



Application of Magnetized Cacti Biomass as Flocculants in Water Treatment

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Abstract

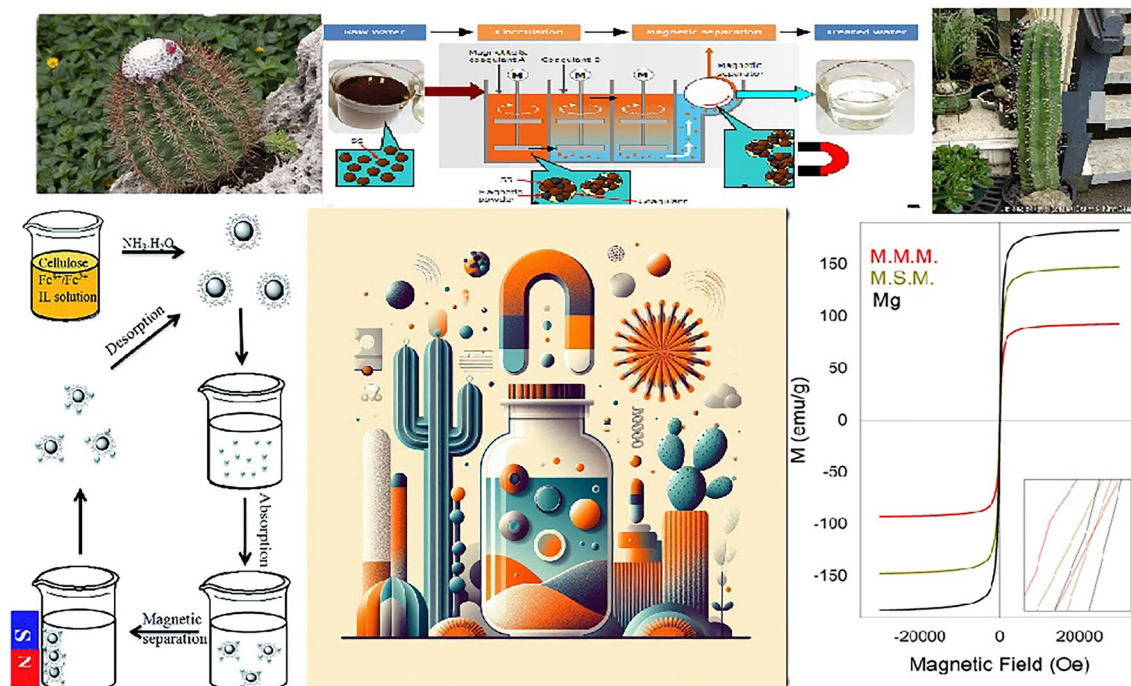
Access to adequate quality water in certain rural areas presents a significant research challenge. This study investigates four magnetized materials with potential application in water treatment as alternatives to conventional flocculants based on iron and aluminum salts. Mucilage and cellulose were extracted from the cacti biomass of *Stenocereus griseus* and *Melocactus* sp. These biopolymers were subsequently magnetized via crosslinking with magnetite (Fe_3O_4). Jar tests were conducted on natural water samples from selected sources to determine the efficacy and optimal dosage of the resulting bioflocculants for turbidity removal. The physicochemical and magnetic properties of the materials were characterized using Fourier transform infrared spectroscopy (FTIR), vibrating sample magnetometry (VSM), X-ray diffraction (XRD), and scanning electron microscopy (SEM). Performance was assessed by evaluating zeta potential (ζ) values, the percentage reduction in turbidity, and achievement of nephelometric turbidity units (NTU) ≤ 2 , conforming to Colombian drinking water standards. Results (removal efficiencies of 98.15% for turbidity, 80.5% for total suspended solids (TSS), and 60.8% for chemical oxygen demand (COD), indicate that magnetic modification of biopolymers, particularly mucilage, offers a promising approach for reducing water turbidity. Furthermore, the influence of pH on the flocculation process was investigated, revealing enhanced performance under acidic conditions. This work has significant implications for the future application of magnetic materials in water treatment and may contribute to addressing water quality challenges in rural communities. This research contributes to the broader pursuit of solutions for social, economic, and environmental issues facing the country, aligning with the United Nations Sustainable Development Goals (SDGs) of the 2030 Agenda.

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Graphical Abstract



Keywords Magnetic flocculant · Mucilage · Magnetite · *Stenocereus griseus* · *Melocactus sp*

Statement of Novelty

Drinking water is a permanent necessity in all cultures and civilizations. For water to be drinkable, it must be treated properly, and flocculation is a part of that process. This research proposes a substitute method for conventional methods, which use substances for coagulation and flocculation that are not always harmless and leave unsafe chemicals as residues. We propose the valorization of cactus residues which, when magnetized and technically applied, help to purify water without leaving dangerous residues and eliminate the turbidity of the water used by the population for consumption.

