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The evaluation of rhodamine FRBT dye removal by adsorption using coffee grounds as an adsorbent material

Avaliação da remoção do corante rodamina FRBT por adsorção utilizando borra de café como material adsorvente

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Abstract

The application of activated carbon as an adsorbent material has been widely studied in several areas of the industry. Different carbonaceous materials with adsorbent potential, such as coffee grounds, have shown interesting adsorption properties regarding the removal of textile dyes from industrial effluents. High volumes of coffee grounds are generated daily, whether as domestic or industrial waste. In this sense, the present study aimed to produce an adsorbent material from coffee grounds and to evaluate its effectiveness as activated carbon in the removal of rhodamine FRBT dye in aqueous solution. To obtain the adsorbents, two types of treatment were carried out: (i) treatment 1 (T1): the adsorbent material was prepared with sulfuric acid; (ii) treatment 2 (T2): the adsorbent material was washed with water for chemical activation. The effectiveness of the adsorbents was evaluated by filtering the dye solution (60 $mg \cdot L^{-1}$ and 180 $mg \cdot L^{-1}$) as a function of the stirring time. The results obtained indicated that the T1 filter showed slight discoloration over time at the concentration of 180 $mg \cdot L^{-1}$ dye. However, a transfer of pigments from the coffee grounds to the coloring solution was observed, indicating that the filter was not effective when prepared with the selected methods and that possible treatment failures may have occurred in the preparation of the adsorbent material. Therefore, it is necessary to review the filter activation interference analysis, as well as possible different chemical and thermal treatments, for a better adsorption of substances by the adsorbent material.

Keywords: adsorbent; agricultural waste; textile effluents.

Resumo

A aplicação do carvão ativado como material adsorvente tem sido amplamente estudada em diversos âmbitos da indústria. Distintos materiais carbonáceos com potencial adsorvente, como a borra do café, têm mostrado propriedades de adsorção interessantes no que se refere à remoção de corantes têxteis de efluentes industriais. Altos volumes de borra de café são gerados diariamente, tanto como resíduo doméstico quanto como industrial. Nesse sentido, o presente trabalho teve como objetivo produzir um material adsorvente a partir de borra de café e avaliar sua eficácia como carvão ativado na remoção do corante rodamina FRBT em solução aquosa. Para a obtenção do material adsorvente, realizaram-se dois tipos de tratamento: (i) tratamento 1 (T1): material adsorvente preparado com ácido sulfúrico; (ii) tratamento 2 (T2): material adsorvente lavado apenas com água para ativação química. A eficácia dos adsorventes foi avaliada pela filtração da solução de corante (60 mg·L-1 e 180 mg·L-1) em função do tempo de agitação. Os resultados obtidos indicaram que o filtro T1 apresentou ligeira descoloração ao longo do tempo em 180 mg·L-1 de corante. No entanto, uma transferência de pigmentos da borra para a solução corante foi observada, indicando que o filtro não se mostrou eficaz quando preparado com os métodos selecionados e que possíveis falhas de tratamento podem ter ocorrido no preparo do material adsorvente. Diante disso, faz-se necessário uma revisão na análise de interferência de ativação do filtro, bem como possíveis tratamentos químicos e térmicos diferentes para uma melhor adsorção das substâncias pelo material adsorvente.

Palavras-chave: adsorvente; subproduto agrícola; efluentes têxteis.

1. INTRODUCTION

The use of industrial and agricultural waste has been the subject of countless studies on the use and reuse for generating new products with high added value (COSTA et al., 2017; HERMANN et al., 2020; SCHIPMANN et al., 2020). The significant volume of this waste generated daily, if not disposed of properly, may lead to different social and environmental problems. These effects have stimulated governmental charges, such as the Paris agreement, in which member countries committed themselves to developing new products and processes that are more sustainable and ecological (MMA, 2017). In order to minimize the costs associated with improper waste disposal, new technologies have been studied, developed, and employed (PEDRI, 2014).

Regarding the production of waste generated by the population, 50% of urban solid waste is organic; generally, its destination is dumps and landfills (MMA, 2017). Among the organic waste discarded daily, whether by domestic, commercial, or industrial use, we have residual coffee grounds as an example. Since Brazil is a large producer and one of the world's biggest coffee consumers, this waste generation is high (FIGUEIREDO; BOTARI, 2017). The presence of these grounds, if disposed of improperly, not only by the residual mass but also by the generation of liquids resulting from its decomposition, can cause phytotoxic effects on the flora and soil, providing an unsuitable environment for earthworms (THODE FILHO et al., 2017; FRANCO et al., 2020); this makes coffee grounds an interesting target in waste conversion research.

