



# Article Magnetic Adsorbent Based on Faujasite Zeolite Decorated with Magnesium Ferrite Nanoparticles for Metal Ion Removal

Mariana Rodrigues Meirelles <sup>1,2</sup>, João Otávio Donizette Malafatti <sup>1,3,\*</sup>, Márcia Tsuyama Escote <sup>4</sup>, Alexandre Henrique Pinto <sup>5</sup> and Elaine Cristina Paris <sup>1,\*</sup>

- <sup>1</sup> National Nanotechnology Laboratory for Agriculture (LNNA), Embrapa Instrumentação, XV de Novembro St., 1452, São Carlos 13560-970, SP, Brazil
- Institute of Chemistry, University of São Paulo, Av. Trab. São Carlense, 400, São Carlos 13566-590, SP, Brazil
  Dopartment of Chemistry, Fodoral University of São Carlos. Rod. Washington Luís. Km 235
- <sup>3</sup> Department of Chemistry, Federal University of São Carlos, Rod. Washington Luís, Km 235, São Carlos 13565-905, SP, Brazil
- <sup>4</sup> Center for Engineering, Modeling and Applied Social Sciences (CECS), Federal University of ABC (UFABC), Avenida dos Estados, 5001, Santo André 09210-170, SP, Brazil
- <sup>5</sup> Department of Chemistry and Biochemistry, Manhattan College, 4513, Riverdale, New York, NY 10471, USA
- \* Correspondence: jmalafatti@hotmail.com (J.O.D.M.); elaine.paris@embrapa.br (E.C.P.); Tel.: +55-16-2107-2868 (E.C.P.)

Abstract: Magnetic nanoparticles are a promising alternative as a support in adsorption processes, aiming at the easy recovery of the aqueous medium. A faujasite zeolite (FAU) surface was decorated with magnesium ferrite (MgFe<sub>2</sub>O<sub>4</sub>) nanoparticles. FAU is a porous adsorbent with high specific surface area (SSA) and chemical stability. The FAU:MgFe<sub>2</sub>O<sub>4</sub> nanocomposite 3:1 ratio (w w<sup>-1</sup>) promotes the combination of the surface and magnetic properties. The results showed the effectiveness of the MgFe<sub>2</sub>O<sub>4</sub> immobilization on the FAU surface, exhibiting a high SSA of 400 m<sup>2</sup> g<sup>-1</sup>. The saturation magnetization (Ms) was verified as 5.9 emu g<sup>-1</sup> for MgFe<sub>2</sub>O<sub>4</sub> and 0.47 emu g<sup>-1</sup> for FAU:MgFe<sub>2</sub>O<sub>4</sub>, an environmentally friendly system with soft magnetic characteristics. The magnetic nanocomposite achieved high adsorption values of around 94% removal for Co<sup>2+</sup> and Mn<sup>2+</sup> ions. Regarding its reuse, the nanocomposite preserved adsorption activity of above 65% until the third cycle. Thus, the FAU:MgFe<sub>2</sub>O<sub>4</sub> nanocomposite presented favorable adsorptive, magnetic, and recovery properties for reuse cycles in polluted water.

Keywords: magnetic nanocomposite; magnesium ferrite; faujasite zeolite; adsorption; metal ions

# 1. Introduction

In recent decades, environmental pollution has become a worldwide challenge for global society due to the various adverse effects caused in different ecosystems [1]. Environmental degradation creates constraints on natural resources, negatively impacting the conditions of life on Earth [2,3]. Inorganic pollutants have been found frequently in water supplies [4]. Typical contaminants found in wastewater are toxic metals, such as iron (Fe), nickel (Ni), lead (Pb), cobalt (Co), manganese (Mn), and cadmium (Cd), among others [5]. In high concentrations, these cations can cause irreversible damage to the body's functioning, sometimes leading to death [6].

In this scenario, decontaminating/recovering polluted aquatic environments, minimizing waste production, and avoiding inadequate disposal are essential points for conserving this vital resource [7]. There are several processes for removing toxic metal ions in aqueous media, such as coagulation-flocculation [8], complexation [9], chemical precipitation [10], electrochemical treatment [11], and adsorption [12]. The adsorption process is one of the most effective methods for this application due to its high efficiency, low cost, and capacity to use in many cycles. However, factors such as adsorbate surface area, pore size distribution, chemical nature, temperature, and concentration of the components can interfere



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**Copyright:** © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). with the adsorption process [13]. Additionally, exploring the intrinsic structure materials elucidates the improved properties of nanoparticles [14].