Highlights

Purpose

The purpose of this work is to provide alternative solutions to the water treatment process and to flocculation, through

the functionalisation (basically magnetisation) of the biomass components of the most abundant cacti in Colombia. Furthermore, the aim is to optimise the flocculation processes so that the aluminium and iron salts, normally used in these processes, can be reduced as much as possible, to avoid the harmful effects that their residues have on the health of the consumer.

- Water treatment systems that aim to obtain drinking water or water of suitable quality for irrigation and cultivation generally use aluminium and iron salts to optimize the process. However, it is necessary to flocculate the water without the use of these salts, due to the environmental impacts they cause. Here we present an alternative proposal.
- The revalorisation of cactus crops magnetising their cellulose and mucilage, is a promising alternative for water flocculation.
- Cactaceae biomasses, which are produced in regions of low soil fertility, become a good quality raw material for extraction, functionalisation, and magnetisation, to be used as water flocculants.

Introduction

Water resources play a fundamental role in the growth of any civilization and are relevant in aspects such as: economic development, poverty reduction and environmental sustainability [1–5]. Water represents almost three-quarters of the earth surface and only less than three percent of this water is characterized as freshwater; unfortunately it is unequally distributed in humanized spaces [6]. On the other hand, evidence suggests that two-thirds of the world population could be living in water-stressed countries by 2025 if current consumption patterns continue. However, despite accumulation of a minor portion of the earth's surface, water resources are arguably indispensable to life [7]. Particularly, Latin America, where approximately one-third of the world's freshwater resources exist, paradoxically faces water scarcity. Approximately 150 million people, roughly a quarter of the region's population, live in water-scarcity areas, and over 400 million lack safe sanitation. This disparity is particularly acute in rural zones.

In these rural areas, water sources are often scarce, and existing aqueducts may not be technically suitable to meet the population's needs. Additionally, climate change disrupts the water cycle, threatening urban and community water security.

Water scarcity in Latin America poses a pressing challenge, especially in rural areas. Research into alternatives like magnetic flocculants derived from native cacti could significantly contribute to addressing this issue and ensuring more equitable water access throughout the region. Traditionally, cacti species have been used as cordovans in water treatment, particularly in ancient civilizations. The interest of this research was to develop an alternative to employ cacti components to improve water quality especially in rural Colombian areas, where it is generally difficult to obtain water suitable for supply. For this purpose, the Suárez River, located in the municipality of Chiquinquirá within the Boyacá Department of Colombia, was considered as the source of treatable water due to its questionable water quality.

Some researchers have considered the mucilage of certain seeds such as basil and quince, or the mucilage of the parenchymatous tissue of some cacti as idoneal for water treatment [8]. The juice from the *Opuntia Ficus indica* pad has proven effectiveness in reducing turbidity by more than 50%, reaching values such as 1.78 NTU, but always accompanied by aluminium sulphate [9–11], which indicates that these materials actually work although they do not improve the water sufficiently to comply with local regulations. On the other hand, magnetic cellulose, MagCell, have been reported not only because it combines

environmental benefits, improved performance, and magnetic properties, making it a promising alternative to traditional flocculants, but because of its relatively easy preparation. However, its suitability depends on the specific wastewater treatment scenario [12, 13]. Against this backdrop, the production of magnetic flocculants from native cactus species offers an innovative solution. By coating flocculant polymers onto magnetic nanoparticles, reusable magnetic flocculants (MFs) can be created. These MFs aggregate suspended particles in water to form magnetically responsive flocs, which can be easily separated and released for reuse in a closed-loop process.

Common coagulants and flocculants used in water treatment include aluminium sulphate (Alum): it's the most widely used coagulant in wastewater treatment [14, 15]. Besides, sodium aluminate, ferric sulphate, ferric chloride. However, their residual effects are controversial from the health point of view [16]. As Flocculants it is frequent to employ polyacrylamides; these are synthetic polymers that enhance the aggregation of particles into larger flocs and natural polymers such as chitosan, which is derived from the shells of crustaceans. These chemicals work by neutralizing the charges on particles in the water, allowing them to bind together and form larger particles that can be more easily removed during the treatment process [17, 18]. The choice of coagulant and flocculant can depend on various factors, including the specific characteristics of the water being treated and the desired outcome of the treatment process¹. Nevertheless, biocoagulants become now as safe alternatives, considering their innocuity and sustainability.

