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**Characterization of natural zeolites to be used in combination with iron oxide nanoparticles for the remediation of contaminated water**

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**RESUMO (Times New Roman, tam 12)**

RESUMO - Magnetite nanoparticles (Fe₃O₄) and a magnetite-zeolite composite were synthesized with the aim of developing efficient adsorbent materials for water treatment. The materials were characterized by X-ray diffraction (XRD) to identify the crystalline phases, and by dynamic light scattering (DLS) to determine the particle size distribution. Adsorption studies were conducted using potassium dichromate (K₂Cr₂O₇) as a model contaminant to evaluate the capacity of the materials to remove hexavalent chromium (Cr(VI)) from aqueous solution. The results demonstrated the successful formation of magnetite and its integration with the zeolite matrix. The composite showed enhanced adsorption performance compared to pure magnetite, indicating synergistic effects between the two phases. These findings suggest that the magnetite-zeolite system is a promising candidate for the remediation of Cr(VI)-contaminated water.

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*Palavras-chave: inserir aqui de 3 a 5 palavras-chave separadas por vírgula (Times New Roman, tam. 10, itálico)*

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**Introdução**



Magnetite nanoparticles (Fe₃O₄) have gained significant attention due to their unique magnetic properties, high surface area, and potential applications in environmental remediation, particularly in the removal of toxic contaminants from aqueous media. Among various strategies, the combination of magnetite with porous materials such as zeolites has shown promising results, enhancing both the stability and adsorption capacity of the composite materials.

In this work, magnetite nanoparticles and a magnetite-zeolite composite were synthesized and characterized in terms of particle size and crystalline phases. The materials were further evaluated for their adsorption performance towards potassium dichromate (K₂Cr₂O₇), a model contaminant commonly used to simulate chromium(VI) pollution. The results contribute to the understanding of the interaction mechanisms between the adsorbent materials and dichromate ions, aiming at the development of efficient and low-cost alternatives for water purification.

**Experimental**

*Synthesis of Magnetite Nanoparticles and the Magnetite–Zeolite Composite.*

The synthesis of magnetite nanoparticles was based on the method described by Andrade et al. (1), strictly following the established procedures. Initially, a solution containing 1 mol/L of Na₂SO₃ and 2 mol/L of FeCl₃ was prepared. After homogenization, diluted NH₄OH was slowly added, promoting the formation of a black precipitate. The resulting material was centrifuged and washed with distilled water. This sample was designated as "MAG".

For the synthesis of the magnetite–zeolite composite, the same procedure used for the synthesis of magnetite was followed. However, after the addition of diluted NH₄OH and the formation of the black precipitate, a measured amount of zeolite was added to the reaction mixture. The resulting composite was then centrifuged and washed with distilled water. This sample was designated as "MAG-ZEO".

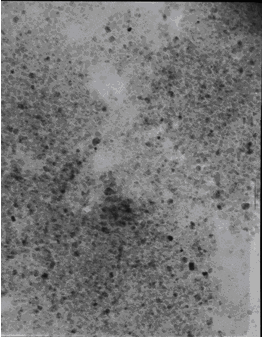
*Characterization*

The particle size of the MAG sample was determined by transmission electron microscopy (TEM), while the size distributions of the zeolite and MAG-ZEO nanocomposite samples were assessed via dynamic light scattering (DLS) using a Zetasizer. The crystalline phases of iron oxide in both magnetite and the composite, as well as the zeolite phase, were identified through Rietveld refinement of X-ray diffraction (XRD) data.

Initial adsorption experiments were carried out using 10 mg of the materials as adsorbents in an aqueous solution containing 50 ppm of potassium dichromate (K₂Cr₂O₇). The suspension was kept in contact for predetermined time intervals. After each period, the suspension was centrifuged, and the supernatant was analyzed by UV-Vis spectrophotometry.

**Results and Discussion**

The TEM micrograph of the MAG sample (Figure 1) reveals particle agglomeration and an average particle size around 9 nm.



**50 nm**

**a**

**Figure 1.** Transmission electron microscopy (TEM) micrograph of the MAG sample

The Rietveld refinement of the natural mordenite structure was performed using the Cmc2₁ space group and pseudo-Voigt function, while the refinement of the magnetite nanoparticles was conducted using the Fd3̅m space group (Fig. 1).

The quality of the Rietveld refinement of the magnetite was evaluated from the profile factor (*Rp*), weighted profile factor (*Rwp*), goodness of fit indicator (*S*), Bragg Factor (*RB*), and crystallographic RF-factor (*RF*) parameters. The refinement yielded *Rp* = 6.08, *Rwp* = 8.01, S = 1.04, *RB* = 2.12, and *RF* = 1.92, indicating good quality of refinement.



**Figure 2.** Powder X-ray diffraction patterns (Black) of the sample MAG. The solid (Red) lines correspond to the profiles fitted through

Rietveld refinement.



The refinement of the zeolite yielded *Rp* = 4.29, *Rwp* = 5.74, *S* = 2.19, *RB* = 2.59, and *RF* = 2.16, indicating good quality of refinement.

The zeolite and MAG-ZEO samples presented comparable particle size distributions, with an average particle diameter of approximately 50 µm. In contrast, the magnetite nanoparticles exhibited a significantly smaller average size of 9 nm, as determined by transmission electron microscopy (TEM).

Preliminary results indicate that the zeolite does not adsorb K₂Cr₂O₇, whereas both the MAG sample and the MAG-ZEO composite exhibit adsorption capacity.



**Conclusion**

The results demonstrate the successful synthesis of magnetite nanoparticles and the formation of a magnetite–zeolite composite through the combination of both materials. Preliminary studies highlight the promising potential of the magnetite–zeolite composite as an effective agent for environmental remediation. Future work will focus on optimizing the composite’s synthesis parameters, evaluating its adsorption capacity under various environmental conditions, and investigating its reusability and stability to establish its practical applicability in treating contaminated water.

**Agradecimentos**

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**References**

1. Andrade, A.L.; Fabris, J.D.; Pereira, M.C.; Domingues, R.Z.; Ardisson, J.D. “Preparation of composite with silica-coated nanoparticles of iron oxide spinels for applications based on magnetically induced hyperthermia”. *Hyperfine Interact,* **2013,** 218, 71-82.