Coffee ground residue has great adsorptive power of chemical pollutants, and therefore, its action is assimilated to activated carbon (LIMA et al. 2014). This characteristic has stimulated studies on its use as a substrate in activated carbon production, which may be an alternative in producing low-cost adsorbents since activated carbons have high cost and need for their regeneration (SILVA et al., 2010; SANTOS; PONTES, 2018; FIGUEIREDO; BOTARI, 2017). The adsorption method using activated carbons has been considered effective in treating effluents containing dyes (FIGUEIREDO; BOTARI, 2017; SANTOS; PONTES, 2018); this is because among the primary pollutants studied in the process of sorption on activated carbon, phenol and its derivatives stand out (GUILARDUCI et al., 2006).

Synthetic dyes used in textile processes are not completely removed by conventional treatment methods due to the high biological stability of these molecules (FIGUEIREDO; BOTARI, 2017). Even at very low concentrations, dyes can have toxic effects and are extremely difficult to be removed due to their complex structure (SUYOG; PARAG, 2017). If these effluents are not properly treated, there may be a serious risk of contamination of the entire aquatic environment where the liquid will be discharged (CASTRO, 2009).

Among the synthetic dyes used in the textile and screen-printing industries is rhodamine, a highly water-soluble cationic xanthene dye with substituted amino groups (HERMANN et al., 2020). Numerous treatment methods have been studied in the decolorization of this dye, including photocatalytic, oxidative, biological, and bioelectro-Fenton (BEF) technologies, which combine the Fenton process with microbial fuel cell (MFC) technology (ALHAMEDI et al. 2009; HE et al., 2009; CUIPING et al., 2011; DAS et al., 2019; HERMANN et al., 2020), in addition to adsorption methods from adsorbents obtained from coffee waste (ANASTOPOULOS et al., 2017).

Using coffee grounds as adsorbent material reduces the costs of obtaining activated carbon, transforming a residue that can be applied in removing dyes from textile industrial effluents, reducing not only the levels of dyes but also the number of chemical agents used in the processes during treatment. Given this, this study aimed to produce an adsorbent material from coffee grounds previously treated with aqueous solution and acid solution and evaluate its effectiveness in removing rhodamine FRBT dye in an aqueous solution.

2. METHODOLOGY

2.1 Preparation of the adsorbent material

The adsorbent material was prepared according to the method described by (SANTOS; PONTES, 2018) and modified. In the pretreatment step, two kilograms (2 kg) of soluble coffee grounds from domestic use provided by private households (Ilhota, SC, southern Brazil) were washed with tap water at room temperature and dried in the sun for three days; 1 kg of washed coffee grounds was obtained. After sun drying, the grounds were again dried in an oven (Nova Ética, 402/3N) at 60 °C for 40 min. The adsorbent material was stored in plastic bags for further use.

Two different treatments for the activation of coffee grounds as adsorbent material were carried out on the pretreated material: In treatment 1 (T1), 240 g of coffee grounds were added to a solution of H2SO4 (0.4 mol L^{-1}) in the proportion of 1:4 (1 part of grounds for 4 of solution), stirring for 15 min in an orbital shaker (Fisatom) at 150 rpm, and resting for 15 min. The mixture was then filtered in a Buechner funnel using half-white mesh (20x20 cm) as a filter medium. After filtration, the coffee grounds retained in the mesh were washed with tap water, repeating the filtration process described above, and dried in an oven at 120°C for 40 min. Afterward, it was weighed to evaluate the extractive yield.

Treatment 2 (T2) consisted of weighing 240 g of coffee grounds from the pretreatment with further processing as described in T1, replacing the H_2SO_4 solution with tap water.

2.2 Adsorption kinetics and dye removal

For the dye removal assays, the method proposed by Santos and Pontes (2018) was used and modified. Two grams (2 g) of coffee grounds from T1 and T2 were weighed separately into 250-mL Erlenmeyer flasks with the addition of 100 mL of a rhodamine FRBT solution at concentrations 1 (C1) 60 mg L^{-1} and concentration 2 (C2) 180 mg·L^{-1}. The Erlenmeyer flasks were kept on a magnetic stirrer (80 rpm, AKSo HS19T) at room temperature (± 25 °C) under constant stirring for 15 min.