Some authors [19], have tested the incorporation of ferromagnetite with alum improving the alum cluster content, inter-layer structures, surface area, and pore size, enhancing the wastewater treatability performance. Particularly, Santos et al. [20] demonstrated that the use of coagulants with magnetic characteristics led to the formation of magnetic flakes that were attracted to magnetic fields, accelerating the sedimentation process and the removal of physicochemical parameters from water. Nevertheless, magnetization of mucilage and celluloses from cacti is not well known and knowledge on the application of natural coagulants and ferromagnetite is limited. Magnetic natural coagulants/flocculants (MN-C/Fs) have demonstrated their suitability to improve the coagulant/magnetic flocculant's responsiveness, which benefits water treatment by improving superior solid–liquid separation efficiency, shortening hydraulic retention time, and achieving efficient, rapid settlement and lower energy consumption [21]. As a part of these compounds, magnetite has been shown to be effective in removing various contaminants from industrial wastewater, including heavy metals, organic compounds, and bacteria. Moreover, magnetite has a high surface area

and can act as an adsorbent, further enhancing its ability to remove contaminants from wastewater.

To improve the flocculating capability of cactus biopolymers (cellulose and mucilage), a polymer magnetization process was carried out by the Cross-linking method, using magnetite (Fe_3O_4). Ferromagnetic particle-shaped Fe^{2+} Fe^{+3} O_4 particles are used in a variety of in vivo biological applications, such as image resolution enhancement, magnetic resonance imaging (MRI), heat therapy, and drug delivery directed [22, 23], for this reason it's greater biocompatibility and low toxicity were one of the reasons to follow the aforementioned methodology and carry out the magnetic modification of biopolymers for use in water treatment.

It is well-known that cacti species may be a viable alternative for biomass production in water-limited environments, also may be used as animal fodder, or for energy purposes. In this work, two mucilage and two celluloses were modified and the abilities of each magnetized material to reduce water turbidity were evaluated. The four materials were characterized by Fourier transform infrared spectroscopy (FTIR), vibrating sample magnetometry (VSM), scanning electron microscope (SEM) and X-ray diffraction (XRD).

Methods

Stenosereus griseus and *Melocactus sp* were collected in the municipality of Tipacoque (Boyacá, Colombia, 6° 26' 3.143"N, 72° 40' 31.616"W). The reagents used for the cellulose and mucilage extraction were Panreac brand sodium hypochlorite, sodium hydroxide, hydrochloric acid. The reagents used for the synthesis of the magnetized biopolymers were Sigma Aldrich brand Magnetite (Fe_3O_4), Glutaraldehyde and ethanol.

Obtaining Magnetic Biofloculants

Obtaining Mucilage

Cacti thorns were removed before cleaning and chopping. Subsequently, the external part was removed from the

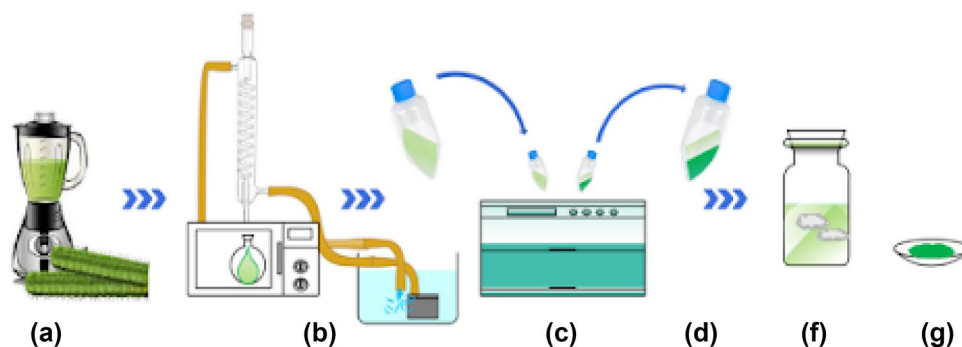
parenchyma of the plant. Then, portions of parenchyma were liquefied, adding water in 3:1 sample/water proportion, as shown in Fig. 1. Furthermore, the liquefied sample was heated in a home microwave oven coupled to Reflux, for 5 min and 8000 Hz. Afterwards, the sample was centrifuged (Hettich—Zentrifugen centrifuge Rotina 46 S) for 20 min and the supernatant was separated. The mucilage was precipitated with ethanol, in a ratio of 3:2 ethanol/sample. Finally, the mucilage was dried in an oven (Lab companion, ON—11E Natural Convection Oven) at 45 °C for 8 h, as indicated by Hosseinzadeh methodology [24]. The precipitate from centrifugation was recovered for cellulose extraction.

Obtaining Cellulose

The cellulose was obtained according to the methodology reported by Moreno et al., [25] and Nagarajan et al., [26], with some modifications. First, the extractables were removed from the sample according to the Tappi 204-cm-97 standard treatment [27, 28]. With this technique, oils, waxes, and resins were removed. Subsequently, it was centrifuged for 5 min at 3000 rpm and washed in triplicate with distilled H_2O at 80 °C.

