Removal of Anionic Dye from Tannery Wastewater Using Peanut Shell Waste as a Biosorbent

by

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Abstract

Contamination of water bodies by organic substances, such as dyes, cause impacts on the environment and human health. Furthermore, the search for environmentally appropriate destinations for solid waste, such as those from agro-industries, has been intensifying, to minimize the impact of the growing generation of waste as a result of population growth. Thus, this work aimed to evaluate the use of raw peanut shells as an alternative adsorbent for the removal of Brown acid dye from synthetic leather dyeing wastewater. The peanut shells were milled and sieved. The material was characterized by Fourier Transform Infrared Spectroscopy. The parameters evaluated in the adsorption tests were pH, contact time, and mass of adsorbent material. The maximum adsorption capacity verified for the peanut shell was 7.753 mg/g and up to 93% of the dye was removed. The best adsorption conditions were at pH 2.5, 10 minutes, and adsorbent mass of 0.3 g in 50 mL of effluent. The results showed relevant removal of the Brown acid dye by the peanut shells' adsorbent material.

Introduction

Leather processing consists of a sequence of physical-chemical and mechanical operations applied to the raw hide using water and organic and inorganic chemicals. During the post-tanning stage, specific characteristics are conferred for each leather application, such as color, mechanical strength, and softness. An average of 360.2 kg of chemicals are applied per ton of shaved leather in the post-tanning process.[1] Among the chemicals used are dyes, which are natural or synthetic complex organic molecules. Dyes are commercially named as direct, acidic, reactive, dispersed, and basic, among others. In the leather dyeing stage, the most used dyes are acidic and reactive. During the process, 20% of the dye load does not fix to the leather and is discharged along with the wastewater. About 700 thousand tons/year of dyes are manufactured in the world, with acid dyes being the second-largest commercialized class, finding wide application in leather process.[2,3] Dyes make the wastewater treatment difficult. The presence of dyes in formulations, such as azo and metal complexes, inhibits aquatic life growth,[4] and induces acute and chronic cytotoxicity.[4,5,6]

The treatment conventionally used for tannery wastewater consists of the application of equalization and neutralization operations, physical-chemical treatment through coagulation and flocculation followed by sedimentation, and biological treatment, with activated sludge being applied more frequently.[7] However, the literature points to sludge generation and low efficiencies for the removal of salts and recalcitrant compounds (including dyes) as vulnerabilities of this treatment.[7-14] Furthermore, environmental regulation is increasingly restrictive regarding the standards for releasing effluents into water bodies, and conventional treatment does not meet the requirements for the reuse of treated effluents in the process.[7]

To overcome the vulnerabilities of conventional treatment of tannery wastewater, advanced technologies have been studied. Adsorption is among the technologies adopted due to its low initial investment and operating costs, simplicity of operation, and high efficiency compared to conventional processes.[2,3,15] Alternative adsorbents such as bark, seeds, and bagasse wastes have already shown high efficiency in the removal of dyes from industrial effluents, presenting low cost, easy obtaining, high volume generated, and simple preparation of the adsorbent.[16] However, the use of peanut shells as a biosorbent for the removal of dyes from the leather industry still needs to be investigated. Thus, this work aimed to evaluate the use of peanut shells as an alternative adsorbent for the treatment of tannery wastewater, applied to the removal of Brown acid dye. The use of an adsorbent prepared with minimal application of chemicals, energy, and water was also an objective of this study, applying only peanut shell size reduction and sieving for the preparation of the sorbent material.

Experimental

Preparation of peanut shells

The raw peanut shells (Figure 1a) were selected, removing impurities. They were milled in a blender and classified in an electromagnetic sieve shaker for 30 minutes, obtaining a fraction of solids passing through the Tyler 42 mesh sieve (0.351 mm) (Figure 1b). After preparation, the peanut shells were stored in a desiccator.