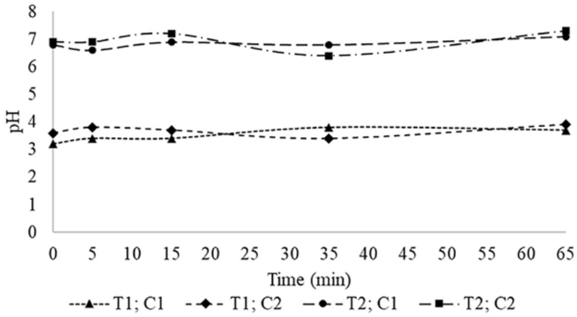
After this period, aliquots of the supernatant (5 mL) were removed using a graduated glass pipette at the following times: 0, 5, 15, 35, and 65 min after sedimentation of the adsorbent and filtered in a Buechner funnel using the qualitative paper (80 g·m^{-2}). After filtration, the filter paper with the dye solution was set aside and dried at room temperature.

The dye removal was determined spectrophotometrically using a UV-visible spectrophotometer (Datamo-lo, 600 TM) by reflectance and analyzed based on a gray scale. All dye removal experiments were performed in triplicate.

2.3 pH Determination

pH analysis was performed in T1 and T2 by the potentiometric method by adding 2 grams of sample in 10 mL of water and stirring for 15 min with subsequent readings in an AK90-Akso portable digital pH meter with a glass electrode after decanting each solution. In the dye removal kinetic assays, the pH reading occurred directly in the conical flask containing the rhodamine dye solution in C1 and C2 at different times (0, 5, 15, 35, and 65 min) after the solution was decanted.

Figure 1 - pH values over time in T1 and T2 on 60 mg L^{-1} and 180 mg L^{-1} of rhodamine FRBT.
T1 = treatment with H_2SO_4 solution; T2 = treatment with aqueous solution; C1 = 60 mg L^{-1}; C2 = 180 mg L^{-1}



Source: The authors (2022)

3. RESULTS AND DISCUSSION

The yield analysis of the adsorbent material obtained after the T1 and T2 for coffee grounds activation showed a higher yield (96.6%; 232 g) of adsorbent material when submitted to T2 compared to T1 (82.5%; 198 g). Usually, solvents such as H_2SO_4 assist in removing constituent compounds of coffee grounds, such as lignin, which can infer the adsorption of the dye on the adsorbent material (SUYOG et al., 2017; HERMANN et al., 2019). Its removal increases the porosity of the adsorbent material and, consequently, the surface area (SUYOG et al., 2017). Moreover, activation from the use of chemical solvents (chemical activation) generates coals with larger pores, which make them more suitable for adsorption applications in the liquid phase (JAIME, 2016). Removing the coffee grounds components, such as lignin, may have contributed to the lower yields in T1.

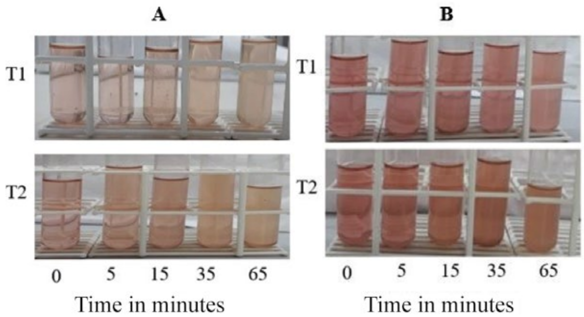
The initial pH values obtained in the adsorbent material after T1 and T2 were 2.0 ± 0.28 and 6.75 ± 0.45, respectively. For the initial rhodamine FRBT solution, the pH values before contact with the adsorbent material were 7.5 ± 0.24 for C1 and 7.5 ± 0.25 for C2. The results obtained for the pH over 65 min in T1 and T2 of the adsorbent material at C1 and C2 of rhodamine FRBT are shown in Figure 1.

The pH maintained a similar profile for C1 and C2 within the same treatment (T1 and T2), showing little difference in their values over 65 min (Figure 1). The lowering of the pH in the dye solution at T1 is associated with the type of treatment used since H2SO4 acid was used to activate the coffee grounds. Nonetheless, the neutralization of the adsorbent material is fundamental for correctly applying and analyzing the adsorption of the dye on the surface of the activated carbon. In this case, washing with tap water was not enough to completely remove the acid from the surface of the adsorbent material (T1), directly influencing the solution's pH. The pH of the solution plays a vital role in controlling the surface charge of the adsorbent, the degree of ionization of the adsorbate in the solution, and the dissociation of various functional groups on the active sites of the adsorbent (ANASTOPOULOS *et al*., 2017).

