

Floating and Magnetic Composite Based on FeMnO_3 /ACC: An Efficient Adsorbent for Removing Toxic Dyes in Aqueous Media

Compósito Flutuante e Magnético à Base de FeMnO_3 /ACC: Um Adsorvente Eficiente para Remoção de Corantes Tóxicos em Meio Aquoso

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Lately, despite the scarcity of water resources, there has been a significant and unsustainable increase in global freshwater consumption, driven by the expanding industrial activity and world population. Water pollution by organic compounds such as dyes, has attracted the attention of the scientific community and government agencies. These pollutants are typically highly toxic, persistent, and prone to bioaccumulation in the environment. Despite these characteristics, they are used in substantial quantities and continually introduced into the aquatic environment, directly impacting animals, humans, and biodiversity. Therefore, in this context, adsorption has gained notoriety due to its high performance, accessibility, simplicity, applicability, and low cost. In this sense, the present work describes the preparation, characterization and application of a magnetic and floating adsorbent composite designed for the removal of toxic organic dyes from the aqueous medium. The application of the composite showed that it is possible to reduce the toxicity of solutions by up to 70%.

Keywords: Adsorbent; float; magnetic; organic contaminants; decreased toxicity.

1. Introduction

Water is one of the most valuable natural resources on the planet, essential for sustaining life.¹ In recent years, even with the growing scarcity of water resources, freshwater consumption has increased unsustainably due to the expansion of industrial activities and global population growth.² Additionally, human activities have caused serious risks to the environment and human health, with profound impacts on soil and water, especially due to the improper disposal of industrial waste, often without adequate treatment.^{3,4}

Water pollution by organic and inorganic compounds, such as dyes, pharmaceuticals, and metal ions, has attracted the attention of the scientific community and governmental agencies, as these pollutants are highly toxic, persistent, and prone to bioaccumulation in the environment.^{5,6} Despite their detrimental characteristics, these compounds are still widely used and continuously introduced into the aquatic ecosystem, directly impacting animals, humans, and biodiversity.⁷

Therefore, the development of materials and processes for the treatment, purification, and reuse of water contaminated with industrial waste is crucial.⁸ This issue has been widely investigated by various research groups around the world.^{9,10} Among the different processes being studied for the removal of contaminants from water, adsorption has gained prominence due to its high performance, accessibility, simplicity, and applicability.¹¹⁻¹³ This process is based on the physicochemical processes of solute accumulation on a surface through mass transfer between the liquid phase and the solid phase.

Various materials have been employed as adsorbents for the removal of contaminants from water, such as activated carbon, zeolites, and synthetic resins.¹⁴ However, a notable operational challenge is the separation of the adsorbent from the solution, which results in increased process costs. To minimize this problem, recent studies have suggested that ceramic magnetic materials hold great promise as adsorbents for wastewater treatment.¹⁵ These materials offer efficient alternatives for separating substances and can be easily removed from the solution using a magnetic field. Among these materials, ferrites, a class of magnetic metal oxides with the empirical formula MFe_2O_4 (M = transition metal), with iron as the main constituent, have shown great potential but remain relatively unexplored for this application.¹⁶

Among magnetic ferrites, manganese ferrite (FeMnO₃) stands out as a perovskite ferrite material due to its highly stable and versatile crystal structure.¹⁷ Perovskite ferrites possess a cubic structure that allows structural modifications to optimize their magnetic properties. FeMnO₃ exhibits exceptional characteristics such as high thermal and chemical stability, high saturation magnetization, and paramagnetic behavior.¹⁸ These properties make FeMnO₃ particularly suitable for wastewater treatment, as it can be easily recovered using a magnetic field, eliminating the issue of separating the adsorbent from the solution.^{17,18}

Additionally, the perovskite structure of manganese ferrites provides resistance to high temperatures, stability in aqueous environments, and high adsorption capacity, factors that make FeMnO₃ ideal for adsorbing toxic organic compounds and metal ions. The structural flexibility of this class of materials also allows for adjustments in composition aspect that can be optimized for enhanced adsorption efficiency.¹⁷

Autoclaved cellular concrete is a promising alternative compared to other materials that are generally used as adsorbents, since it is a relatively less expensive material, and can present good efficiency.¹⁹

In this context, the present work describes the preparation, characterization, and application of a composite based on manganese ferrite (FeMnO₃) and autoclaved cellular concrete (ACC) as a floating magnetic adsorbent, for the removal of toxic organic dyes from aqueous media. Furthermore, the toxicity of the solutions after the adsorption process was investigated, demonstrating that the application of the composite as a sorbent results in significantly less toxic solutions.