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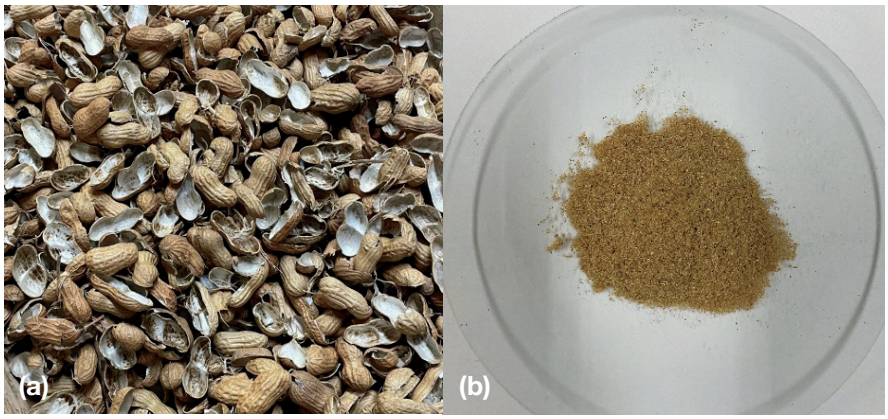

Figure 1. Peanut shells before **(a)** and after **(b)** the milling and sieving process

Analysis of peanut shells by Fourier Transform Infrared Spectroscopy

Peanut shells prepared by milling and sieving were characterized by Fourier Transform Infrared Spectroscopy (FTIR) before and after adsorption tests. FTIR analyzes were performed in the region of 4,000-650 cm^{-1}, with a resolution of 4 cm^{-1} in the Agilent Cary 630 equipment. The analysis aimed to evaluate the main functional groups present in the peanut shell that allow its binding with the Brown acid dye.

Preparation of synthetic wastewater containing dye

The Brown acid dye (CAS number 70210-34-3) was used in this work. The chemical structure of the dye (Figure 2) presents sulfone and nitro functional groups that are negatively charged sites capable of binding to the leather structure. In this study, the synthetic effluent consisted of a Brown acid dye solution at a concentration of 50 mg/L. A UV-Visible spectrophotometer (SP-220 Biospectro) was used to determine the dye concentration. Measurements were determined at the wavelength of maximum absorbance (468 nm).

Adsorption experiments

The variables evaluated in the adsorption experiments were pH, time, and adsorbent mass. The efficiency of dye removal from synthetic wastewater was determined using equation 1 and the amount of dye adsorbed per gram of adsorbent was calculated using equation 2. All analyzes were performed in duplicate.

$$\% \text{ Removal} = \frac{Co - Cf}{Co} \times 100 \quad (1)$$

$$q_t = \frac{(Co - Cf) * V}{m_{ads}} \quad (2)$$

Where:

Co – initial concentration of Brown acid dye (mg/L)

Cf – final concentration of Brown acid dye (mg/L)

q_t – amount of dye adsorbed (mg/g)

V – volume of solution (L)

m_{ads} – mass of adsorbent (g)

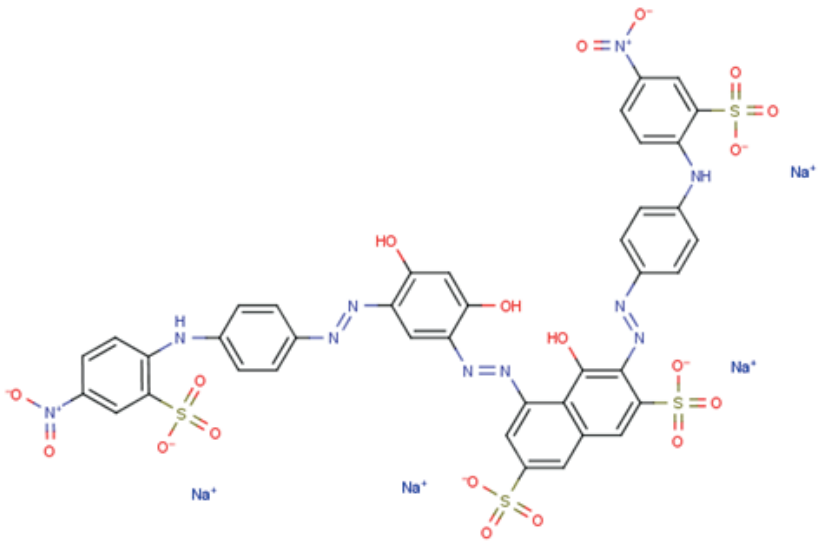

Figure 2. Chemical structure of the Brown acid dye

To determine the best pH for adsorption of the Brown acid dye, 50 mL of synthetic wastewater (50 mg/L of Brown acid dye) were added to 250 mL Erlenmeyer and 0.5 g of peanut shells were used. The pH was adjusted to 2.50, 4.00, 5.00, 6.00, 7.00, 8.00 and 9.00, with 0.1 mol/L HCl and 0.1 mol/L NaOH solutions. The system was stirred at 100 rpm at 25°C for 24 hours to ensure equilibrium between the solid and liquid phases. After this process, the solutions were centrifuged at 10,000 rpm for 10 minutes and the absorbance was measured using a UV-visible spectrophotometer with a wavelength of 468 nm.