3.1 Adsorption kinetics

Figure 2 shows the results of the contact time of the adsorbent material on dye removal in T1 and T2 for the concentrations of 60 and 180 mg L^{-1} over 65 min.

Figure 2 - Visual discoloration over 65 minutes for treatments T1 and T2 at the concentration of 60 mg L^{-1} (A) and 180 mg L^{-1} (B) of rhodamine FRBT. T1 = treatment with H_2SO_4 solution; T2 = treatment with aqueous solution



Source: The authors (2022)

We did not observe any significant color reduction over 65 min for any of the treatments tested at the concentrations analyzed (Figure 2). A trend towards turbidity was observed for T2 at 60 (A) and 180 mg.L^{-1} (B) after 35 min of contact of the dye solution with the adsorbent material. This turbidity may be associated with pigments from the coffee grounds that were not completely extracted during pretreatment and aqueous treatment (T2) and subsequently transferred to the dye solution.

Although the visual increase in turbidity is more evident for T2, the results obtained by spectrophotometric analysis (Figures 3 and 4) show an increase in color early on the contact of the dye solution with the adsorbent material.

Figure 3 - Kinetic profile of dye decolorization between 0 and 65 min for the concentration of 60 mg L^{-1} of rhodamine FRBT. T1 = treatment with H_2SO_4 solution; T2 = treatment with aqueous solution. Lines: black = pure solution at concentration of 60 mg L^{-1}; red = 0 min; green = 5 min; blue = 15 min; orange = 35 min; purple = 65 min.