Zeolites stand out as a promising adsorbent due to high surface area values, regularity in pore size distribution, selectivity, and ion exchange capacity [15,16]. Zeolites are hydrated aluminosilicates with a structure accommodating sodium, potassium, and calcium ions. The tetrahedrons [SiO<sub>4</sub>] and [AlO<sub>4</sub>]<sup>-</sup> form the zeolite crystal lattice [17]. The high surface area comes from the microporous structures formed due to the union of tetrahedrons. The tetrahedra grouping can occur in different ways, giving rise to different types of zeolites [18]. Faujasite (FAU) group zeolites have interesting properties for the adsorption process, such as high pore volume, empty intercrystalline space, and several acidic sites on the structure's outer surface [19,20]. The FAU zeolites present a cubic symmetry structure with pores arranged perpendicularly with a diameter of 7.4 Å [21]. The different faujasite structures depend on the SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> molar ratio. A smaller Si/Al ratio supplies a more significant negative charge on the system, balanced by cations in non-structural positions. Thus, cation exchange processes are an essential property of zeolites [22].

One of the significant challenges of using the adsorbent systems is adequate immobilization to present efficient removal and reuse, maintaining efficacy in many cycles [23,24]. Magnetic adsorbents are an alternative that promote the easy adsorption of pollutants with practical posterior separation from effluents using a magnetic field [25–29]. Ferrites are double oxides of iron and another metal of the general formula Mfe<sub>2</sub>O<sub>4</sub>, where M is a bivalent metal element (usually, Mn<sup>2+</sup>, Fe<sup>2+</sup>, Zn<sup>2+</sup>, Co<sup>2+</sup>, Ni<sup>2+</sup>, Cu<sup>2+</sup>, or Mg<sup>2+</sup>), generally in a spinel-type structure [1,30]. Ferrites can be synthesized for different applications, such as sol-gel [31], coprecipitation [32], combustion [33], hydrothermal [34], and microwave [35], by controlling the diameter, phase structure, and morphology, as well as the magnetic properties. The synergism between magnetic and electronic properties allows the possibility of ferrite application in batteries [36], sensors [37], solar cells [38], adsorption [39], and catalysis [24].

Magnesium ferrite (MgFe<sub>2</sub>O<sub>4</sub>) is attractive due to its magnetic behavior and high specific surface area (SSA) in a non-toxicity material, maintaining desirable properties and environmentally friendly aspects. In the MgFe<sub>2</sub>O<sub>4</sub> spinel structure, the Fe<sup>3+</sup> ions are homogeneously distributed between the octahedral and tetrahedral sites [40]. Therefore, the MgFe<sub>2</sub>O<sub>4</sub> magnetic point occurs due to uncompensated iron spins distributed in the system [41]. Nonkumwong and colleagues [42] synthesized MgFe<sub>2</sub>O<sub>4</sub>-Au nanoparticles and studied the in vitro cytotoxicity response. The nanocomposites obtained did not exhibit cytotoxicity in the test performed on mammalian cells in vitro. In this sense, the MgFe<sub>2</sub>O<sub>4</sub> compound presents magnetic behavior, promoting the removal after application with a magnetic field, permitting the system recovery and reuse in an environmentally friendly way.

Magnetic zeolites can potentially remove toxic ions from contaminated water. Peng and coworkers [43] obtained magnetic FAU from molybdenum and iron II/III oxides ore. The material showed high efficiency in removing cadmium (204.2 mg g<sup>-1</sup>) ions in wastewater. The authors also found that ion exchange is the magnetic zeolite's primary mechanism of cadmium adsorption. In a study using naturally activated zeolites and Fe<sub>3</sub>O<sub>4</sub>, the authors evaluated the removal of chromium ions from contaminated water [44]. The results indicated a compound with high efficiency and competitiveness in removing chromium ions (84–99%). Mthombeni and colleagues [45] evaluated the remotion of chromium ions using a magnetic natural zeolite–polymer composite, finding that the system could remove up to 99.99% of the contaminant at an ideal pH. In this way, the magnetic nanocomposite based on zeolites with ferrites shows promising results in polluted water environmental remediation.

