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CUTTING-EDGE CHITOSAN MAGNETITE NANOCOMPOSITE IN TREATING INDIGENOUS SURFACE WATER

Anca Andreea ȘĂULEAN[1], Ecaterina MATEI[1], Claudia Ionela TARCEA[1, *], Andra Mihaela PREDESCU[1], Cristian PREDESCU[1], Andrei-Constantin BERBECARU[1], Ionuț Cosmin CHIVA[1], Ciprian MANEA[1], Maria POPANTON[2], Grigore VLAD[2], Dumitru VÂJU[2]

The objective of the study was to prepare, characterize, and test a chitosan-magnetite (CS-Fe_3O_4) nanocomposite as an efficient adsorbent used in the removal of iron ions and decrease in turbidity from a real water sample. The CS-Fe_3O_4 was characterized using FTIR spectrometry, OM, SEM, and XRD analyses, and the physical and chemical properties of the water samples were determined using standardized methods. The CS-Fe_3O_4 presented a good adsorption capacity for Fe ions and a decreased turbidity after 24 hours. One advantage of this research is the high efficiency of the nanocomposite and the easy possibility of recuperating, regenerating, and reusing the material in future research.

Keywords: magnetite, chitosan, heavy metals, adsorption, turbidity

1. Introduction

Today, water treatment has become one of the most important topics worldwide. Population growth and industrialization mainly affect the contamination of water (surface water as well as groundwater) and the environment in general [1]. One significant issue is the rising levels of heavy metals in water, posing a threat to water quality and public health due to their toxicity, persistence, and ability to accumulate in biological systems [2].

To address this problem, studies have focused on the use of natural polymers [3], like alginate [4], cellulose [5], starch [6], and chitosan [7]. Chitosan is a polysaccharide derived from chitin, which is obtained by treating shrimp shells and other crustaceans with sodium hydroxide. When placed in water, chitosan behaves as a polymer, effectively capturing heavy metals and other dissolved substances from the water [8]. To retain these dissolved metals, chitosan-coated magnetite can be used, and once the pollutants adhere to the

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[1] National University of Science and Technology POLITEHNICA of Bucharest, 313 Splaiul Independentei, 060042, Bucharest, Romania,
[2] ICPE Bistrita, 7 Parcului Street, 420035 Bistrita, Romania.
* Corresponding author: claudia.tarcea@upb.ro

chitosan surface, they can be easily extracted from the water using magnets and then reused [9].

So far, significant research efforts have focused on the removal of heavy metals from contaminated water using the adsorption process [4,10,11]. In theory, this adsorption method offers flexibility in design and operation and often results in high-quality treated effluent. Various types of adsorbent materials have been employed in different heavy metal removal applications, including activated carbon [12], carbon nanotubes, polymer-based adsorbents [2,13,14], metal oxides [15], and bio-adsorbents. Among these adsorbents, iron-based magnetic nanoparticles stand out due to their unique attributes, such as a high surface area-to-volume ratio, minimal chemical consumption, and the absence of secondary pollutants.

This composite is expected to improve the efficiency and speed of pollutant removal from wastewater by utilizing an external magnetic field to enhance separation and purification processes. The primary objectives of the study are to prepare, characterize, and test this environmentally friendly nanocomposite, described as spherical particles with a magnetic core and a biodegradable polymeric shell. The aim is to develop an effective and sustainable solution for removing heavy metal contaminants from wastewater, contributing to both environmental protection and public health.

2. Materials and methods

Reagents p.a. were purchased from Sigma-Aldrich, which provided the sodium hydroxide (NaOH), ferrous sulfate heptahydrate (Fe^{2+}), ferric chloride hexahydrate (Fe^{3+}), acetic acid, ethanol, and chitosan (with a deacetylation level of 75–85%) needed in the synthesis of magnetized coagulants. Deionized water (ELGA WATERLAB, PURELAB Option-Q water deionizer (High Wycombe, UK)) was used for the preparation of the stock solutions. ICPE Bistrița provided a real water sample from the Nerău region in Timiș County, Romania.