2. Experimental

2.1. Reagents and synthesis of polymeric resin precursor of FeMnO₃

The precursor polymeric resin of FeMnO₃ was obtained by the Pechini method,²⁰ using Fe(NO₃)₃·9H₂O (Sigma Aldrich), Mn(NO₃)₂·4H₂O (Sigma Aldrich), citric acid (Sigma Aldrich), and ethylene glycol (Sigma Aldrich) as starting reagents. All reagents were used as received without further purification.

The Pechini method is a chemical synthesis technique that entails the formation of a gel-like precursor through a complexation reaction, typically utilizing citric acid as a chelating agent. The subsequent heating process is aimed at decomposing the organic components, facilitating the formation of a metal oxide. The complexation reaction was conducted by dissolving Fe(NO₃)₃·9H₂O, Mn(NO₃)₂·4H₂O, and citric acid in distilled water. The mixture was stirred slowly and heated between 60 and 70 °C for the Fe/Mn complexation process. Subsequently, ethylene glycol was added to the solution obtained from complexation process

for the polyesterification or polymerization reaction of the Fe/Mn. This procedure was carried out under slow stirring and heating at 50 °C. The stoichiometric proportion used for the precursor reagents in the synthesis of the polymeric resin was 1 mol Fe(NO₃)₃·9H₂O : 1 mol Mn(NO₃)₂·4H₂O : 4 mol citric acid : 16 mol ethylene glycol (1:1:4:16).

2.2. Syntheses of the FeMnO₃/ACC composite

After the synthesis of the polymeric resin, pieces of ACC were cut into prismatic shapes (approximately 2 × 2 × 1 cm) and immersed in this resin for 60 seconds. After this procedure, they were dried in an oven for 15 minutes at 110 °C. Empirically, it was determined that performing this procedure five times would be necessary to achieve complete coverage of the ACC surface. Finally, the ACC prisms coated with the precursor resin of FeMnO₃ were heat treated at 600 °C for 3 hours to obtain the FeMnO₃/ACC composite.^{19,21}

2.3. Characterization of ferrite precursor polymeric resin

2.3.1. Viscosity measurements

In order to quantitatively determine the viscosity of the polymeric resin obtained, measurements were made on a Brookfield model DV2T viscometer. Experiments were carried out in triplicate under the following conditions: rotation 100 RPM, spindle 61, and torque percentage of approximately 14%.

2.3.2. Thermogravimetric analysis

With the aim of optimizing the heat treatment of the polymeric resin in order to obtain the inorganic phase (ferrite), a thermogravimetric study of the resin in powder form was carried out. It was conducted on a Shimadzu thermobalance, model DTG 60, in a synthetic air atmosphere and with a heating rate of 10 °C min⁻¹.

To obtain the FeMnO₃ powder, a quantity of polymeric resin was constantly stirred and heated between 80 and 110 °C to evaporate excess water. The paste obtained was heated to 300 °C with a heating rate of 5 °C min⁻¹ and a plateau time of 120 min to eliminate organic matter and form FeMnO₃ agglomerates. Subsequently, the FeMnO₃ clusters were disaggregated in a crucible and sieved through a 270 mesh (53 μm) sieve. TG analysis of FeMnO₃ powder was carried out on a Shimadzu thermobalance, model DTG 60, in a synthetic air atmosphere (add flow rate) and with a heating rate of 10 °C min⁻¹.

2.4. Characterization of the FeMnO₃/ACC composite

The X-ray diffraction (XRD) patterns were collected, at room temperature, in a Bruker model D8 Advance ECO, diffractometer, using Cu Kα radiation (0.15406 nm), with instrument power of 40 kV and 25 mA and 2θ scan range from 5 to 90° with and speed of 4.6° min⁻¹. The

obtained patterns were compared to those data deposited at JCPDS (International Centre for Diffraction Data®). The morphology was analyzed using a VEGA3 TESCAN Scanning Electron Microscope (SEM) operating in the secondary electron mode. Chemical characterization was carried out using Energy Dispersive Spectroscopy (EDS) coupled to SEM. For the SEM experiments, ACC fragments with and without ferrite were fixed to aluminium supports using carbon tape.

