 5.656 mg/g for the dye on the Carbon.

INTRODUCTION

The advance in industrial activities in recent years has caused new problems due to the elimination of toxic waste, derived from by-products generated by the industry, and with this the growing concern around the contamination of natural waters. Within this context, the textile sector has a special emphasis due to the generation of large volumes of effluents, which, when not properly treated, can cause serious problems of environmental contamination [1]. Textile processing involves three stages of production: yarn formation; fabric formation and finishing. The stages of formation of the threads and formation of the fabric participate with a very small contribution in the generation of effluents, in opposition to the wet processes (finishes), which are generators of the greater part of these effluents. The effluents generated by the textile industries are quite complex, and may contain a wide variety of dyes, dispersants, acids, bases, salts,

detergents, humectants, oxidizers, etc [10]. Effluents with dyes are one of the most difficult effluents to be treated due to the high concentration of organic matter, suspended solids and toxic compounds, in addition to the easy identification of waste by its color [2] . The dyes released into the environment are toxic to various organisms and affect the ecosystem mainly by blocking sunlight, which causes a reduction in oxygen production through photosynthesis of algae and plants present in the water body. The dye molecules are made up of two main parts: the chromophore group, which gives the color to the compound, and auxiliary groups (auxochromes), which facilitate its affinity for the substrate and which are responsible for fixing the dye to the fiber, fabric, leather, hair, paper, among others, giving a color resistant to light and washing [3]. Dyes can be classified according to the method of attachment to the fiber as: acids, reactive, direct, azoic, dispersive, vat and pre-metallized, of which the only ones that do not apply to the textile industry are pre-metallized and basic. Different methods are found in the literature that are used to treat colored aqueous effluents [11-12]. There is no general method for discoloring aqueous effluents from the textile industry. Most industries perform treatment processes based on the precipitation / coagulation operation, followed by biological action, mainly with activated sludge. Treatment techniques based on a coagulation process followed by flotation or sedimentation are highly efficient in removing particulate matter, but the removal of color and dissolved organic compounds is deficient. According to Donnaperna et al (2009) [5] adsorption is one of the techniques that has been used successfully in the effective removal of dyes. This process finds great industrial application, as it associates low cost and high removal rates [4]. In addition, in some cases, it makes it possible to recover the dye without losing its chemical identity, as it is a non-destructive method. Adsorption is a surface phenomenon, which is related to the available area of the adsorbent in relation to the masses of adsorbate and adsorbent, pH, temperature, ionic strength and the chemical nature of the adsorbent and adsorbate, which can be reversible or irreversible. The molecules that are present in a fluid, liquid or gas, are spontaneously concentrated on a solid surface. Generally, adsorption appears to occur as a result of unbalanced forces on the surface of the solid that attract the molecules of a fluid in contact for a finite time. Donnaperna et al (2009) investigated the removal of the reactive dye Blue Drim KBL in ripped shale and observed that the increase in the concentration of the dye caused the amount of it retained in the adsorbent to tend to a maximum value. Oliveira studied the removal of the Remazol Black B dye by adsorption using sugarcane bagasse and activated carbon in a batch reactor as adsorbents. The balance data were adjusted to the Langmuir model and indicated irreversible adsorption of the dye. It was found that activated carbon had a greater capacity for adsorption than sugarcane bagasse [5]. The maximum amount of dye removed can vary dramatically and will depend on the chemical and physical characteristics of the adsorbents, as these properties are directly related to the adsorvate's chemisorption and physisorption processes. The characteristics of each dye also cannot be overlooked in the adsorption process. The size of these molecules allows for only partial diffusion, mainly in adsorbents with high microporosity.

The dyes can be visible in some cases in concentrations as low as one ppm and in small amounts they affect the transparency of the water and the solubility of the gases. These pigments have, in general, high biological stability, high molecular weight and the presence of aromatic rings. Due to the high toxicity, its treatment is necessary, the most used methods for removing effluent dyes are biological and physical-chemical processes such as precipitation, coagulation, adsorption, etc. (The dye used in this work was Remazol Black B 133% Gran (OP.PN10503, $C_{26}H_{21}N_5O_{19}S_6Na_4$, 991.82 g/mol, Figure 1), composed of the class of azo dyes (present one or more groups -N = N- linked to aromatic systems), widely used in the textile industry, with about 60% of the dyes, used in the area, of this type. This dye has 2 sulfonate groups and two other sulfatoethyl sulfone groups, with negative charges, even in highly acidic solutions, due to its pKa with values less than zero. The treatment method chosen for the removal of this dye was adsorption. According to Soto et al (2011) [6], adsorptive processes stand out for their simplicity of design and operation, insensitivity to toxic substances, ease of regeneration and low cost, in addition to avoiding the use of toxic solvents. The objective of this research was to evaluate the removal of the Remazol black B dye through the adsorption process using