To our knowledge, the better elucidation concerning the adsorption of toxic metal ions using the FAU zeolite decorated with magnesium ferrite nanoparticles is essential for environmental remediation science. The proposed system (FAU:MgFe<sub>2</sub>O<sub>4</sub>) was not found in the current literature, since magnesium ferrite is an alternative as a low-toxicity compound composed of Fe and Mg ions, both nutrients for the soil. Thus, the present work aimed to obtain a porous magnetic nanocomposite from immobilizing magnesium ferrite nanoparticles on the FAU zeolite surface to perform the adsorption and reuse of toxic metal ions, such as  $Co^{2+}$  and  $Mn^{2+}$ .

## 2. Materials and Methods

# 2.1. Synthesis

## 2.1.1. FAU Zeolite Synthesis

Faujasite zeolite (FAU) was obtained by the sol-gel process followed by hydrothermal treatment adapted from Romero [19]. The reagents used were Silica (Tixosil<sup>®</sup> 333, donated by Evonik Degussa, Essen, Germany), the mineralizing NaOH (Synth), and Sodium Aluminate (Sigma Aldrich, St. Louis, MI, USA), as sources of Si, Na, and Al, respectively. First, a sodium aluminate solution was prepared in 50 mL of deionized water. Next, 50 mL of a NaOH solution (2.7 M) was added under magnetic stirring at room temperature. After that, the colloidal silica was progressively added until a gel formed. The viscous gel remained in static, aging for 24 h on a bench at room temperature. Then, the gel underwent hydrothermal treatment at 100 °C for 2 h. Finally, the material was centrifuged at 10,000 rpm to neutralize the solution and was dried in a circulating air oven at 60 °C for 24 h.

#### 2.1.2. Magnesium Ferrite Synthesis

Magnesium ferrite was adapted from the method by Omer et al. [46]. The starting reagents were FeCl<sub>3</sub>·6H<sub>2</sub>O (Synth) and MgCl<sub>2</sub>·6H<sub>2</sub>O (Synth). First, each salt precursor was solubilized in 25 mL water, resulting in a solution of 0.4 mol L<sup>-1</sup>. After the solubilization, the solutions were mixed to complete homogenization. Then, the 25 mL NaOH solution (3 mol L<sup>-1</sup>) was kept under constant stirring. The resulting suspension was washed and centrifuged at 8000 rpm for 10 min. Finally, the resulting material was dried in a circulating air oven. Ultimately, the ferrite calcination was carried out in an EDG 3000 muffle furnace. The thermal treatment was performed using heating to 300 °C for 4 h (controlled organic elimination), with subsequent treatment at 500 °C for 2 h to obtain magnetic ferrite.

#### 2.1.3. FAU:Ferrite Nanocomposite Synthesis—FAU:MgFe<sub>2</sub>O<sub>4</sub>(3:1)

The anchoring of MgFe<sub>2</sub>O<sub>4</sub> nanoparticles on the faujasite zeolite (FAU) surface was based on Yamaura [47]. Initially, FAU was suspended in deionized water and mixed with MgFe<sub>2</sub>O<sub>4</sub> at a 3:1 (w w<sup>-1</sup>) ratio of the FAU:MgFe<sub>2</sub>O<sub>4</sub> nanocompound. Next, the process was completed using an ultrasonic tip at a 20% amplitude for 30 min in an ice bath. After complete dispersion and homogenization, the obtained composite was centrifuged at 8000 rpm for 5 min and then dried in an oven at 60 °C for 12 h. Finally, the nanocomposite was calcinated in the conditions to obtain the magnetic MgFe<sub>2</sub>O<sub>4</sub> described in Section 2.1.2.

#### 2.2. Characterization Techniques

The structural analysis of the compounds was performed through X-ray diffraction. The equipment used was Shimadzu XRD-6000, operating with Cu-K $\alpha$  radiation,  $\lambda$  1.5406 Å at 1 °C min<sup>-1</sup> from 20 5° to 80°. The images were obtained via scanning electron microscopy (SEM) equipment, JEOL<sup>®</sup> model 6701F, using at 5 kV. The samples were also analyzed through energy dispersive spectroscopy (EDS) at 132 eV resolution in a model 6742<sup>a</sup> Ultradry Silicon Drift Detector. For specific surface area analysis, N<sub>2</sub> physisorption/desorption was performed using the Brunauer, Emmett, and Teller (BET) method. In addition, the magnetic properties of magnesium ferrites (MgFe<sub>2</sub>O<sub>4</sub>) and the nanocomposite were evaluated through magnetic curves via an optical magnetizer (Model PPMS 9 Evercool—Quantum Design) at room temperature (300 K) and with a magnetic field between –20,000 to 20,000 Oe.