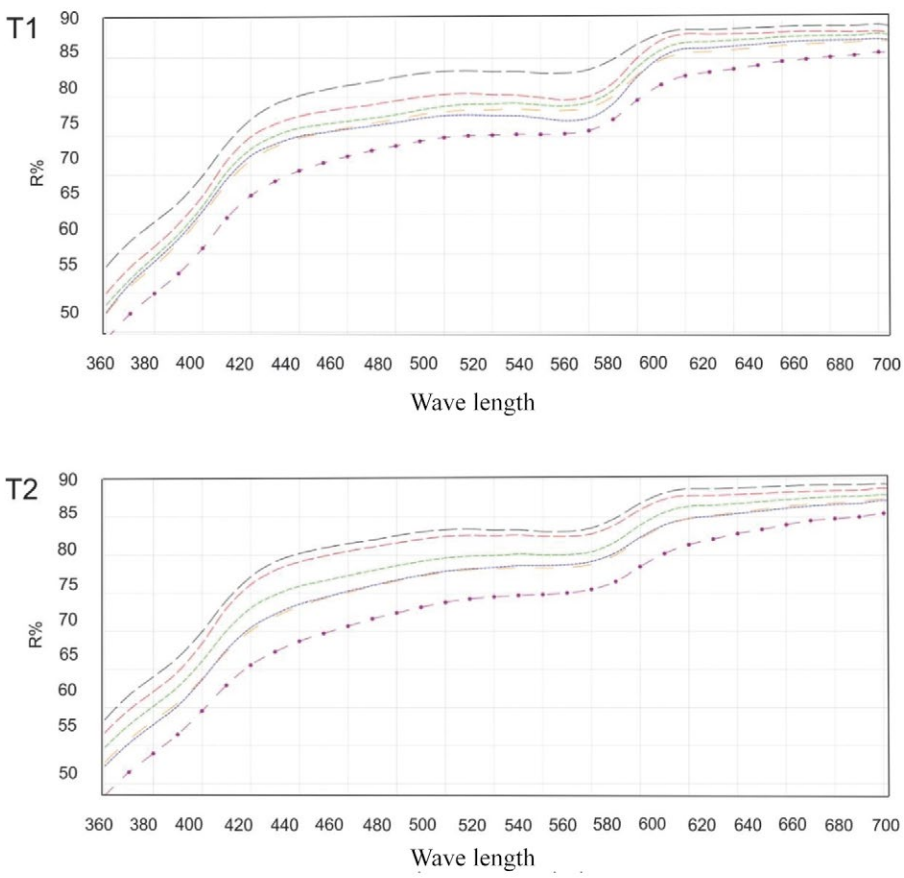


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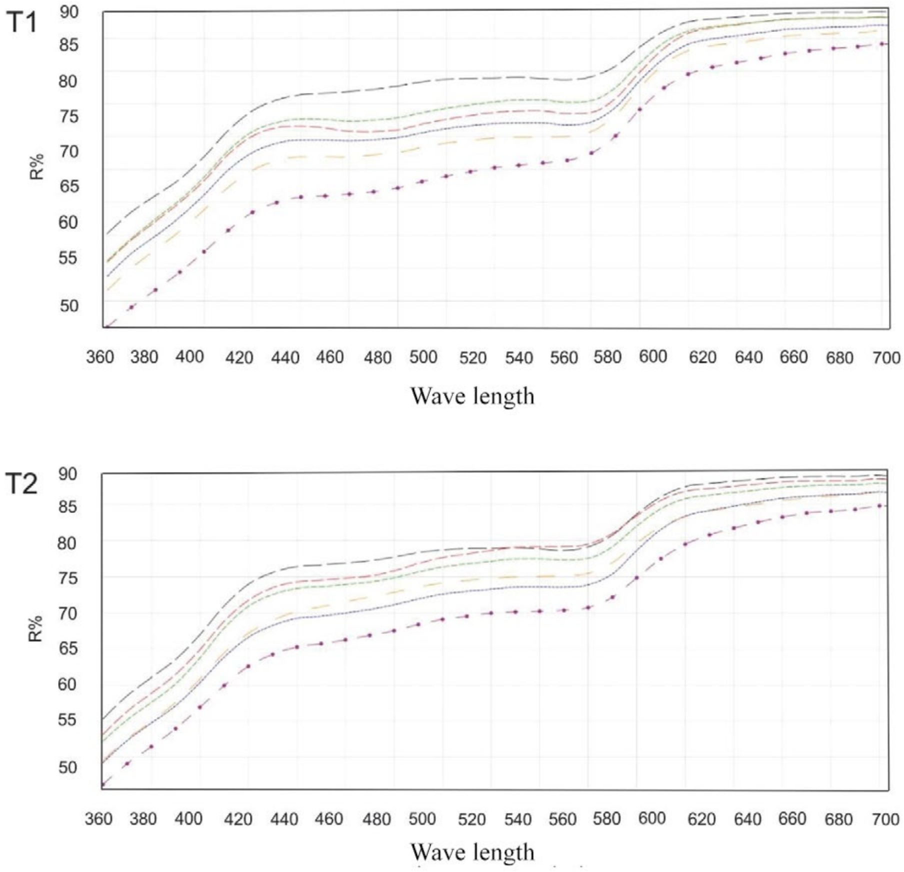
Figure 4 - Kinetic profile of dye decolorization between 0 and 65 min for the concentration of 180 mg L-1 of rhodamine FRBT. T1 = treatment with H2SO4 solution; T2 = treatment with aqueous solution. Lines: black = pure solution at concentration of 180 mg L-1; red = 0 min; green = 5 min; blue = 15 min; orange = 35 min; purple = 65 min.



Source: The authors (2022)

Rhodamine is a dye used in industrial textile processes and screen printing (HERMANN et al., 2020). It has a well-defined wavelength, considering a visible spectrum range of 500–650 nm. The literature shows wavelengths around 557 nm (HERMANN et al., 2020) and 600 nm (MANOEL, 2017). This study performed a scan between 360 and 680 nm, where the largest reflectance peaks occur in the 600 nm range. As the contact time of the dye solution with the adsorbent material increases (0–65 min; Figures 3 and 4), the reflectance index (%R) decreases, showing that some pigments from the coffee grounds were not fully extracted during the pretreatment and during the aqueous treatment migrated into the solution, as evidenced in Tables 1 and 2 in grayscale values.

Table 1 - Grayscale for 60 mg L^{-1} of rhodamine FRBT for 65 min

TIME	GRAYSCALE VALUES	
	T1	T2
0 min	3.16 ± 0.20	3.48 ± 0.08
5 min	3.32 ± 0.21	3.34 ± 0.14
15 min	3.19 ± 0.09	3.42 ± 0.11
35 min	3.01 ± 0.19	3.27 ± 0.08
65 min	2.96 ± 0.08	3.10 ± 0.23
Dye solution	3.83	

T1 = treatment with H_2SO_4 solution;
T2 = treatment with aqueous solution

Source: The authors (2022)

Table 1 - Grayscale for 180 mg L^{-1} of rhodamine FRBT for 65 min

TIME	GRAYSCALE VALUES	
	T1	T2
0 min	3.12 ± 0.20	3.63 ± 0.20
5 min	3.00 ± 0.15	3.62 ± 0.29
15 min	3.18 ± 0,11	3.39 ± 0.11
35 min	3.04 ± 0,21	3.43 ± 0.10
65 min	2.60 ± 0.30	3.31 ± 0.11
Dye solution	3.33	

T1 = treatment with H_2SO_4 solution;
T2 = treatment with aqueous solution

Source: The authors (2022)

Applied in the textile industry to measure color fastness, grayscale values are used to evaluate the color change or coloration of the sample. The color change or coloration scale consists of nine pairs of gray and white color chips of values from 1 to 5, whereby the closer to 5, the lower the coloration of the sample.