Material preparation

To prepare the CS and CS-Fe_3O_4 materials the ionic gelation method was applied. Initially, a 1% (w/v) chitosan solution was prepared in 1% acetic acid using a magnetic stirrer at 80°C and 400 rpm, until the CS completely dissolved. To this solution, 1 g Fe_3O_4 nanomaterial was added under ultrasound bath stirring to ensure a better dispersion. Fe_3O_4 nanomaterial as a single material was obtained according to Matei et al. [16]. After 30 minutes of dispersion, the mixture was reticulated with 25% glutaraldehyde solution forming cross-linked spheres that were filtered, washed several times with distilled water, and then dried naturally for 48 hours. The final product was CS-Fe_3O_4 nanocomposite at a mass ratio of

1:1. From the previous work [17], in order to prevent Fe_3O_4 dissolution and its magnetic properties, a stable mixture with CS offers mechanical resistance and an easy way to be collected and avoid the aging of Fe_3O_4.

Fig. 1 shows the modified schematic diagram of synthesizing the CS-Fe_3O_4 nanocomposite magnetized coagulant.

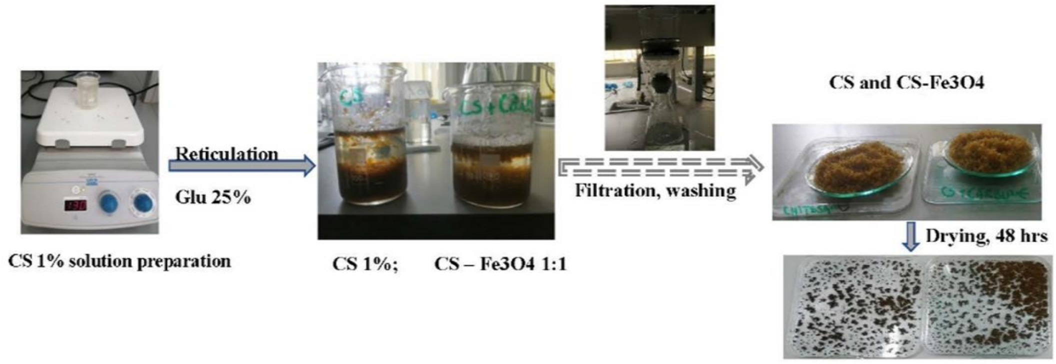

Fig. 1. Modified schematic diagram of synthesizing the CS-Fe_3O_4 nanocomposite preparation

Material characterization

The structure and morphology of the CS and CS-Fe_3O_4 were investigated with Optical Microscopy (OM) (OLYMPUS BX51 M microscope, Tokyo, Japan) and Scanning electron microscopy (SEM) coupled with energy-dispersive spectra EDS (QUANTA 450 FEG microscope, Eindhoven, The Netherlands).

Real water sample characterization

Real surface water samples were obtained from the Nerău area, Banat, Timiș County, Romania. The physical and chemical parameters of the surface water were determined using standard methods [18]. Turbidity was measured using a Lovibond portable laboratory turbidimeter model TB 210 and was expressed in nephelometric turbidity units (NTU). Samples were filtered through a Whatman filter paper having a pore size of 0.45 mm. Further, the heavy metals concentration was determined using SR 8662-2:1996 and SR EN 1233:2003, and for Fe total concentration SR ISO 6332-96 was used. Also, other parameters such as chlorides (Cl^-), hardness (D_T), and suspended solids (as TDS) were investigated according to adequate standards and analytical methods.

Coagulation procedure

The jar test is the most widely applied method for evaluating and optimizing the coagulation–flocculation process based on standard methods [19]. Coagulation tests were carried out using a standard jar test apparatus (Phipps and Bird, Model 300). The standard procedure involved 1 min of rapid mixing (120 rpm) followed by 10 min of slow mixing (45 rpm) for flocculation. Then, the treated water was allowed to settle for 15 min, and the supernatant sample was withdrawn by a syringe from approximately 2 cm below the liquid level for analysis. The initial pH of the surface water was adjusted to a desired value with 1 M NaOH or 1 M HCl. The same coagulation test was conducted with no coagulant as a blank. The impact of initial turbidity concentration (NTU), coagulant quantity, and some heavy metals concentration as variable parameters was evaluated as different conditions in this research. All the experiments were conducted in triplicate (the accuracy is considered to be 84%) to ensure the reproducibility of the results; the mean of these three measurements is presented herein. The turbidity removal efficiency (TE) was calculated using Eq. (1) as follows:

$$TE\ \% = \frac{(T_0 - T)}{T_0} * 100 \qquad (1)$$

where T_0 and T represent the initial and final turbidities (NTU) of water, respectively.