activated carbon, in order to determine process equilibrium isotherms, the adsorption kinetic curves and to evaluate the dye removal capacity in a synthetic effluent. Thus, the present study aimed to evaluate the ability to remove Remazol Black B Dye from a synthetic aqueous solution, using Activated Carbon, as well as the influence of chemical treatments on the kinetics of the adsorptive process.

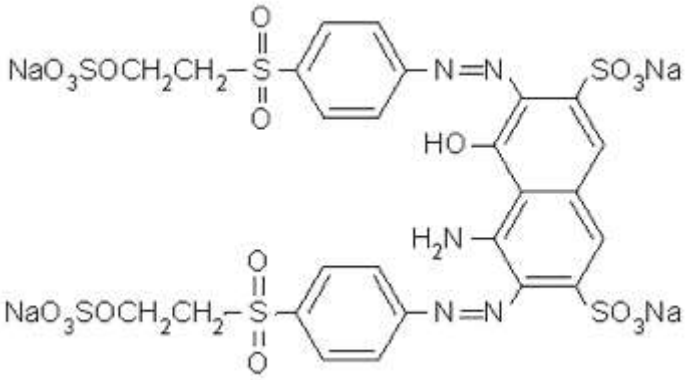

Figure 1. Structural formulation of the textile dye Remazol Black B (RB).

METHODOLOGY

In this work, the adsorption method was used as a dye separation technique, for which commercial activated carbon (CAS no7440-44-0, Activated Charcoal (Decolorizing Powder) LR Grade, SD fine Chemical Ltd) was used as an adsorbent. In order to determine its adsorptive capacity in the middle of the dye solution, the physical-chemical properties of this adsorbent were characterized from the following techniques: thermo gravimetric analysis (TGA), zero point charge (pH_{ZPC}) and textural characterization method (Brunauer, Emmett, Teller - BET).

EXPERIMENTAL PROCEDURE

The initial concentration of the synthetic solution was prepared from the commercial powder dye Remazol Black B (RB) dissolved in distilled water at a concentration of 400mg/L. The Remazol solutions used later in the study were obtained by diluting this initial solution. All the adsorption kinetics experiments were conducted in a glass bed reactor in a batch process, at a constant stirring speed of 300rpm, in a thermostatic bath at 32°C where the synthetic dye solution and the adsorbent were placed. Samples were removed with a porous filter, approximately 5mL each, in the first hour 14 samples were taken, in the second hour, 4 (every 15 minutes) and in the next hours every 30 minutes until the reaction stabilized. Where concentration readings were taken on a spectrophotometer [Double Beam UV-Visible Spectrophotometer SL 210 – Elico], at a wavelength of 595nm. The amount of dye mass adsorbed by carbon mass at equilibrium was calculated using Equation 1:

$$q = \frac{Ci-C}{m} \times V \quad \text{...Eq -1}$$

q being the amount of the adsorbate in mg of adsorbent/g of adsorbent, Ci the initial concentration (mg/L), C the concentration at the time of sample removal (mg/L), V the volume of the solution (L) in an adsorbent mass (g).

The adsorption model used to evaluate the process equilibrium data was Langmuir-Freundlich (Equation 2). For this study experiments were carried out using 2.8g of activated carbon in contact with 700ml of synthetic solution of the dye in concentrations of 4; 16; 28 and 40mg/L.

$$qe = \frac{q_{\max} K_{LF} C_e^{1/n}}{1+K_{LF} C_e^{1/n}}$$ .....Eq -2

Where qe is the amount of adsorbate adsorbed at equilibrium (mg/L), qmax the maximum adsorptive capacity, K_{LF} is the Langmuir-Freundlich constant, Ce is the concentration at equilibrium (mg/L) and n the order of the equation.