#### 2.3. Adsorption Experiments

For the kinetic assays, the conditions of  $1.0 \text{ g L}^{-1}$  for FAU:MgFe<sub>2</sub>O<sub>4</sub> (3:1) and 10 mg L<sup>-1</sup> for each ion (Co<sup>2+</sup> and Mn<sup>2+</sup>) were established. Likewise, the composite was previously weighed, and the ion solution was added mechanically. Then, the adsorption tests were carried out at 15, 30, 45, 60, 120, 180, 240, 300, 360, 480, and 960 min. Afterward, the material was centrifuged, and 2 mL aliquot was used for atomic absorption spectrometry analysis. Finally, the adsorbed percentage amount of each ion at different times was determined.

The percentage of removal for each ion was calculated through the following equation:

$$Removal (\%) = \frac{c_i - c_f}{c_i} \times 100 \tag{1}$$

The adsorption capacity of the adsorbent for each ion as a function of time, qt (mg g<sup>-1</sup>), was determined according to the following equation:

$$qt = \frac{Ci - Ct}{m}V \tag{2}$$

where *Removal* % (mg L<sup>-1</sup>) is the amount (percentage adsorbed by each ion), *Ci* is the initial ions concentration (mg L<sup>-1</sup>),  $c_f$  is the the final concentration of the ions still present in the solution, and *Ct* (mg L<sup>-1</sup>) is the concentration of ions remaining in the solution at time t (h). The experiments were conducted at pH 7 and room temperature (25 °C).

The kinetic models of pseudo-first and pseudo-second order were evaluated. The equations of the parameters of these models are presented below:

Pseudo-First Order kinetic model: 
$$qt = qe(1 - e^{-k_1 t})$$
 (3)

where *qe* is the equilibrium constant (mg  $g^{-1}$ ) and  $k_1$  is the rate constant (min<sup>-1</sup>).

Pseudo-Second Order kinetic model: 
$$qt = \frac{t}{\left(\frac{1}{k_2 q_e^2}\right) + \frac{t}{q_e}}$$
 (4)

where  $k_2$  is the constant rate (g mg<sup>-1</sup> min<sup>-1</sup>).

For the reuse test, the conditions of 1.0 g  $L^{-1}$  of FAU: MgFe<sub>2</sub>O<sub>4</sub> and 0.01 g  $L^{-1}$  for each ion (Co<sup>2+</sup>, Mn<sup>2+</sup>) were fixed. The tests were performed in triplicate. Initially, aliquots of the aqueous solutions were added into flasks containing the previously weighed nanocomposites, as described for the adsorption assays. The flasks remained in mechanical agitation for 24 h and were then recovered. Then, the aliquots of the supernatant solution were removed and atomic absorption spectrometry analysis was performed. The centrifugation was carried out in the laboratory to minimize equipment damage due to the presence of particles.

Next, a fresh 0.01 g  $L^{-1}$  solution of the cations was added to the same vial containing the adsorbent. After 24 h, the flask was separated and an aliquot was taken for analysis. This reuse process was conducted in three cycles. Then, the adsorbed percentage amount of individual ions was obtained by Equation (1).

#### 2.4. Magnetical Recovery

The mass recovery was evaluated to verify the nanocomposite capacity to be attracted by a magnetical field. First, FAU:MgFe<sub>2</sub>O<sub>4</sub> (0.1 g) was inserted in a water flask. After stabilization in ambient temperature for 30 min, a neodymium magnet was utilized for separation from the aqueous medium. Next, the material was washed with ethanol alcohol and dried at 80 °C for mass determination on an analytical scale. Thereafter, the system (FAU:MgFe<sub>2</sub>O<sub>4</sub>) was resuspended in water, and the process was repeated five times.

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# 3. Results

The structural properties of the individual synthesized compounds and the FAU:MgFe<sub>2</sub>O<sub>4</sub> nanocomposite were evaluated. The XRD patterns are illustrated in Figure 1 to assess the synthesized materials' phase formation and structure.



**Figure 1.** X-ray diffraction analyses for FAU, MgFe<sub>2</sub>O<sub>4</sub>, and FAU:MgFe<sub>2</sub>O<sub>4</sub> (3:1) on the left and the high magnification on the right.