Next, cellulose was extracted according to the methodology reported by Singh, et al. [29], with some modifications. In a beaker, the fibre was added along with 20 mL of 0.5 M NaOH; the temperature was maintained within 84–90 °C and constant stirring for 20 min. Then, it was centrifuged for 5 min at 3000 rpm and this process was repeated and washed with distilled water until a neutral pH. Afterwards, the supernatant was removed and again the fibre was preserved. Subsequently, the fibre was added to a 20 mL $\text{CH}_3\text{COOH}/\text{H}_2\text{O}_2$ solution (10 mL of concentrated glacial acid (99%) and 10 mL of 35% hydrogen peroxide). It was allowed to react with constant stirring for 4 h and at room temperature. Then, it was centrifuged at 3000 rpm for 5 min and washed with distilled water. The supernatant was again removed, and the fibre was preserved. The fibre was placed in a beaker along with 20 mL of hydrochloric acid, 30% HCl. The solution was kept under constant stirring at 90 °C for 1 h. It was then allowed to cool and centrifuged

Fig. 1 Obtaining mucilage from cactus *Stenosereus griseus* and *Melocactus sp*. **a** the parenchyma was liquefied, **b** microwave heating, **c** centrifugation process, **d–e** separation, **f** dried mucilage



again at 3000 rpm for 5 min. Finally, it was dried at 50 °C until constant weight.

Preparation of Magnetic Biofloculants

The magnetic modification of cellulose and mucilage was carried out, thus obtaining four (4) materials that were tested flocculants. Magnetic modification of cellulose and mucilage of *Stenosereus griseus* and *Melocactus* sp, was followed according to the Cross-Linking Method proposed by Hizam et al., [30], with minor modifications, as shown in Fig. 2.

Briefly, 1 g of biopolymer and 1 g of magnetite (Sigma—Aldrich) were dispersed in 100 mL of distilled water. This solution was heated to 65 °C in a shaking water bath for 6 h, followed by cooling to room temperature. Next, 1.5 mL glutaraldehyde (25%—Sigma Aldrich) was added dropwise and stirred for 1 h at room temperature to allow cross-linking. Finally, it was filtered and washed with ethanol: water in equal proportions. The sample was then decanted and dried at 50 °C to constant weight.

Application of Biofloculants in Water Samples. Coagulation-Flocculation Process

The performance of the synthesised biofloculants was studied using a jar Test in triplicate. The purpose of the jar test is to estimate the minimum coagulant dose required to achieve certain water quality goals. The method consists of a six-paddle stirrer Jar test, linked together by a geared instrument, providing a speed control. The pre- and post-treatment analyses were carried out in accordance with the Colombian Standard that regulates the quality conditions of treated water, as established in the Colombian technical standard 3903 of 1996.

Firstly, the optimal dose was determined and selected doses are reported in Table 1. Due to the nature of the biofloculants (materials magnetized solids), some variations were made, as explained next. The magnetic biofloculant was added to 1L baker and mixed on a rotary shaker at 120 rpm, 19 °C for specific times. The flocculant flocs were concentrated and separated from the suspension medium using a cubic permanent Nd2 Fe14B magnet (5 cm L × 5 cm W × 1 cm H) with a magnetic induction intensity of 2000 G (Hiway electrical Co., Ltd.), which was placed at the bottom