2.5. Adsorption tests

Adsorption studies were carried out with the systems at room temperature. There was no need to centrifuge the aliquots before readings in the spectrophotometer, since the prepared composite used as adsorbent fulfilled its role of facilitating removal at the end of the process. Solutions of the malachite green (cationic) and the Congo red (anionic) dyes were used as adsorbates as received from Sigma-Aldrich Company. The concentration of the dyes was measured indirectly through spectrophotometric measurements on a T-80 UV/Vis Spectrophotometer equipment, using the λ_{\max} of each dye (malachite green 617 nm and Congo red 500 nm). The adsorption efficiency at each time was calculated from Equation 1.

$$\text{Adsorption Efficiency(\%)} = \frac{(C_i - C_f) \times 100}{C_i} \quad (1)$$

where: C_i = initial concentration and C_f = final concentration.

2.6. Ecotoxicological test

Toxicological tests were carried out by exposing the solutions arising from the adsorption processes against brine shrimp (*A. salina*). These tests were carried out following a protocol already used in other studies.^{10,19,22} Hence, an aqueous solution of sea salt (at 30 g L⁻¹) was prepared, filtered and added to a 2 liters prismatic rectangular container of glass. Subsequently, *A. salina* eggs were added in only one half of this recipient, which was kept protected from light for 48 h, whereas the opposite half was continuously irradiated by a LED lamp. After the eggs hatched, the *A. Salina* organisms migrated to the lit side, then a small portion of this solution containing 10 adult individuals was collected and transferred to a cylindrical glass vial (2 cm diameter). The volume was then adjusted to 5.0 mL by adding the aforementioned sea salt solution. Afterwards, 5.0 mL of a solution aliquot, collected from the adsorption experiments, were transferred for the vial. The vial was then left to stand under light for 48 h and the percentage of immobilized organisms was determined. Assays with each aliquot were performed in triplicate to estimate the toxicity of the solutions generated after the adsorption process.

3. Results and Discussion

3.1. Characterization of ferrite precursor polymeric resin

3.1.1. Viscosity measurements

After synthesis of the polymeric resin as previously described in subsection 2.1, its viscosity was empirically adjusted to improve adhesion and wettability on the ACC surface. Despite the qualitative adjustment, quantitative determination of viscosity was made using a Bookfield viscometer.

The ideal viscosity of 8.32x10⁻⁶ m² s⁻¹ was chosen after preliminary tests showed that higher viscosities made adhesion to ACC more difficult, while lower viscosities did not ensure complete surface coverage.

3.1.2. Thermogravimetric analysis

As stated in section 2.1, the manganese ferrite precursor polymer resin was prepared according to the method reported in the literature with some modifications. After synthesis of the polymeric resin, thermal analysis measurements were carried out for the resin with the aim of investigating the ideal temperature for its heat treatment, based on the temperature of complete decomposition of the organic phase, and the consequent formation of the inorganic phase (ferrite). Figure 1 shows the TG curve for the polymer resin sample up to 800 °C.

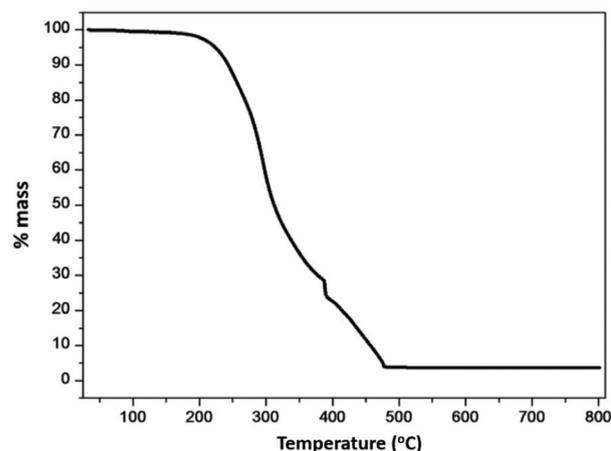


Figure 1. TG curve of the polymeric resin showing the percentage of mass loss of the organic phase during thermal decomposition

From Figure 1, it was observed that at approximately 200 °C the decomposition of the organic phase began, which was characterized by the loss of mass in the TG curve. The decomposition process of the organic phase was completed at 475 °C, beyond which no additional mass loss from the material was observed. At this temperature, the complete decomposition of the organic phase occurred, leading to the subsequent formation of the inorganic phase (ferrite). Based on the results of the thermal analysis, it was decided to thermally treat the composite at 600 °C, in order to



Figure 2. ACC fragment, ACC fragment with ferrite precursor resin and $\text{FeMnO}_3/\text{ACC}$ composite (from left to right)

guarantee the total decomposition of the organic phase, and achieve greater crystallinity in the resulting inorganic phase. Although TG provides information on the mass loss associated with decomposition, the improvement in crystallinity was confirmed by X-ray diffraction (XRD), which indicated the formation of the FeMnO_3 phase.