The pH that showed the highest efficiency in the removal of the Brown acid dye was used to evaluate the influence of the adsorption time on wastewater treatment. Fifty milliliters of synthetic wastewater (50 mg/L of Brown acid dye) and 0.5 g of peanut shell were added to 250 mL Erlenmeyer. The Erlenmeyer was stirred at 100 rpm at 25°C. The removal efficiency (equation 1) of the Brown acid dye was evaluated at time intervals of 10, 30, 60, 90, 120, 150, and 180 minutes. The solutions were centrifuged at 10,000 rpm for 10 minutes and the absorbance was measured using a UV-visible spectrophotometer at a wavelength of 468 nm.

The experiments to evaluate the effect of the adsorbent mass on dye removal were carried out in 250 mL Erlenmeyers containing 0.3, 0.4, 0.5, 0.6, and 0.7 g of the adsorbent material and 50 mL synthetic wastewater (50 mg/L Brown acid dye). The Erlenmeyers were stirred at 100 rpm for 90 minutes at 25°C. The solutions were centrifuged at 10,000 rpm for 10 minutes and the absorbance was measured in a UV-visible spectrophotometer with a wavelength of 468 nm.

Statistical analysis

Dye removal data obtained for different pH values, contact times, and adsorbent mass were analyzed by one-way Analysis of Variance (ANOVA) followed by Tukey's post-hoc test and were considered significant when $p<0.05$. All statistical analyzes were performed using GraphPad Prism 5.0 software.

Results and discussion

Analysis of peanut shells by Fourier Transform Infrared Spectroscopy

The peanut shell FTIR spectra are shown in Figure 3, before (Figure 3a) and after (Figure 3b) adsorption of the Brown acid dye.