The synthesized zeolite showed characteristic peaks of the faujasite phase, according to the JCPDS data sheet 043-0168. For the synthesized MgFe<sub>2</sub>O<sub>4</sub>, all peaks correspond to the JCPDS number 71–1232. No additional peaks were observed. The results obtained by XRD evidenced the crystallinity and purity of the precursors for the nanocomposite production. In the FAU:MgFe<sub>2</sub>O<sub>4</sub> (3:1) diffractogram, the FAU phase maintenance can be observed. Regarding the MgFe<sub>2</sub>O<sub>4</sub> reflections, the FAU peak superposition makes it difficult to verify whether the ferrite phase was retained. However, the red highlights and the graph magnification (Figure 1 right) confirm the presence of the most intense MgFe<sub>2</sub>O<sub>4</sub> peak in the composite. Thus, the presence of FAU and MgFe<sub>2</sub>O<sub>4</sub> in the obtained nanocomposite is evident. Similar research on magnetic nanocomposites has shown the synthesis of MgFe<sub>2</sub>O<sub>4</sub> nanocomposite without any structural change in the support. Hosseini et al. [48] synthesized the nanocomposites of graphene oxide and MgFe<sub>2</sub>O<sub>4</sub> and observed the phase maintenance of the precursors.

The FAU, MgFe<sub>2</sub>O<sub>4</sub>, and FAU:MgFe<sub>2</sub>O<sub>4</sub> (3:1) SEM images are shown in Figure 2. FAU has an average size of 0.5 to 1  $\mu$ m. Ferrite magnetic exhibits agglomerated nanoparticles with a diameter inferior to 50 nm. There is also a high agglomeration degree in MgFe<sub>2</sub>O<sub>4</sub>, usually reported for superparamagnetic compounds, due to the dipolar interactions between the magnetic phases of the nanoparticles [49]. As observed in the FAU:MgFe<sub>2</sub>O<sub>4</sub> (3:1) image, the FAU appears with smaller particles on its surface, which can be attributed to the presence of MgFe<sub>2</sub>O<sub>4</sub>.



Figure 2. SEM images of FAU, MgFe<sub>2</sub>O<sub>4</sub>, and FAU:MgFe<sub>2</sub>O<sub>4</sub> (3:1).

 $MgFe_2O_4$  distribution on the FAU surface elements for the nanocomposite was mapped using energy-dispersive X-rays via an SEM microscope (SEM-EDS). In Figure 3, the distribution and overlapping of Si and Al refer to the FAU elements. Additionally, Fe and Mg comprise the  $MgFe_2O_4$ . It can be seen that the ferrite is distributed in an agglomerated form in the zeolite surface regions. Thus, the SEM-EDS images confirm that the magnetic nanocomposite can be obtained by impregnating the magnetic nanoparticles in the FAU zeolite surface. These results, associated with the X-ray diffractogram (Figure 1), show that the production of the magnetic nanocomposite was successful.



Figure 3. Images of SEM-EDS mapping for FAU:MgFe<sub>2</sub>O<sub>4</sub> (3:1), showing element distributions.

The values of the micropore volume ( $V_{Micro}$ ) and total (SSA<sub>BET</sub>) and external surface (S<sub>Ext</sub>) areas for the materials are shown in Table 1. A decrease in S<sub>BET</sub> from 650 m<sup>2</sup> g<sup>-1</sup> (FAU) to 400 m<sup>2</sup> g<sup>-1</sup> in the FAU:MgFe<sub>2</sub>O<sub>4</sub> nanocomposite can be noted.  $V_{Micro}$  is verified as having a minimal reduction, from 0.30 cm<sup>3</sup> g<sup>-1</sup> to 0.20 cm<sup>3</sup> g<sup>-1</sup>, in the nanocomposite. The values are coherent given the desired material composition, since the nanocomposite was prepared at a ratio of 3:1 FAU:MgFe<sub>2</sub>O<sub>4</sub> (w w<sup>-1</sup>), preserving surface properties from the zeolite. This result can be attributed to the agglomeration effect caused by the magnetic nanoparticles. Magnetic particles promote spin orientation that occasions attraction and agglomeration [50].

**Table 1.** N<sub>2</sub> physisorption analysis through the BET mathematical model of FAU, MgFe<sub>2</sub>O<sub>4</sub>, and FAU:MgFe<sub>2</sub>O<sub>4</sub> samples.