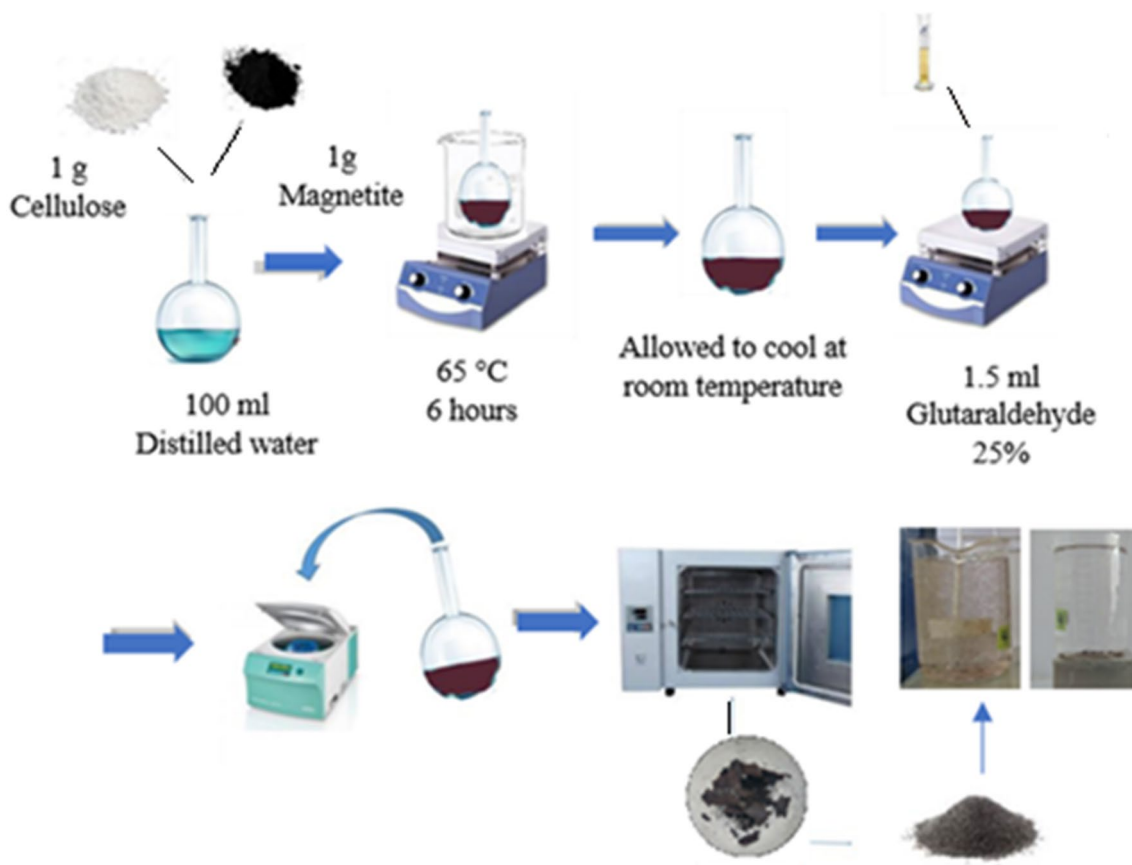


Fig. 2 Magnetization of biopolymers

Table 1 Nomenclature and optimal dose

Initial polymer	Magnetized biopolymer	Nomenclature	Optimal dose (mg/l)
<i>Stenocereus griseus</i> cellulose	Magnetized <i>Stenocereus griseus</i> cellulose	C.S.M	140
<i>Melocactus sp</i> cellulose	Magnetized <i>Melocactus sp</i> cellulose	C.M.M	160
<i>Stenocereus griseus</i> mucilage	Magnetized <i>Melocactus sp</i> mucilage	M.S.M	150
<i>Stenocereus griseus</i> mucilage	Magnetized <i>Stenocereus griseus</i> mucilage	M.M.M	120

of the vessel for 30 min. The magnetic separation conditions were investigated by varying the pH, which was adjusted in 5,6,7,8 and, using either 1 M HCl or 1 M NaOH. All experiments were carried out at 19 °C.

Analytical Analysis

The results were evaluated by varying the pH of the water sample (5,6,7,8 and 9). A fixed effects factorial design was carried out to evaluate the effect of pH or the type of BFM on the final turbidity results in flocculation, taking compliance with resolution 2115 mentioned above as a reference. Five pH levels were considered (5, 6, 7, 8 and 9), and four bioflocculant samples (C.M.M, C.S.M, M.S.M, M.M.M). All analytical procedures were conducted in triplicate and followed the methodology of the Standard Methods for the Examination of Water and Wastewater [31].

Turbidity removal tests were carried out in triplicate, and the response variable measured was turbidity measured as nephelometric turbidity units (NTU).

Characterization

Before evaluating the performance of the bioflocculants, the materials were characterized using a PANalytical X'pert PRO MPD Diffractometer (DRX), Ultra-fast, in order to identify if there was a change in the crystallinity of the material. In addition, the magnetic properties of the bioflocculants were examined using a Vibrating Sample Magnetometer (VSM) Model Lakeshore 665. VSM measurements were determined in a known amount of sample. They were studied by measurements at a temperature of 300 K and with magnetization measurements depending on the applied field in a range between – 30 kOe to 30 kOe, in the Materials Physics laboratory of the U.P.T.C., Colombia, to evaluate the magnetic susceptibility and quantify the magnetic response of each one.

Besides, the bioflocculants were characterized by a Nicolet IS 50 FT-IR with ATR accessory, infrared spectrophotometer (FTIR) to identify the functional groups in each material. The samples were recorded between 4000 and 400 cm⁻¹. To obtain morphological and microstructural characteristics of

the materials, a JEOL JSM 6490 LV Scanning Electronic Microscope, was used. The samples were fixed on a graphite tape, a thin gold (Au) coating was applied (DENTON VACUUM Desk IV equipment) and analysed in the scanning electron microscope in high vacuum to obtain high resolution images. The secondary electron detector was used to evaluate the morphology and topography of the samples. Elemental analysis was performed using an X-ray Microprobe-EDX (reference INCA PentaFETx3 Oxford Instruments).