3.2. Preparation of the $\text{FeMnO}_3/\text{ACC}$ composite

Figure 2 shows images of the fragmented ACC on the left-hand side, the fragmented ACC impregnated with the ferrite precursor resin in the center, and the composite $\text{FeMnO}_3/\text{ACC}$ after the synthesis process on the right-hand side. The difference in colour between the two fragments was notable; furthermore, it was also observed that the synthesis resulted in a complete coating of the ACC surface.

3.3. Composite characterization

3.3.1. X-ray diffraction (XRD)

After defining the ideal heat treatment temperature necessary to obtain the Ferrite/ACC composite material, the support (ACC) containing the polymeric resin underwent heat treatment at 600 °C for 180 minutes in an ambient atmosphere. The formation of the phase of interest was confirmed by XRD analysis. Figure 3 shows the XRD pattern for the FeMnO_3 sample synthesized by the Pechini method. The XRD pattern revealed only the presence of characteristic peaks of FeMnO_3 particles with a cubic structure and space group Ia-3. The phase was indexed according to the crystallographic form ICDD 76–76.

3.3.2. Scanning electron microscopy and Energy Dispersive Spectroscopy (SEM/EDS)

Fragments of the ACC and $\text{FeMnO}_3/\text{ACC}$ materials were subjected to scanning electron microscopy analysis with the aim of obtaining information about the morphology of the samples before and after the deposition of ferrite on the ACC surface.

Figure 4 shows the micrographs obtained by SEM for the materials. The micrographs obtained for the ACC are

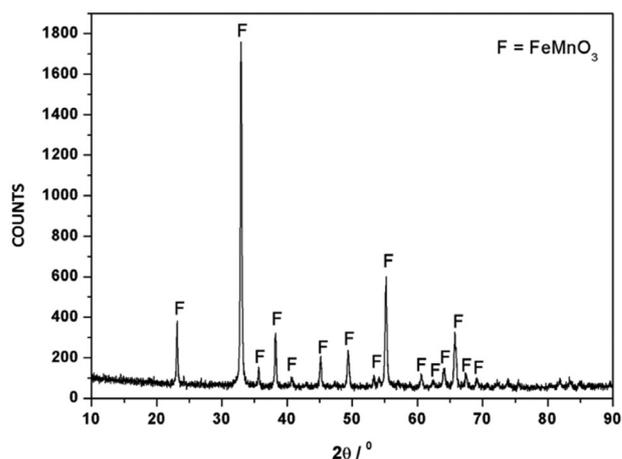


Figure 3. XRD pattern obtained for FeMnO_3 powder synthesized by the Pechini method

presented in A and B, where the presence of pores of varying sizes and the absence of deposited material on the ACC surface can be observed. In images C and D, the surface of the ACC can be seen with deposited Ferrite. Cracks can be observed, and have been highlighted by yellow arrows. This highlights a very different morphology of the ACC before the deposition of ferrite on its surface. Furthermore, these cracks were probably formed after heat treatment of the sample, which decomposed the polymer resin and led to the formation of the inorganic phase.

Figure 5a shows the SEM image of the composite, while Figures 5b and 5c depict the EDS map of the sample. The results reveal a strong interaction between the matrix (ACC) and the surface-adhered FeMnO_3 . It is noteworthy that the EDS maps identify the presence of calcium, manganese, and iron, which are the main constituents of ferrite. This result provides evidence of composite formation, *i.e.*, the map clearly shows the presence of FeMnO_3 on the surface of ACC, confirming that the material used to treat the aqueous system is indeed a composite formed through the interaction between ACC and FeMnO_3 .

Finally, the chemical map revealed the highest presence of Fe and Mn precisely in the area where manganese ferrite was deposited. Comparing iron content in the region with ferrite deposition to the iron content at the surface of ACC,