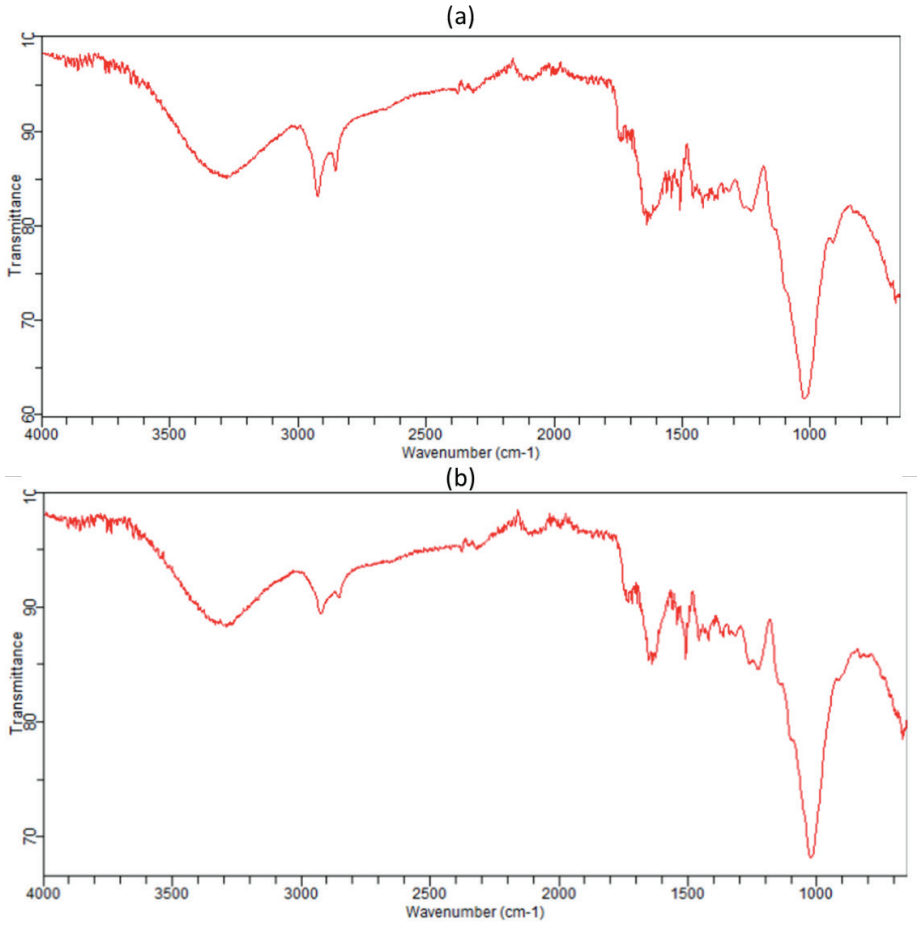

Figure 3. FTIR spectra of peanut shell before **(a)** and after **(b)** adsorption experiments

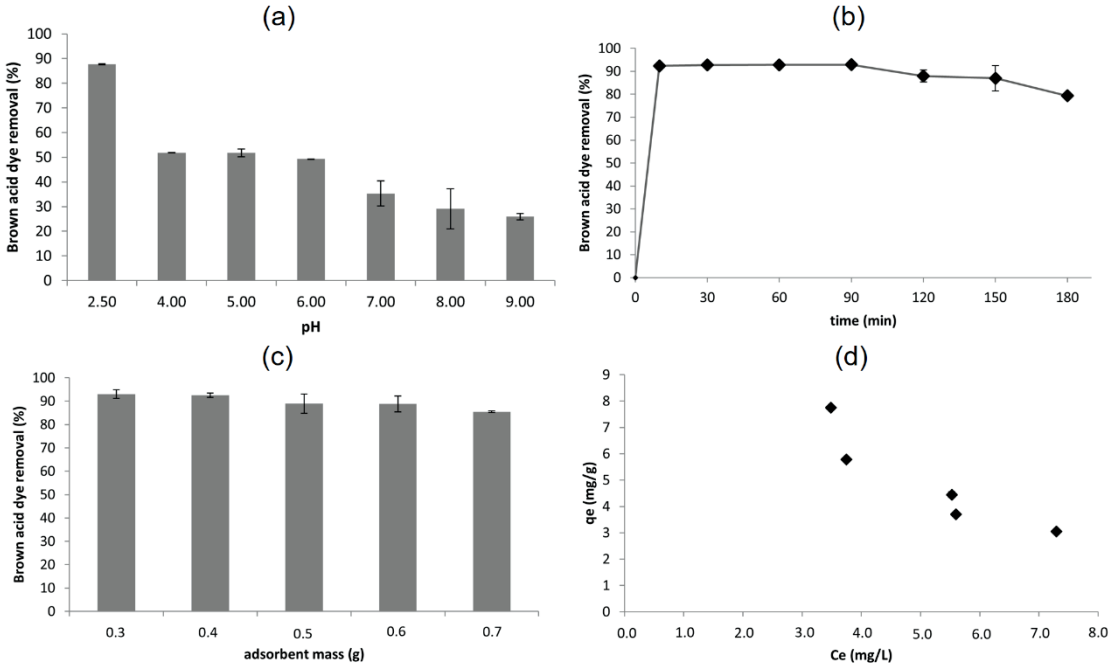

Figure 4. Effect of pH **(a)**, time **(b)** and adsorbent mass **(c)** on Brown acid dye removal, and sorption capacity by peanut shells **(d)**.

The FTIR spectra (Figure 3a) showed in the range from 3200 to 3500 cm^{-1} a band that belong to the functional groups -OH and/or -NH. Around 2800 and 3000 cm^{-1}, vibrational peaks with asymmetric and symmetrical stretching, respectively, of the C-H bond of CH_2 groups were verified (Figure 3a). The band around 1700 cm^{-1} is characteristic of the stretching of C=O, a possible constituent of carbonyl, carboxyl or aromatic compounds. The vibrations at 1400 and 1050 cm^{-1} can be attributed to the C-O bonds of the carboxylic and alcoholic groups. A band between 1300 and 1400 cm^{-1} represents the methyl group CH_3 and between 1350 and 1550 cm^{-1} the nitro group[17]. The groups amide, carboxyl, and hydroxyl in the chemical composition of the biomass structure is related to the presence of proteins, fatty acids and carbohydrates in the peanut shell.[18,19] Also, the CH_2 groups can represent the lipidic compounds in the peanut shell. The C=O functional group is also present in the chemical structure of lipids.[20]

The chemical structure of the Brown acid dye (Figure 2) presents sulfone and nitro groups, and the FTIR analysis showed that the raw peanut shell (Figure 3a) contains amide groups. Thus, considering that the acidic environment can protonate the amide groups[21] present in the structure of the peanut shell, it is possible to observe the potential interaction of the sulfone and nitro groups (negatively charged) present in the dye with the amide groups (positively charged) in the peanut shell, enabling the adsorption process.