Results

Determination of the Optimal Dose

The application of Al₂(SO₄)₃ was necessary as an adjuvant. The adjuvant optimal dose (without magnetic bioflocculant) turned out to be 2 mg/L, included in the optimal doses showed in Table 1. The magnetized flocculant far outperformed the adjuvant, but the alum best performing dose was also far lower compared with other reports [32].

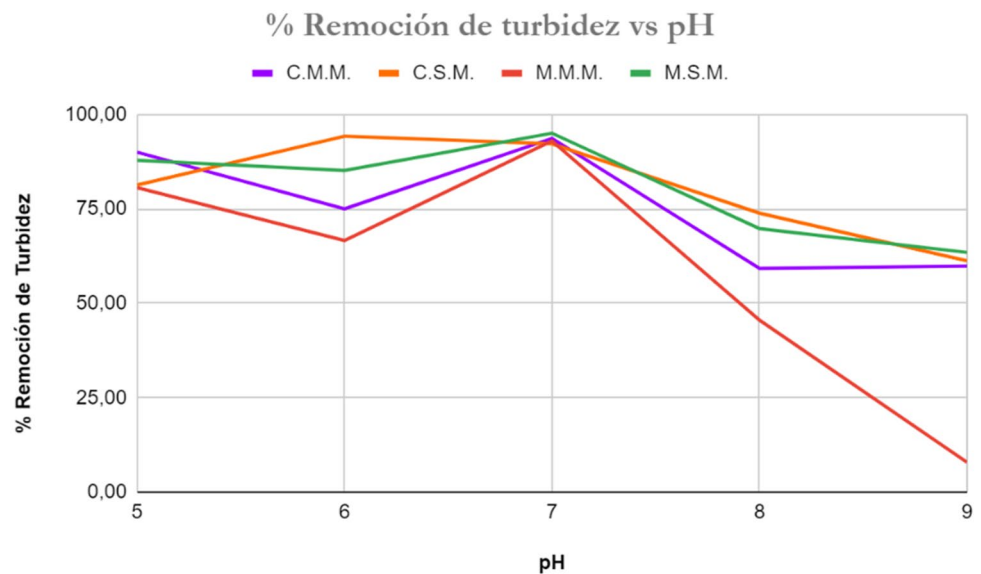
Effect of pH in Water Treatment

The statistical analysis was carried out based on the results provided by the R statistical package. The Table 2 shown below was obtained:

From the previous result, it is inferred that both, the variation in the pH of the treated water and the choice

Table 2 Anova table

Response Variable: Turbidity					
	Df	Sum Sq	Mean Sq	F value	Pr (> F)
BFM	3	26,411	8,804	12,312,9	< 2,2 e-16***
pH	4	259,132	64,783	90,605,7	< 2,2 e-16***
BFM:pH	12	52,534	4,378	6122,8	< 2,2 e-16***
RESIDUALS	40	0,029	0,001		

Fig. 3 Turbidity removal % vs pH

of the type of bioflocculant, used during the treatment, interfere significantly with the final turbidity since the *Pr* value ($> F$) is less than 0.05[33].

Furthermore, Fig. 3 demonstrates a significant impact of the bioflocculant on reducing turbidity across various pH levels. The study reveals that bioflocculant performance is notably better under acidic conditions. This result disrepute with the results reported by Mohamed Noor et al. [34].

According to the data reported in the Table 1, the material with the best performance was M.S.M. because it showed the lowest turbidity value, in concordance with the Colombian regulation and with a lowest flocculant-coagulant dose corresponding to 120 mg/L. Likewise, it is evident that this material allows better results at pH between 5.0 and 7.0 (the neutral value), increasing this way its range of applicability. Nevertheless, all materials have a better performance at slightly acidic pH, in opposition to former results, as those reported by Wilson, J, et al. [16].

The second most favourable result was produced by the bioflocculant marked C.S.M., whose results are in accordance with the national standard, and additionally its applicability is optimal at pH 6.0 and neutral.

As shown in Fig. 3, the turbidity results vary as a function of pH. All four materials perform best at slightly acidic pH and the best results are at neutral pH. This is a favourable condition considering that the sample does not require variations in pH for the magnetised bioflocculant to perform favourably and efficiently in the treatment [16]