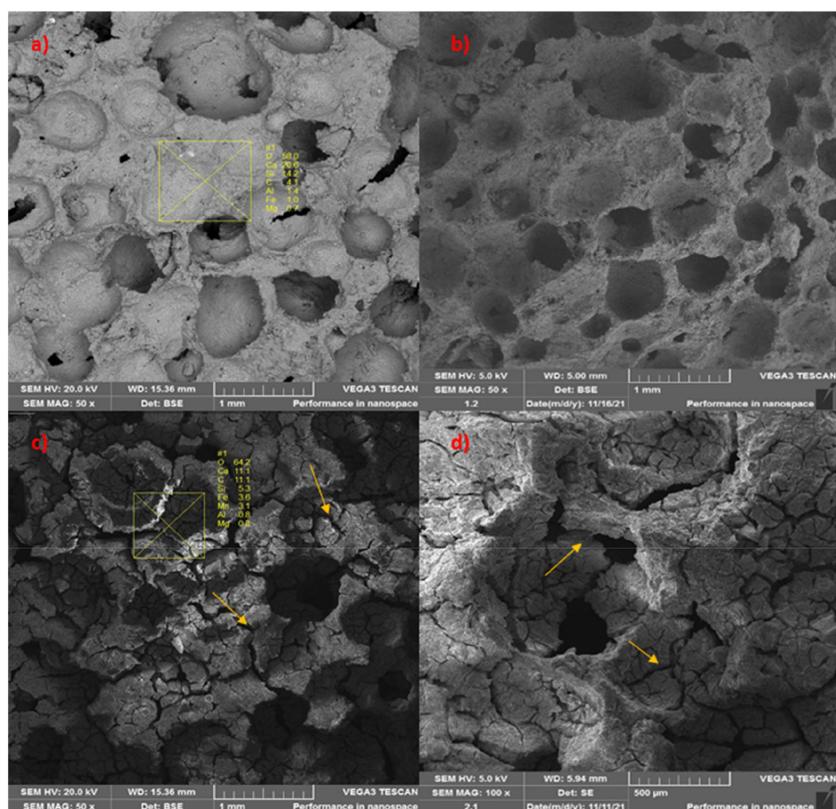


Figure 4. SEM images of the materials: (a) and (b) ACC before impregnation; (c) and (d) ACC after impregnation with FeMnO_3

the amount of iron was approximately 70% higher in the former.

3.4. Study of removal of toxic dyes from aqueous media

The prepared composite was tested for its adsorption capacity using Congo red (anionic) and malachite green (cationic) dye solutions. In all cases, the adsorption efficiency of the composite was compared to that of ACC without ferrite deposition.

These dyes were chosen as model molecules because they are formed by relatively stable structures and exhibit toxicity to various organisms, including mutagenic and carcinogenic effects.^{10,23,24} In addition, one molecule is

cationic (malachite green) and the other anionic (Congo red), which helps to investigate the versatility of the composite in removing positively or negatively charged molecules. It is worth noting that the study was conducted at a pH close to neutrality, seeking to investigate the potential of the composite under these conditions, without the need to add more reagents to the medium, which would naturally make the process more expensive. The structure of the two dyes is shown below (Figure 6).

3.4.1. Congo red dye adsorption

In the Congo red test, aliquot withdrawal times were set at 0, 24, 48, and 72 hours. Figure 7 presents the results of experiments conducted with ACC and the FeMnO_3 /