Sample	$S_{Ext}$ (m <sup>2</sup> g <sup>-1</sup> )	$SSA_{BET}$ (m <sup>2</sup> g <sup>-1</sup> )	$V_{Micro}$ (cm <sup>3</sup> g <sup>-1</sup> )
FAU	390	650	0.30
MgFe <sub>2</sub> O <sub>4</sub>	14	19	0.051
FAU:MgFe <sub>2</sub> O <sub>4</sub>	34	400	0.20

Magnetic curves were analyzed in order to understand the MgFe<sub>2</sub>O<sub>4</sub> magnetic behavior (Figure 4). The results presented in Figure 4 show that MgFe<sub>2</sub>O<sub>4</sub> presents 5.90 emu  $g^{-1}$ for saturation magnetization (Ms) and 0.10 emu  $g^{-1}$  for remaining magnetization (Mr). A similar result was obtained by Sheykhan et al. [51] for MgFe<sub>2</sub>O<sub>4</sub> synthesized via the coprecipitation method and calcined at 550 °C for 6 h. The authors observed a Ms value of 6.00 emu  $g^{-1}$  and Mr of 0.0008 emu  $g^{-1}$ . Figure 4a shows the typical behavior slope for materials with soft magnetic nature, as widely reported in the literature [52-55]. The narrow shape of the curves also demonstrates that the material can be readily demagnetized, affirming its superparamagnetic nature [56]. When analyzing the FAU:MgFe<sub>2</sub>O<sub>4</sub> (3:1) nanocomposite's magnetic capacity, a decrease in Ms from 5.90 to 0.47 emu  $g^{-1}$  and Mr from 0.10 to 0.01 emu  $g^{-1}$  (Figure 4b) is observed. Paris et al. [57] prepared Nb<sub>2</sub>O<sub>5</sub>:MgFe<sub>2</sub>O<sub>4</sub> magnetic photocatalysts, obtaining a Ms of 3 emu  $g^{-1}$ . This study showed a decrease in magnetization compared to pure magnetic material due to the content of magnetic nanoparticles in the composite. Su et al. [58] studied the ZnO:MgFe<sub>2</sub>O<sub>4</sub> (1:1) composite. A decrease in Ms from 40 to 20 emu  $g^{-1}$  for the ferrite in the nanocomposite form was observed. The reduction in the nanocomposite magnetic effect concerning isolated ferrite corresponded to

the ratio used, which was equivalent to 25%. This result corresponds to the present work, since FAU zeolite in the nanocomposite consists of a non-magnetic component.

As shown in Figure 4c, the developed system can be removed after adsorption with a magnet at the laboratory scale or projected via the appliance in natural environmental conditions, according to the applied magnetic field. In addition, it is worth mentioning that the main advantage of resorting to  $MgFe_2O_4$  is its low toxicity, especially when compared to other ferrites, such as ferrites that contain cobalt [59–61], since Fe and Mg ions are soil nutrients.



**Figure 4.** Magnetic curves: (a)  $MgFe_2O_4$ ; (b) FAU:MgFe\_2O\_4; and (c) image of soft magnetic  $MgFe_2O_4$  ferrite.

Concerning the nanocomposites, as observed in Figure 4, in terms of magnetic behavior, in the evaluated FAU:MgFe<sub>2</sub>O<sub>4</sub> 3:1 (w w<sup>-1</sup>) proportion, the weak properties and the low values contributed to the difficulty of complete separation in an aqueous medium. As seen in Figure 5a, the nanocomposite is attracted to the magnetic in dry form (Figure 5a (left)) and an aqueous medium (Figure 5a (right)). However, the nanocomposite in a 3:1 ratio (w w<sup>-1</sup>) does not allow the complete attraction of particles (turbidity), keeping it partially separated in suspension. The nanocomposite magnetic capacity was evaluated via mass recovery through five cycles using a neodymium magnet (Figure 5b). At the end of the first cycle, the weight decreased from 0.1 g to  $0.039 \pm 0.004$  g, corresponding to around 60% recovery. It is noticed that the recovery percentage after the 1st cycle remained very similar until the fifth cycle. In the present system, FAU:MgFe<sub>2</sub>O<sub>4</sub> at a ratio of 3:1 (w w<sup>-1</sup>) would be necessary to increase the proportion of magnesium ferrite in the nanocomposite for a better magnetical response. However, recovering approximately 60% of the adsorbent in the evaluated conditions is possible.