Water characterization

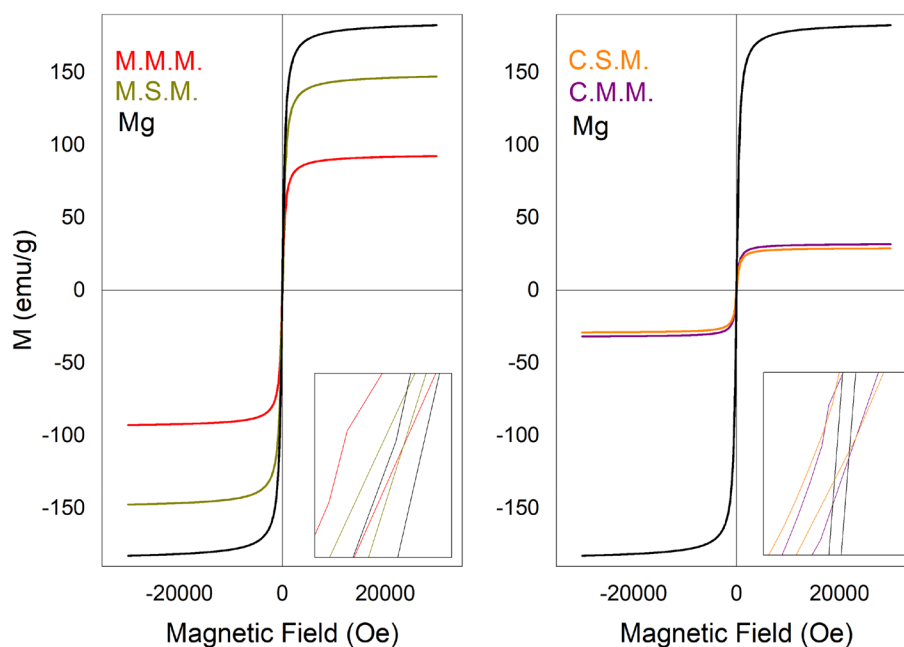
Water samples were collected in a single sampling to guarantee its uniformity, and was then homogenized, fractionated and appropriately stored under refrigeration for later use. The source was the Suárez River located in Chiquinquirá, Colombia, and samples were collected at different times and with different levels of precipitation. These effluent supplies water to a broad rural population, which is why it was considered suitable for sampling. Water samples in reported results ranged from artificially contaminated water, which would provide unequivocal replication without the influence of external variables, to different natural water supplies, influenced by environmental conditions, or a hybrid of these two types of water, i.e. a natural water source with artificially created parameters. For this work natural water samples were selected, because of the needing of provide suitable alternatives to rural populations.

The selected water samples were due to the conditions of the different parameters analysed by a laboratory, accredited by the Colombian Accreditation Body of Colombia, and whose results are summarised in Table 3, it was decided to work with samples from this water source.

The microbiological data shown in Table 3 do not meet the required parameters since the water was not subjected to a disinfection process, which according to Colombian regulations must exist in the treatment of raw water and in turn be preceded by filtration.

Table 3 Chemical and microbiological results test

Values of the parameters required by the Colombian standard		Results	
Chemical characteristics	Maximum acceptable value	Pre	Post
pH	6,5–9.0	6,98 (19,2 °C)	6,65
Total Organic Carbon (mg/L)	5,0	4,94	13
Nitrites (mg NO ₂ ⁻ /L)	0,1	< 0,0140	0,0300
Nitrates (mg NO ₃ ⁻ /L)	10	< 1,38	1,61
Aluminium (mg Al ³⁺ /L)	0,2	< 0,0460	< 0,0460
Total Hardness (mg CaCO ₃ /L)	300	128	141
Sulphates (mg SO ₄ ²⁻ /L)	250	< 8,90	< 8,90
Microbiological characteristics	Maximum acceptable value		
Total coliforms (UFC/100 cm ³)	0	378	406
Escherichia coli	0	304	268

Fig. 4 VSM results

Biofloculant Characterization

VSM Analysis

The magnetization curves of magnetite (Mg), M.M.M., M.S.M., C.S.M. and C.M.M are displayed in Fig. 4. They exhibit ferromagnetic behavior at room temperature due to non-zero coerciveness and remanence [16]. Accordingly, both a) and b) show how magnetization increases as the intensity of the magnetic field increases until magnetization saturation (Ms) is reached (Table 4).

When a magnetic material is subjected to a uniform magnetic field, it generates a dipole moment proportional to the material. As the sample vibrates, it introduces disturbances

Table 4 Magnetic characterization results

Sample	Hc (Oe)	Mr (emu/g)	Ms (emu/g)
Mg	− 30,216	8,280	182,591
MMM	− 35,290	6,021	92,560
MSM	− 11,760	2,466	147,313
CSM	− 45,856	2,523	28,806
CMM	− 50,496	2,729	31,751

into the external magnetic field. Consequently, the dipole field of the sample is converted into an alternating current electrical signal, which produces a response signal. This response signal enables the interpretation of the magnetic

properties inherent to the material, thereby facilitating the classification of the magnetic material.

As expected, the M_s value of magnetic biofloculants was lower compared to magnetite. This is because the magnetization is affected by the particle size [17]. The structure of each of the biopolymers was modified with magnetite, Mg, which conferred magnetic properties to the materials, as indicated by the decrease in M_s for both magnetite with cellulose, and magnetite with mucilage.