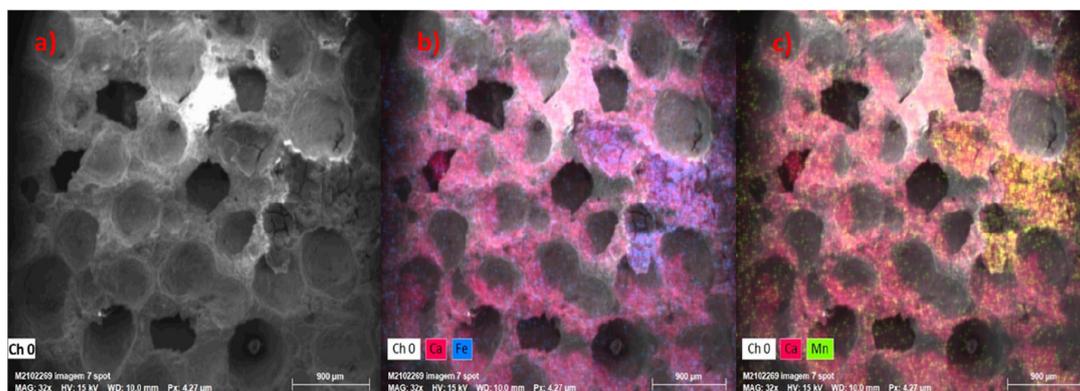


Figure 5. SEM and EDS maps of the FeMnO_3 /ACC composite

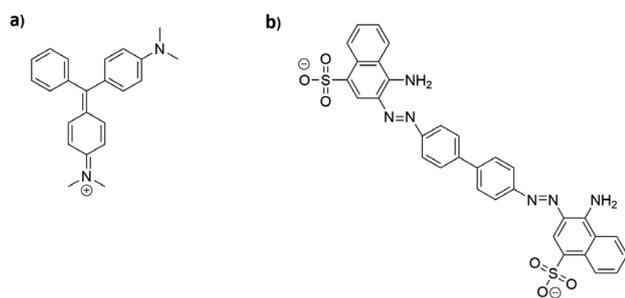


Figure 6. Structure of target molecules: (a) malachite green and (b) congo red

ACC composite. The findings demonstrate that Congo red dye removal was significantly more effective when the composite material was used, with enhanced efficacy observed at all time points. The removal efficiency calculated after 72 hours, based on Equation 1, showed that the $\text{FeMnO}_3/\text{ACC}$ composite achieved approximately 99% efficiency, while ACC alone reached around 63%. Thus, the presence of ferrite on the surface of ACC increased its adsorption capacity for Congo red by approximately 33%. Additionally, it is noteworthy that the magnetic properties of the composite facilitate easier separation of the dye from the adsorption system compared to ACC without ferrite.

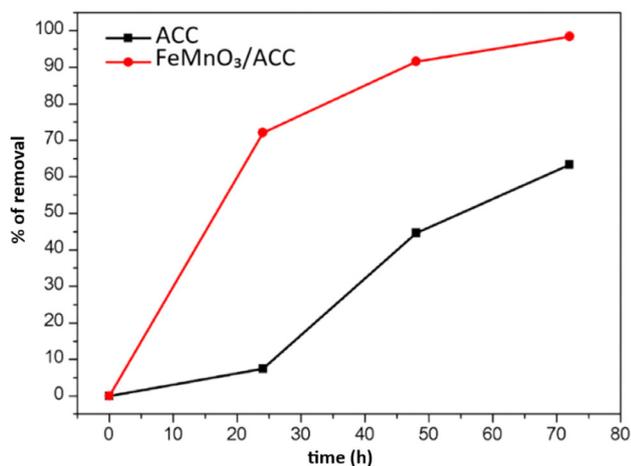


Figure 7. Congo red adsorption efficiency (%) in ACC and $\text{FeMnO}_3/\text{ACC}$ composite solutions

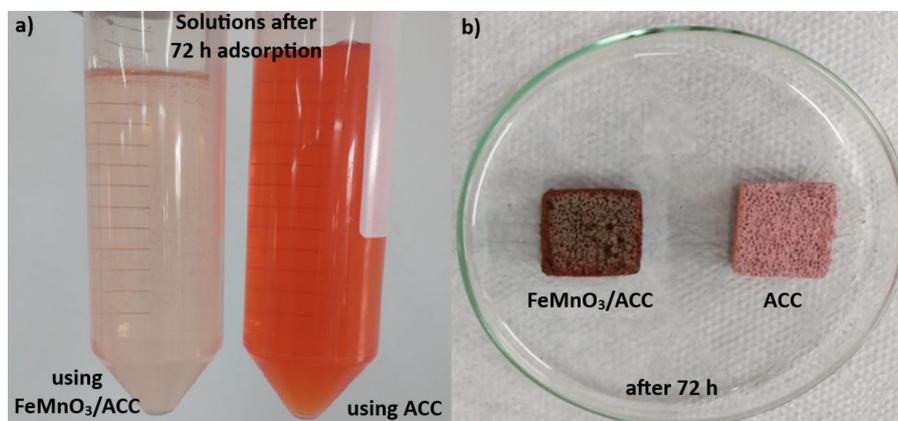


Figure 8. Visual comparison of the solutions (a) and materials (b) at the end of the 72-hour adsorption

Figure 8 illustrates the final appearance of the solutions and materials after the 72-hour experimental period. In Figure 8a, the solution on the left was treated with the $\text{FeMnO}_3/\text{ACC}$ composite, while the solution on the right received treatment with ACC alone. A visibly greater colour removal is observed in the composite-treated solution, indicating superior adsorption performance. Figure 8b presents the post-adsorption appearance of the materials ($\text{FeMnO}_3/\text{ACC}$ composite and ACC), as labelled in the figure.

3.4.2. Adsorption of malachite green dye

In the adsorption test carried out with malachite green dye, the aliquot withdrawal times were: 0, 4, 8 and 24 hours. In Figure 9, the results of the test with the prepared composite and ACC are presented. The results indicate that at the end of the experiment (24 hours), the solution treated with the composite is nearly free from dye, whereas a more intense colour is observed in the solution treated with ACC (Figure 10). Furthermore, within just 4 hours of experiment, the composite demonstrated an impressive adsorption capacity, adsorbing approximately 78% of the dye in the solution. In comparison, ACC adsorbed approximately 58% of the contaminant during the same time interval. Thus, the composite presented an adsorption capacity approximately 20% greater than that of ACC in the first 4 hours of the experiment.