The color evaluation occurs within the ABNT technical evaluation standard, following the ISO 105-A02 standard of textile color fastness test part A02 (grayscale to evaluate color changes) (CRUZ, 2014). In the tests performed in this study, we observed that with the passage of time (Tables 1 and 2), a greater coloration in the sample was measured, evidencing the transfer of pigments from the adsorbent material to the effluent in addition to its inefficiency. For this material as an adsorbent, it is necessary to be washed until the water is completely clear, thus avoiding a transfer of pigments from the grounds to the effluent and compromising the adsorbent material's efficiency. Moreover, completely removing the acid solution used in the pre-preparation (T1) is also fundamental since, in acidic solutions, dyes such as rhodamine show lower removal percentages compared to removal percentages in neutral or basic solutions (QUINTINO, 2021). Not only does the pH show influence in removing the dye by adsorption, but also interaction forces between the adsorbate and the functional groups present on the surface of the adsorbent (e.g., hydrogen bridges) influence the adsorption, consequently removing dye from the aqueous solution (QUINTINO, 2021). Furthermore, the coffee grounds were washed with tap water, in which several dissolved ions and organic matter predominate, which can compete for the active site of the adsorbent material (QUINTINO, 2021), thereby compromising the adsorption of other components such as rhodamine.

The activation of coffee grounds as activated carbon and its subsequent removal in dyes (e.g., methylene blue, rhodamine B, and rhodamine 6G) has been evaluated from activations with acidic, basic, and neutral solutions (FIGUEIREDO; BOTARI, 2017; SHEN; GONDAL, 2017). Percentages of methylene blue removal above 90% were obtained elsewhere by Figueiredo and Bondari (2017) after 20 min of contact of the solution with activated carbon, showing more effective results for the activated material with H_3PO_4, with 92% removal of the dye after 9 min. In this study, using coffee grounds as an adsorbent material for removing rhodamine FRBT proved unsatisfactory. This low efficiency in removing the dye may be associated with the difficulty of adsorption of the dye on the adsorbent material because of components still present in the coffee grounds due to lack of adequate pretreatment in obtaining the adsorbent. The low drying temperature (60 °C) of the coffee grounds in the pretreatment may have also contributed to the dye's adsorption inefficiency. The pyrolysis or carbonization step, carried out at temperatures up to 800 °C, removes the volatile materials present in the sample and forms pores. These pores will remain clogged until activation, which is fundamental for activated carbon effectiveness (DE ALMEIDA E SILVA et al., 2017). Due to the limitations of the equipment used, such as the lack of muffle for high temperatures, this carbonization step was not performed. Moreover, the changes required on the surface of the adsorbent material to obtain the activated carbon are influenced by the agent used (chemical and physical), time, and activation temperature (DE ALMEIDA E SILVA et al., 2017). The procedures used in this study at T1 and T2 to activate the material were ineffective in changing the grounds' chemical surface to become an active material in removing rhodamine dye

4. CONCLUSIONS

The adsorption potential of the adsorbent material produced from the residual coffee grounds in relation to the rhodamine FRBT dye was ineffective under the evaluated conditions. The absence of high-temperature thermal treatment, performed at the beginning of the process, may have compromised the adsorption potential of the coffee grounds since the volatilization of compounds in the material and the formation of pores are essential for subsequent activation. Chemical activation is widely applied in activated carbon production. Nevertheless, the low solvent removal and its interference with the pH of the solution may have contributed to the low adsorption of the dye and its removal. Moreover, the components present in tap water used to wash the material may compete with the active sites of the sample, interfering in the process. Given the abundance of coffee grounds generated daily and the development of new alternative materials to treat aqueous effluents containing contaminants from the textile industry, new tests involving improvements in the preparation of the adsorbent material, such as heat treatment, pH control, and temperature, are necessary to evaluate the efficacy of coffee grounds as an adsorbent material in the treatment of textile effluents using other types of dyes.

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