CHARACTERIZATION OF THE ADSORBENT

Thermo gravimetric analysis: The sample was subjected to thermogravimetric tests to obtain the intervals and percentages of mass losses through heat. The analysis was performed on Perkin Elmer thermo balance, model STA 6000, with a heating rate of 20°C/min, under a flow of 20 mL/min of N_2. The mass of material used in the platinum crucibles was fixed at 10 mg and they were heated from 30°C to 800° C.

pH Zero point Charge: It was determined by placing 0.1 g of the material in contact with distilled water, in a pH range between 2 and 11, which were adjusted with hydrochloric acid solutions (0.1 mol/L) and sodium hydroxide (0.1 mol/L) with the aid of a pH meter, under agitation of 300 rpm. The pH was measured again after the contact time, which was 24 h. For the determination of the pH_{ZPC} the graph (final pH – initial pH) vs. $pH_{initial}$, in which the curve that intersects the axis of pH's corresponds to the point where the material's surface charge is zero.

Textural Characterization: The specific surface area of the materials used in this work was determined through the adsorption of N_2 at 77 ± 5 K. To remove moisture from the sample surface, a pre-treatment at 333 K was performed under vacuum for 3 hours.

RESULTS AND DISCUSSION

Thermo gravimetric Analysis: The results of the thermo gravimetric analysis of activated carbon are shown in Figure 2, where it can be seen that the adsorbent does not suffer significant losses of mass at the temperature used during the process described in this work.

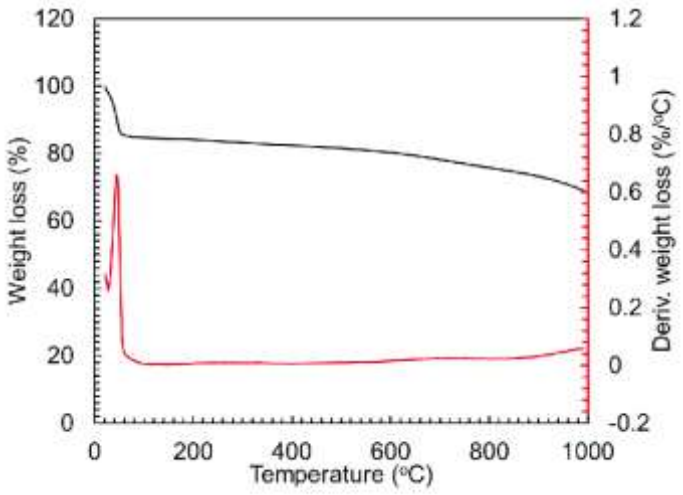

Figure 2. Thermogravimetric analysis of activated carbon.

Zero point Charge: It can be seen in Figure 3 that the intersection with the horizontal axis was at pH = 7.2, which corresponds to the pH at the Zero point Charge (pH_{ZPC}). This means that at pH above this value, the charges on the surface of the carbon are negative, favoring the adsorption of cations, while for pH values below 7.2 [7], the charges on the surface are positive, favoring the adsorption of anions, as this is the case of the dye under study.

In view of this pH value at the zero point charge of the carbon used, it was chosen to work at the natural pH of the solution, since it has a value less than 7.2, thus enabling the adsorption of the dye on the adsorbent.

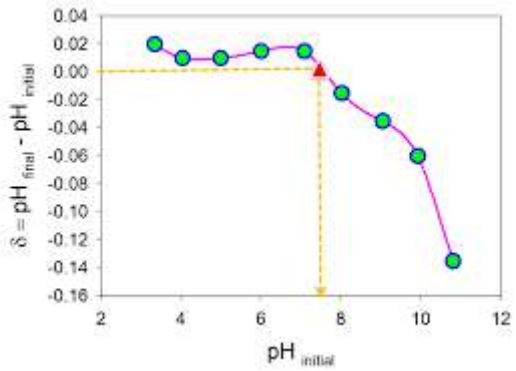

Figure 3.pH zero point charge of activated carbon

Textural Characterization: The result of BET through N_2 adsorption, for activated carbon are shown in Table 1:

Table 1. Results obtained by adsorption of N_2.

Material	Surface area (m^2/g)	Total pore volume (cm^3/g)	Pore diameter (Å)
Activated Carbon	829.4	0.476	11.25

According to IUPAC (1982), the carbons used are classified as mesoporous. It has a large surface area which may indicate that it has the potential to be a good adsorbent [8]. Reaction kinetics: The kinetics in the process of removing the dye in contact with the carbon was evaluated experimentally in tests between 0 and 60 minutes. The equilibration time for adsorption occurred after 240 min, where the removal rate is approximately 84%, stabilizing after 330 min, with a rate of 90% removal of the dye (Figure 4). A maximum time was not established for carrying out the equilibrium experiments, in order to guarantee that it had been reached, samples were taken every 30 min and read in the spectrophotometer and only after the reaction stabilized the experiment was ended.