In Figure 10a, the malachite green solutions can be seen after 24 hours of experiment. On the left side, it is possible to visualize the solution in which the composite was applied and on the right side, the one treated only with ACC. Just as occurred with Congo red, a much greater difference in color contrast is noted in the solution where the composite was applied, presenting a paler color. Figure 10b shows an image of the materials (ACC and composite) after the adsorption process.

It is noteworthy that the kinetic of dye removal increased for all types of dyes studied. In other words, regardless of the dye present in the solution, the composite exhibited a faster removal of the contaminant compared to ACC (see Figures 7 and 9). This characteristic holds paramount importance in reactor design and process dimensioning.

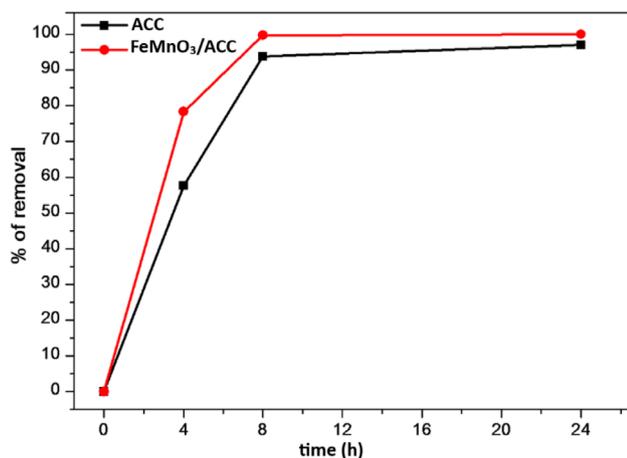


Figure 9. Malachite green adsorption efficiency (%) in ACC and FeMnO₃/ACC composite solutions

In addition, at the end of the adsorption processes, the composite was able to remove approximately equal percentages of both dyes. This indicates that the adsorption processes should not be greatly influenced by the dye charge, and should be governed by other interactions, such as π -type interactions. It is likely that they are primarily responsible for the adsorption of both dyes. In addition, malachite green was adsorbed at a higher rate than Congo red. This may be related to steric factors, since the malachite green molecule is considerably less voluminous than the Congo red one.

It is important to mention that the dyes used in the present study as target molecules are relatively well investigated due to their respective toxicities. In the literature, there are several studies with materials developed for removal by adsorption of these dyes.²⁵⁻³⁰

In most of these studies, the materials are in powder form, which can generate additional experimental procedures for the removal of the adsorbent from the medium. The material in powder form usually needs to be filtered or centrifuged to be removed from the solution. In this context, the material prepared in the present work does not have this problem,

since the adsorbent is in the form of magnetic fragments that are easily removed.

It is also important to highlight the simplicity of the synthesis of the FeMnO₃/ACC composite, and the fact that it is an inorganic material that does not present toxicity. There are studies in the literature that prepare materials that present excellent adsorption percentages for the dyes investigated in the present work, however, the synthesis is relatively more expensive,²⁷ or they are adsorbents prepared with polymeric material, whose synthesis requires the use of more aggressive reagents than those used in the present work.²⁹

3.5. Ecotoxicity tests

Ecotoxicity tests were carried out in order to investigate the toxicity of the initial solutions (Congo red 50 mg L⁻¹, and malachite green 50 mg L⁻¹), against the toxicity of the solutions after the adsorption process by the composite.

As can be seen in Figure 11, solutions with an initial concentration of Congo red equal to 50 mg L⁻¹ showed significant toxicity for *Artemia*, and eliminated approximately 40% of the exposed population, whereas after the adsorption process with the composite (FeMnO₃/ACC) this percentage dropped to 20%. Malachite green showed relatively greater toxicity than Congo red for brine shrimp. The original solution (50 mg L⁻¹) eliminated approximately 70% of individuals exposed to this dye. The solution after the adsorption process with the composite prepared in the present work, however, reduced this percentage to 40%. Therefore, it is evident that the application of the material as an adsorbent, in addition to improving an organoleptic property (colour), reduces the toxicity of the solutions, due to the decrease in their concentration in solution.