3. Results and Discussions

CS-Fe$_3$O$_4$ characterization

The structural and morphological properties of the CS-Fe$_3$O$_4$ nanocomposite were investigated by means of OM and SEM analyses. The microscopies of the nanocomposite are presented in Fig. 3 ((a) - OM and (b) - SEM) and for comparison, the microscopies for CS sample are presented in Fig. 2.

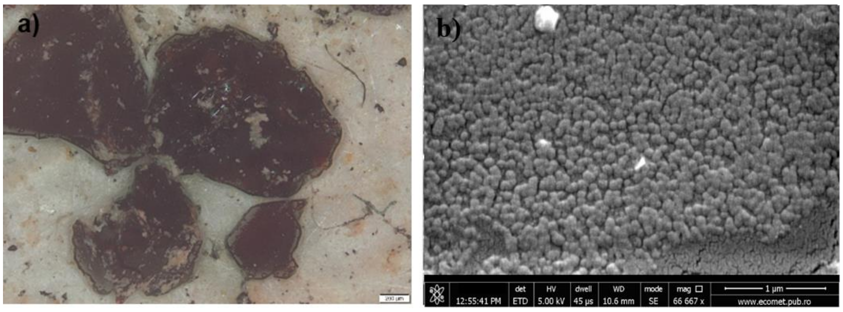

Fig. 2. Optical microscopy (a) and SEM microscopy for CS sample

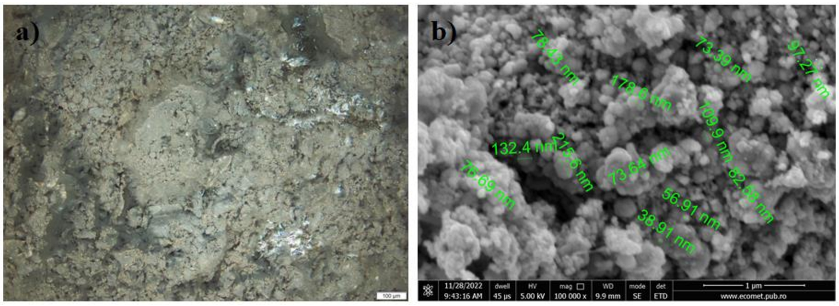

Fig. 3. Optical microscopy (a) and SEM microscopy for CS-Fe_3O_4 nanocomposite

The OM analyses revealed the presence of small black aggregates distributed uniformly across the chitosan surface, indicating the even dispersion of magnetite within the CS matrix. The SEM microscopy indicates the presence of magnetite nanoparticles in the form of aggregates embedded into the CS structure. From the OM analysis (Fig. 3 a)) it can be seen that for the CS-Fe_3O_4 nanocomposite, the magnetite is spread evenly throughout the CS mass as small black particles, with sizes in the range of 10-50 µm. From the SEM analysis (Fig. 3 b)) it was observed that with the addition of Fe_3O_4 on the surface of the CS, irregularities were formed, different aggregates, indicating the possibility of encapsulation of Fe_3O_4 in the chemical structure of the CS.