The superposition of the FTIR spectra before (Figure 3a) and after (Figure 3b) the adsorption experiments showed changes in the region of 1700 cm^{-1}, with a modification in the peak of the C=O bond. Furthermore, in the region of 1500 and 1600 cm^{-1}, a vibration from the N-H bond occurred, possibly due to the interaction of the amide group in the peanut shell with the sulfone and nitro groups in the Brown acid dye.

Adsorption experiments

The results of Brown acid dye removal are shown in Figure 4 under different conditions of pH (Figure 4a), adsorption time (Figure 4b), adsorbent mass (Figure 4c). The sorption capacity of Brown acid dye by peanut shells is also presented (Figure 4d).

The wastewater pH affects dye removal since the surface charge of the adsorbent controls the interaction of its ions. The Brown acid dye has sulfone and nitro anionic groups, thus, a positive charge on the surface of the biosorbent must have a higher and better dye adsorption ability.[22] The maximum removal occurred at pH 2.5 (Figure 4a), where the amount of H^+ ions increases, improving the electrostatic attraction between the dye and the biosorbent.[23]

The statistical analysis of the experimental data showed a significant difference among the various pH values used in the adsorption ($p < 0.0001$). Tukey's post-hoc test showed that there was a significant

difference in dye removal when pH 2.5 was compared with the other pH values, confirming that Brown acid dye removal is significantly higher at pH 2.5. There was no significant difference in dye removal when pH values 4.0, 5.0 and 6.0 were compared. This pH range shows intermediate efficiency for dye removal. When comparing the removal efficiency for pH values 7.0, 8.0 and 9.0, there was also no significant difference, with this pH range being the least efficient.

A previous study[24] found that the zero-charge point of peanut shells occurs at pH 7.10 ± 0.10. Values of pH below the zero-charge point cause a positive charge to the peanut shell surface, and pH values above the zero-charge point cause a negative charge to the adsorbent surface.[25] This zero-charge point is consistent with the results found in this study for adsorption at different pH values, with higher removals at pH values below 7.0.

The evaluation of the adsorption time of the Brown acid dye was carried out in the best pH condition found in the previous step (pH 2.5). The dye removals for the tested contact times are presented in Figure 4b. High removals were promptly achieved. Removal efficiencies greater 92% were obtained in the first 10 minutes of adsorption. This behavior occurs due to the free sites on the surface of the adsorbent material, considering that at the beginning of the adsorption process, the binding sites are available in greater quantity.[26] Maximum removal occurred at 90 minutes (92.9 ± 0.1 %). After this period the removal efficiency gradually decreased.

The statistical analysis of the data showed a significant difference in the dye removals obtained for the contact times ($p = 0.0051$). Tukey's post-hoc test showed that although the highest dye removal occurred at 90 minutes, no statistical difference was identified in removal efficiency for the period from 10 to 150 minutes. Though, the removal was significantly lower when the adsorption was carried out up to 180 minutes. The reduction of removal with time is not an expected behavior in an adsorption process and may be related to the release of colored compounds from the adsorbent material due to the similarity between the colors of the peanut shell and the Brown acid dye. When peanut shells were used to remove blue dyes from the textile industry (Reactive Blue 203[27] and methylene blue[24]), and yellow, green, and amaranth anionic dyes,[28] this effect was not observed, supporting the theory that peanut shells released substances with a color similar to the Brown acid dye evaluated in the present study.