4. Conclusion

The present work described the synthesis and

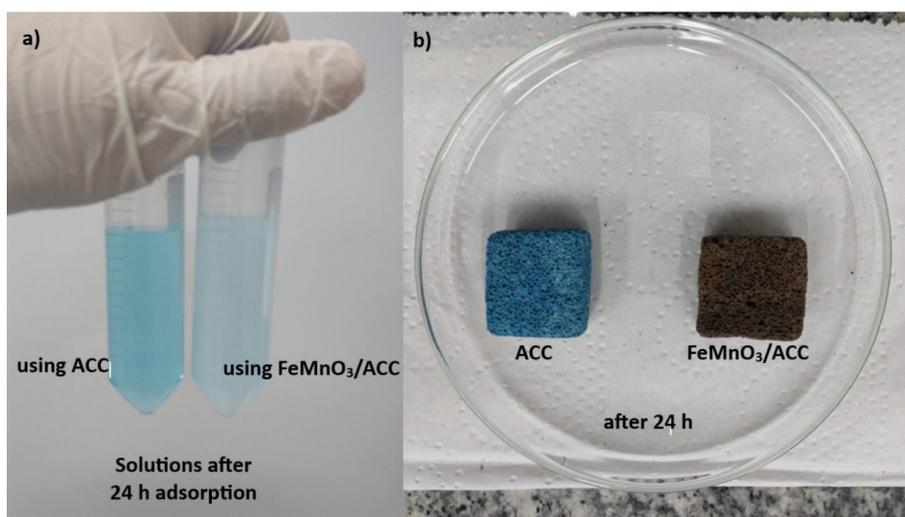


Figure 10. Visual comparison of the solutions (a) and materials (b) at the end of the 24-hour adsorption

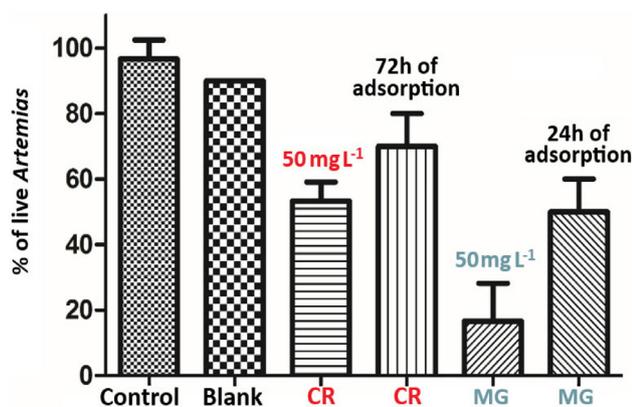


Figure 11. Ecotoxicity test with brine shrimp, comparing solutions before and after dye adsorption by the composite FeMnO₃/ACC

characterization of a composite consisting of MnFeO₃/ACC. The composite was applied as an adsorbent for model organic contaminants (anionic and cationic dyes).

The magnetic phase of the composite was synthesized using polymeric precursors. Thermal analysis of the precursor resin revealed that the optimal heat treatment temperature for the formation of the desired phase was approximately 600°C. The effectiveness of the thermal treatment was verified by XRD characterization of the ferrite, where it was possible to identify the characteristic diffraction peaks of the desired phase.

The surface of the materials (composite and ACC) were analysed by SEM and EDS. The constituents derived from FeMnO₃ were identified in the sample at locations where deposition occurred. The deposited regions of the sample exhibited approximately 72% more iron than the areas where FeMnO₃ was not deposited. Furthermore, in the regions of the sample where resin deposition occurred, large cracks formed along the composite, further confirming the formation of the aforementioned phase. Moreover, in regions with high iron concentration, there is also a high concentration of manganese, which corroborates the results of the formation of the phases of interest.

The removal tests for the target molecules under study yielded highly satisfactory results. For tests carried out with Congo red dye, the composite showed an adsorption efficiency of approximately 99% after 72 hours of experiment. For malachite green dye, a removal efficiency of 78% was found after 24 hours of experiment. It is worth noting that, in addition to the composite having a higher removal percentage than ACC, it was also easier to extract from the adsorption system than ACC. Therefore, the results obtained in the study indicate that the composite holds significant potential for application in the treatment of effluents containing organic contaminants.

Regarding the toxicity tests with Artemias, it can be concluded that the application of the composite substantially decreased the toxicity of the solutions post the adsorption process. This signifies a noteworthy improvement in water quality. For Congo red dye, there was an approximate 50% reduction in the toxicity percentage, while for malachite

green dye, which exhibited higher toxicity, an approximately 57% reduction was observed. This indicates that the composite effectively contributes to mitigating the toxicity caused by these organic contaminants in aqueous systems.

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