The adsorption assays of  $Mn^{2+}$  and  $Co^{2+}$  ions were carried out in an aqueous medium at 10, 50, and 100 mg L<sup>-1</sup>, using the nanocomposite FAU:MgFe<sub>2</sub>O<sub>4</sub> at different dosages (0.25, 0.5, and 1.0 g L<sup>-1</sup>) for 24 h. Figure 6 shows the graphs of the adsorption test results.



**Figure 5.** FAU:MgFe<sub>2</sub>O<sub>4</sub> (3:1) magnetic attraction: (**a**) dried (left) and aqueous medium (right); (**b**) mass recovery through neodymium magnetic.



**Figure 6.** Adsorption assays using FAU:MgFe<sub>2</sub>O<sub>4</sub> (0.25, 0,5, and 1.0 g L<sup>-1</sup> dosages) over 24 h for Co<sup>2+</sup> and Mn<sup>2+</sup> ions concentrations: (a) 100 mg L<sup>-1</sup>; (b) 50 mg L<sup>-1</sup>; (c) 10 mg L<sup>-1</sup>.

Concerning Figure 6, the adsorption test results showed that for a contaminant concentration of 10 mg  $L^{-1}$ , the highest adsorption percentages are 94.5% and 94.9% for the Co<sup>2+</sup> and Mn<sup>2+</sup> ions, respectively, using a nanocomposite dosage of 1.0 g  $L^{-1}$ .

Two interesting trends were observed in Figure 6. First, the removal percentage increased with increasing adsorbent dosage for a particular contaminant ion concentration. This behavior is due to the increase in the adsorbent dosage increasing the number of adsorption sites. The second trend is that the removal percentage increased with the decreasing contaminant ion concentrations. This can be explained by the fact that, for a fixed adsorbent dosage, there is a certain number of available adsorption sites in the FAU:MgFe<sub>2</sub>O<sub>4</sub>. So, for more diluted solutions, it is possible to adsorb as many contaminant ions as possible without filling up all the available adsorption sites. Then, as the contaminant ions concentration increases, more contaminant ions will be present on the same number of adsorption sites, leading to a lower removal percentage [62].

Paris et al. [50] evaluated the  $Pb^{2+}$  ion removal with a nanocomposite of faujasite zeolite and magnesium/cobalt ferrite, also at a 3:1 ratio (w w<sup>-1</sup>). The authors stated that the nanocomposite removed 99% of  $Pb^{2+}$  in an aqueous medium, demonstrating high efficiency.

The kinetics study provided information on the adsorption capacity in an aqueous medium as a function of time. Therefore, the best removal results of the adsorption assays were used. The conditions for the kinetic evaluation were 10 mg  $L^{-1}$  of the contaminant in contact with 1.0 g  $L^{-1}$  of FAU:MgFe<sub>2</sub>O<sub>4</sub>. Figure 7 presents graphs depicting the kinetic assay with varying adsorption times.



**Figure 7.** Kinetic assays: the adsorption of  $Co^{2+}$  and  $Mn^{2+}$  ions using 1 g L<sup>-1</sup> of the FAU:MgFe<sub>2</sub>O<sub>4</sub> nanocomposite versus time.

Under the evaluated conditions, the adsorption/desorption equilibrium is reached in the first 15 min (0.25 h) of contact between the adsorbent (FAU:MgFe<sub>2</sub>O<sub>4</sub>) and the adsorbate (metallic ions). For  $Mn^{2+}$  and  $Co^{2+}$ , respectively, approximately 95% and 94% of these contaminants were removed, remaining constant until the end of the test.

The kinetic model's pseudo-first order and pseudo-second order results were evaluated. Figure 8 and Table 2 represent the fits of the models considered.

The results of the kinetic evaluation show that the removal of ions is high in the initial stages of adsorption and reaches equilibrium quickly. According to Reference [22], it is known that the adsorption process occurs preferentially on the surface of the FAU via ion exchange. The R<sup>2</sup> parameter showed that pseudo-first and pseudo-second order models adjusted for the adsorption kinetics in these assays are comparable, meaning that either model could satisfactorily explain the kinetic data of this situation. The parameters determined by the models are 3.88 and 3.58 mg g<sup>-1</sup> for Co<sup>2+</sup> and Mn<sup>2+</sup>, respectively. Liu et al. [63] synthesized zeolite Y and studied the adsorption kinetics of toxic metal ions, such as Co<sup>2+</sup> and Ni<sup>2+</sup>. The authors performed nonlinear kinetic adjustments and verified that



both kinetic models, pseudo-first and n-th order reactions, adequately described the kinetic adsorption behavior.