The effect of the adsorbent mass on the dye removal efficiency was evaluated under the best conditions of pH (2.5) and adsorption time (90 minutes). Dye removal efficiencies are presented in Figure 4c. The results demonstrate higher dye removal efficiencies for the 0.3 g (93.0 ± 1.9%) and 0.4 g (92.5 ± 0.9%) peanut shell masses. The removal ratio decreased when the adsorbent mass increased, with removal efficiencies between 88.8 ± 3.4% and 85.4 ± 0.3% for the peanut shell masses of 0.6 and 0.7 grams, respectively. These results are consistent with the phenomenon observed in the time experiments. They corroborate the thesis that although the adsorbent material can remove the Brown acid dye, it releases colored substances with increasing time and adsorbent mass. The statistical analysis of the data indicated that, despite the behavior of the experimental data showing that the increase in the adsorbent mass reduces the dye removal, no significant differences were observed with the different adsorbent masses ($p=0.1376$).

The sorption capacity of Brown acid dye by peanut shells (q_e) (Figure 4d) was calculated (equation 2) and evaluated as a function of the dye concentration in the liquid phase. This evaluation demonstrates how the dye molecules are distributed between the biosorbent and the synthetic wastewater. The highest sorption capacity occurred with the lowest peanut shell masses. The dye concentrations in the synthetic wastewater after adsorption were 3.5 ± 0.9 mg/L and 3.7 ± 0.5 mg/L for the 0.3 and 0.4 g of peanut shell masses, respectively. Thus, the equilibrium concentrations (C_e) were the lowest for the lowest masses of adsorbent. When the mass of adsorbent increases, a decrease in color removal is observed. This behavior is not expected as the dye removal generally increases with the amount of adsorbent and the number of adsorption sites.[29] However, this behavior can be attributed to colored substances released by peanut shells used in the present study. The highest sorption capacity experimentally obtained was 7.753 mg/g. The maximum sorption capacities and removal efficiencies obtained by other studies that applied raw peanut shells are shown in Table I.

The maximum removal efficiency of the Brown acid dye obtained in the present study (93%) was superior to the removal of the Reactive blue 203[27] and the Crystal violet[19] dyes, being close to the removal efficiencies of Amaranth, Sunset yellow, Fast green FCF[28] and Methylene blue.[24] Due to the sorption capacity decreasing with increasing adsorbent dosage, the sorption capacity obtained in the present study was lower than that obtained for other dyes, but above the sorption capacity found for the methylene blue dye.[24]

The spent adsorbent applied to remove the Brown acid dye may require disposal after desorption of the dye or disposal even without desorption. There are four approaches that have been employed in the management, disposal and reuse of used adsorbents:[30] reuse (as soil amendment, depending on material toxicity), incineration (facilitating recovery of energy and hazardous materials), landfill (depending on the concentration of adsorbate in the spent adsorbent) and other safe disposals (e.g.: stabilization/solidification in inert material[31] and phytoremediation). Additional research related to recovery, regeneration and final disposal of used adsorbents is needed to deliver better results and provide a new management path.[30]

Table I

Removal efficiency and sorption capacity of dyes by peanut shell

Dye	Removal efficiency (%)	Sorption capacity ($mg.g^{-1}$)	Author
Reactive Blue 203 (textile reactive dye)	66.83 %	49.29	27
Amaranth (Am), Sunset yellow (SY) and Fast green FCF (FG)	Am: 98.7 % SY: 99.57 % FG: 99.12%	Am: 14.90 SY: 13.99 FG: 15.60	28
Methylene blue (textile basic dye)	> 90 %	3.65	24
Crystal violet (cationic dye)	74 %	24.37	19

Conclusion

The results presented in this work showed the possible use of raw peanut shells as an adsorbent for the removal of Brown acid dye from wastewater. The adsorptive capacity of peanut shells increased with the decrease in the pH of the solution (pH 2.5 showed the highest removal of the Brown acid dye). The contact time with the highest efficiency in dye removal was 90 minutes. However, with 10 minutes of adsorption, 92.3% of the dye was already removed, and there was no significant increase in removal efficiency between 10 min and 90 min. Thus, it is recommended the use of 10 min of adsorption.

The smallest masses of peanut shell showed greater efficiency in removing the Brown acid dye, which was related in this study to the release of colored substances by the adsorbent material, due to the similar color of the peanut shells compared to the evaluated dye. The highest sorption capacity of dye by the peanut shell was 7.753 mg/g, corresponding to 93% removal. This result was obtained when the lowest peanut shell mass was added (0.3 g). The results demonstrate that the biosorbent is a viable alternative for the removal of Brown acid dye.

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