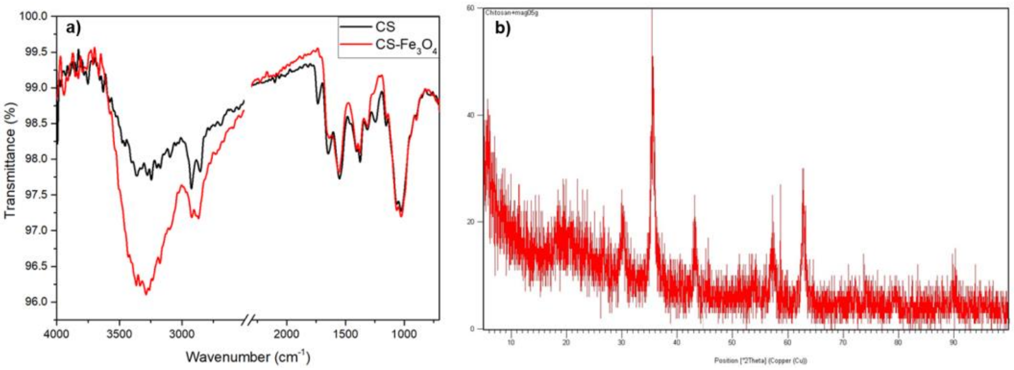

Fig. 4. FTIR (a) for CS and CS-Fe_3O_4 and XRD (b) for CS-Fe_3O_4 nanocomposite

Fig. 4 (a) presents the FTIR spectra of CS and CS-Fe_3O_4 samples. It can be observed that the cross-linked chitosan material presented a characteristic peak at 3276 cm^{-1} indicating the stretching vibrations of N-H groups [20], –OH groups, and inter-hydrogen bonds of polysaccharides [21]. The minor shift observed in the CS-Fe_3O_4 nanocomposite from 3276 cm^{-1} (CS) to 3265 cm^{-1} is attributed to the formation of weak bonds between Fe_3O_4 and –OH groups [10]. The band at 1551 cm^{-1} is attributed to the asymmetric stretching vibrations of the carbonyl group and stretching vibrations of C-O bounds from the pyranose ring at 1024 cm^{-1} [22]. In the spectrum of CS-Fe_3O_4, when compared with the CS spectrum, the 1550 cm^{-1} peak of N–H bending vibration shifted to 1560 cm^{-1}, and a new sharp peak at 1645 cm^{-1} appears, indicating that chitosan reacts with glutaraldehyde to form the Schiff base, which in CS-Fe_3O_4 material shifted at 1635 cm^{-1} [9].

The XRD pattern of the CS-Fe_3O_4 nanocomposite sample, which is presented in Fig. 4 b, presents six strong characteristic diffraction peaks at 2θ values of 30,16°; 35,53°; 43,12°; 53,6°; 57,1°; and 62,61° which correspond to (220), (311), (400), (422), (511), and (440) crystalline planes of Fe_3O_4 phase [6]. Chitosan does not offer strong diffraction peaks due to the amorphous nature of the biopolymer, but a peak at 2θ of 21,05° confirms the presence of cross-linked chitosan in the nanocomposite [23].

Real water sample characterization

The physical and chemical quality indicators were investigated for the real water samples and the average values were obtained from 3 replicates and are presented in Table 1.

Table 1

Analytical methods and standards for analyzed water samples.

Indicator, unit value	Standard	Analytical Method	Values for water sample	NTPA-013 (A2 category)
pH, unit pH	SR EN ISO 10523:2012	pH analysis	7.6	5,5 - 9
EC, µS/cm	-	Conductometry	1722	1000
TDS mg/L	SR EN 15216:2021	Conductometry	921	250
Cl^- , mg/L	SR ISO 9297:2001	Volumetry	14.2	200
D_T , °d	STAS 3326/76	Volumetry	5.17	-
Fe mg/L	SR ISO 6332-96	AAS	0.359	1
Mn mg/L	SR 8662-2:1996	AAS	0.188	1
Cr mg/L	SR EN 1233:2003	AAS	0.504	0,05
Turbidity, NTU	SR EN ISO 7027-1:2016	Turbidimetry	14.7	< 5

Adsorption of Fe ions and turbidity tests for CS-Fe_3O_4 nanocomposite

To determine the performance of CS-Fe_3O_4 and to improve the chemical retention performance of CS, 100 mL of real water sample, showing Fe content, was brought into contact with 0.1 and 1 g CS-Fe_3O_4 respectively, and sampled for analysis over 60 minutes and 24 hours. The Fe total concentration was analyzed with flame-atomic absorption spectrometry (FAAS). The results are shown in Table 4.

Table 4.

Evolution of Fe concentration in real water sample on contact with CS-Fe_3O_4

Time (min)	0.1 g CS-Fe_3O_4		1 g CS-Fe_3O_4	
	mgFe/L	Efficiency, %	mgFe/L	Efficiency, %
0	0.359	0	0.359	0
15	0.178	50.41	0.168	53.20
30	0.141	60.97	0.157	56.26

45	0.104	71.25	0.107	70.47
60	0.029	92	0.058	84
24 h	< 0,01	~100	0.043	88

Settling time represents an important parameter used for the coagulation and flocculation process efficiency. The evolution in time of the process influences the economic benefits of the coagulation process [24]. The analyzed time was between 30 minutes and 24 hours, after a settling period of about 30 minutes as observed in the settling tanks from different sewage treatment plants [25]. Fig. 5 (a) shows an evolution for Fe removal and Fig. 5 (b) shows the turbidity removal efficiency (NTU). Two nanocomposite quantities were chosen for the experiments (0.1 and 1 g) and were compared with 0.1g CS. The literature indicates the use of CS as a single solution, with various concentrations between 2 and 40 mg/L [25-28]. To increase the time of separation after the coagulation process, in this research, the CS was used as a solid support matrix with a magnetic core. Thus, different dosages of CS-Fe_3O_4 nanocomposite were used for turbidity and heavy metals removal.