**Figure 8.** Pseudo-first and -second order models for  $\text{Co}^{2+}$  (**a**) and  $\text{Mn}^{2+}$  (**b**) adsorption. Concentrations: 10 mg L<sup>-1</sup> of metallic ions and a 1.0 g L<sup>-1</sup> dosage of FAU:MgFe<sub>2</sub>O<sub>4</sub>.

**Table 2.** The parameters of nonlinear kinetic models considering pseudo-first and pseudo-second order values for  $Co^{2+}$  (a) and  $Mn^{2+}$  ions (b). Concentrations: 10 mg L<sup>-1</sup> of metallic ions and a 1.0 g L<sup>-1</sup> dosage of FAU:MgFe<sub>2</sub>O<sub>4</sub>.

Model	Parameter	Value	
		Co (a)	Mn (b)
Pseudo-First Order	$\frac{R^2}{k_1 \text{ (min}^{-1})}$	0.99941 $7.89 \times 10^{3}$	$0.99998 \\ 8.80 \times 10^2$
	qe	3.85	3.54
Pseudo-Second Order	$ \begin{array}{c} R^2 \\ k_2 \ (\text{g mg}^{-1} \ \text{min}^{-1}) \\ qe \end{array} $	$\begin{array}{c} 0.99941 \\ 1.02 \times 10^{20} \\ 3.85 \end{array}$	$0.99997 \\ 1.08  imes 10^{19} \\ 3.54$

Finally, the reusability of the nanocomposite FAU:MgFe<sub>2</sub>O<sub>4</sub> was evaluated. In this reuse test, the material was separated via centrifugation after 24 h of contact with contaminating ions. Three reuse cycles were performed, and Figure 9 shows the behavior seen in the tests.



Figure 9. Reuse assays of the  $Co^{2+}$  and  $Mn^{2+}$  ions (10 mg  $L^{-1}$ ) for 24 h with FAU:MgFe<sub>2</sub>O<sub>4</sub> (1 g  $L^{-1}$ ).

The material proved to be efficient in the reuse tests. In the first cycle, the FAU:MgFe<sub>2</sub>O<sub>4</sub> removed 91.3% of Co<sup>2+</sup> and 85.3% of Mn<sup>2+</sup>. The adsorption percentage decreases with reuse, as expected. In the third cycle, the nanocomposite removed 73.5% and 68.8% of Co<sup>2+</sup> and Mn<sup>2+</sup> ions, respectively. This decrease can be attributed to saturation ions on the zeolite surface, minimizing active sites. The results indicate that the nanocomposite can be reused as an adsorbent.

Furthermore, zeolites present stability mainly when subjected to hydrothermal treatments [64], as in this work. The hydrothermal treatment used for synthesis causes chemical changes that ensure zeolite structure stability at different pHs and temperatures [65]. Thus, the structural maintenance of zeolites makes it possible for this material to be reused. Therefore, the magnetic nanocomposite is able to treat water contaminated by toxic metal ions in routine activities. After waste treatment, a magnet is able to attract the adsorbent and separate it from the aqueous medium. Additionally, from an analytical perspective [66,67], the adsorbent enables ion availability, facilitating ion analyses.

#### 4. Conclusions

The FAU:MgFe<sub>2</sub>O<sub>4</sub> (3:1) nanocomposite was obtained, maintaining the integrity of the precursors, faujasite zeolite and magnesium ferrite. The material demonstrated soft superparamagnetic characteristics, possibly able to remove the nanocomposite after adsorption. In the adsorption tests with 1.0 g L<sup>-1</sup> of FAU:MgFe<sub>2</sub>O<sub>4</sub> and 10 mg L<sup>-1</sup> of contaminant concentration, the nanocomposite showed the efficient removal of the ions, with 94.5% for Co<sup>2+</sup> and 94.9% for Mn<sup>2+</sup>. The FAU:MgFe<sub>2</sub>O<sub>4</sub> nanocomposite displayed reuse capacity, supplying adsorption values of higher than 65% in the third cycle for both ions. The mass magnetical recovery evaluation demonstrated a 60% capacity until the fifth cycle. In this way, the FAU:MgFe<sub>2</sub>O<sub>4</sub> nanocomposite is a promising alternative for removing metallic ions in polluted water, promoting recovery and reuse.

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