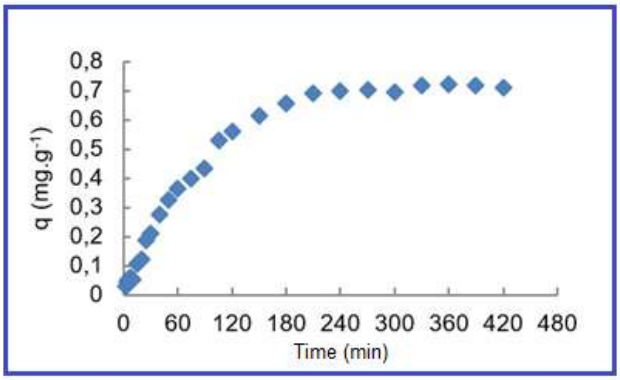

Figure 4. Kinetic behavior of the adsorption of Remazol Black B. T = 30° C; Stirring speed = 300 rpm, mass of adsorbent: 2.8g; Initial concentration of the synthetic dye solution = 4mg/L.

Adsorption equilibrium: The Langmuir-Freundlich model (Equation 2) was used to represent the adsorption behavior of RB on the surface of the activated carbon [9]. The adsorption isotherm is represented in Figure 5 and the linearization of the model is represented by Figure 6, where it is possible to estimate the maximum adsorption capacity qmax, and the equilibrium constant of Langmuir, Keq, represented in Table 2.

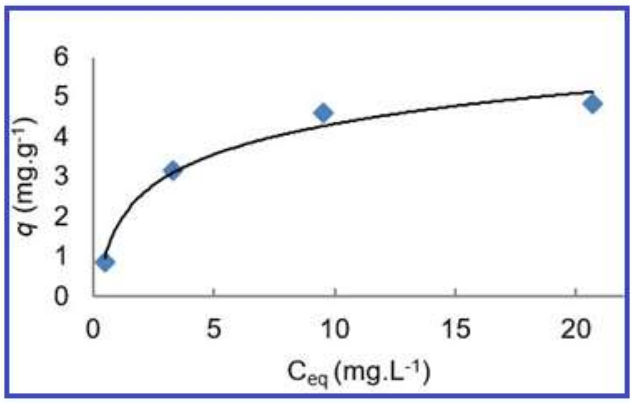

Figure 5. Freundlich Adsorption isotherm of Remazol Black B dye on activated carbon. T = 30° C; Rotation speed = 300 rpm; mass of the adsorbent = 2.8 g.

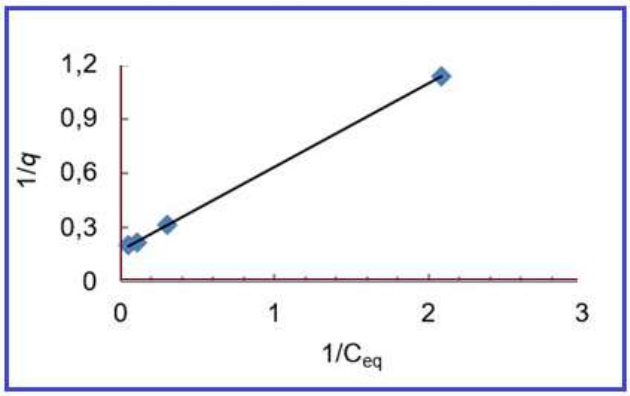

Figure 6. Linearization of the Langmuir model.

Observing the graphs, it is noted that the Langmuir model adjusted well to the experimental points, considering that a good correlation coefficient was obtained in the linearization (R^2 = 0.9999).

Table 2. Data used to develop the kinetic model of adsorption.

Parameters	Values
m	2.8 g
V	0.7 L
C_o	4 - 40 mg/L
q_{max}	5.656 mg/g
K_{LF}	0.3894 L/mg

Conclusions

The study of the removal of dyes in synthetic solutions demonstrated a maximum adsorption capacity evaluated by the Langmuir equation of 5.656mg/g for the dye Remazol Black B on activated carbon. The evaluation of the equilibrium isotherm leads to experimental results satisfactorily modelled by the Langmuir equation, with the adsorption equilibrium constant equal to 0.3894 L/mg, evaluated under these conditions. The adsorption kinetics was identified as being 1st order. Activated carbon showed good adsorptive capacity.

The positive surface charge observed in this adsorbent, through the study of the zero point charge, demonstrates the feasibility of using it without varying the pH of the medium, since the dye under study is an anionic substance in solution.

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