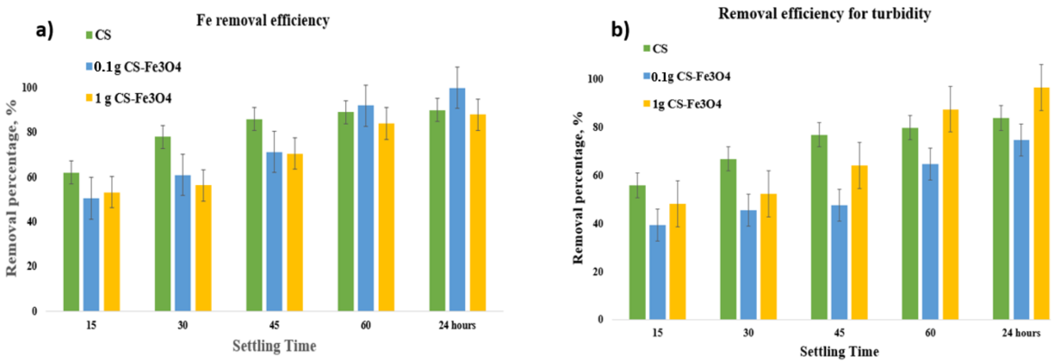

Fig. 5. Iron removal efficiency (a) and Turbidity removal efficiency for the CS and two different dosages of CS-Fe_3O_4

The use of 1 g CS-Fe_3O_4 in 100 mL treated water provides an efficiency of about 70% in 60 minutes, so for 1 m^3, 10 kg CS-Fe_3O_4 will be needed. At the same time, in the case of using 0.1 g, an efficiency of about 50% is ensured after the first 15 minutes, achieving saturation of the active centers of the eco-product, so that in this case 1 kg CS-Fe_3O_4/1m^3 water will be needed to achieve such an efficiency. The maximum removal efficiency of ~100% was obtained for 0.1 g of CS-Fe_3O_4 nanocomposite after 24 hours of contact time with the real water sample. When compared to simple CS, the magnetic nanocomposite presented better performances in the removal of Fe ions from the water sample.

The water showed a yellowish coloration before experiments were conducted, coloration which disappeared after about 24 hours of contact with both dosages of CS-Fe_3O_4 (<5 NTU), and the results are shown in Table 5. A higher efficiency of 87.68% is found when using 1 g CS-Fe_3O_4 to decrease the turbidity of the water sample.

Table 5

Evolution of NTU values for real water sample

Time (min)	0.1 g CS-Fe_3O_4		1 g CS-Fe_3O_4	
	NTU	Efficiency, %	NTU	Efficiency, %
0	14.7	0	14.7	0
15	8.90	39.45	7.60	48.29
30	8.00	45.57	6.98	52.51
60	7.68	47.75	5.24	64.35
24 h	3.68	74.96	1.81	87.68

4. Conclusions

A chitosan magnetite nanocomposite was obtained by ion gelation method from magnetite and chitosan and used in the purification process of real water samples. Real surface water samples were provided from the Nerău area, Banat, Timiș County, Romania, and the physical and chemical parameters were determined using standard methods The structural, morphological, and stability properties were investigated for the CS-Fe_3O_4 nanocomposite by means of OM, SEM, XRD, and FTIR analyses. These OM and SEM investigations revealed that the magnetite nanoparticles are homogenously dispersed throughout the CS matrix. The XRD analysis presented the characteristic peaks associated with crystalline planes of Fe_3O_4 phase. Specific peaks for stretching and bending vibration of –OH, N-H, and other groups were observed from the FTIR spectrum, suggesting the successful impregnation of magnetite in the chitosan matrix.

The material was further tested for the retention of Fe ions and a decrease in turbidity from the real water sample. When using 0.1 g of nanocomposite the retention efficiencies reached values of almost 100% after 24h contact. Future investigations will be conducted for recuperation, regeneration, and reutilization of the chitosan magnetite nanocomposite, kinetic, and adsorption studies.

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