However, it was evident that the celluloses decreased the M_s value to a greater extent. This means that the magnetized celluloses have a lower “maximum capacity” to align and maintain their magnetic moments in the direction of the applied field [15]. Likewise, when comparing mucilage with cellulose, the coercivity (H_c) in the cellulose BFM decreased to a greater extent, which translates into greater susceptibility to the influence of external magnetic fields, which can cause a greater tendency to reduce its magnetic capacity. Nevertheless, since the purpose of this synthesis was to obtain magnetized materials, it is demonstrated with the remanence values (M_r), that the biofloculants maintain values different from zero, being M.M.M. the one with the highest value. This indicates that even after functionalization, biofloculants still show high saturation although not completely superparamagnetic characteristics. According to Okoli et al. [35], superparamagnetic materials have a high saturation of magnetisation and energies of coercivity, and remanence equal to zero. The above allows to assert that ferromagnetism was maintained in the four materials and, therefore, the magnetic characteristics that magnetite conferred on them [20].

DRX Characterization

Figure 4 represents the diffractograms obtained. There, magnetic biofloculants were compared with their biofloculant precursors and with magnetite.

In each one of the 4 BFM, 6 characteristic signals are evident, evidencing the magnetite incorporation on celluloses (and mucilage), and the consequent modification of each matrix. As shown in Fig. 4, in the composites containing magnetite, show some reflections of magnetite at different values of 30.5° , 36.2° , 41.6° , 50.7° , and 63.3° , assigned to the (220), (311), (400), (511), and (440) planes, respectively. Furthermore, there is no additional signal that could suggest contamination in the sample [21].

The two materials with the presence of cellulose show a typical slight signal at approximately $2\theta = 26^\circ$ that may indicate the presence of a certain degree of crystallinity in the cellulose [36] and that correlates with the signal in both non magnetized celluloses[37].

Additionally, although mucilage does not reflect significant signals for each diffractogram, (Fig. 5., M.S. and M.M.), a difference is evident compared to magnetite, the Mucilage BFM exhibit signals with larger widths, which can be correlated with a successful coating.

FTIR

Biofloculants functionalisation was examined with the FT-IR spectroscopy. In Fig. 6 the spectra show the possible peaks of both celluloses and mucilage, at about $550\text{--}4000\text{ cm}^{-1}$, characteristic of the carboxylic acid, alcohols and aromatic absorption bonds, among others.

In general, cellulose and magnetized cellulose, show a medium transmittance at frequency of 1640.60 cm^{-1} . This is a stretching vibration attributed to the --C=C-- in alkene. The band appearing at $1020\text{--}1630\text{ cm}^{-1}$ indicates the stretching vibration of the aliphatic amines. The peaks between 3000 cm^{-1} and 2800 cm^{-1} indicated the presence of --CH and --CH_2 group of cellulose [38]. The theoretical value of 2910 cm^{-1} would correspond to the antisymmetric stretching of the CH bond of cellulose while that of 2850 cm^{-1} would correspond to the symmetrical one. Former peaks appear at 2898 cm^{-1} and 2917 cm^{-1} in the FTIR spectra of MS and MM. The peak at 719 cm^{-1} is also characteristic of the C–H group in the cellulose [39]. Besides, the band at 570.92 cm^{-1} mainly contributed by Fe–O functional group from magnetic powder during crosslinking. As it was found in this work, a transmittance range that goes from 0 to 40, two representative signals are evident, a signal at 3454.2 cm^{-1} corresponding to the O–H bond tension vibration and another finer signal at 2381.3 cm^{-1} attributed to a stretch attributed to O=C=O . When modifying C.S.M., a new signal appears at 2898.5 cm^{-1} that corresponds to the C–H aldehyde bond, which is also reflected in the C.M.M.

In the IR spectrum of C.M.M. The C–H aldehyde signal is accompanied by a new signal at 2917.1 cm^{-1} that corresponds to the --C--H alkane bond [25, 26], both because glutaraldehyde is a part of the reaction system.

IR spectra of mucilage M.S. and M.M., present two characteristic signs; one at 3350.5 cm^{-1} that corresponds to the O–H vibrational bond and another signal at 1603.8 cm^{-1} corresponding to the C=O bond of the sugars in the sample.

The IR spectrum of M.S. It additionally presents three more signals such as 2919.5 cm^{-1} and 2846.1 cm^{-1} that correspond to the alkane and aldehyde C–H bond respectively, which are conserved after magnetization [38, 40]. Peaks between 1430 and 1420 cm^{-1} in the identical spectra of C.S.M. and C.M.M. are due to the CH_2 scissoring motion in cellulose. After the structural modification of the cellulosic materials, the peaks around 2988 and 2292 cm^{-1} , which are associated with the C–H stretching of the

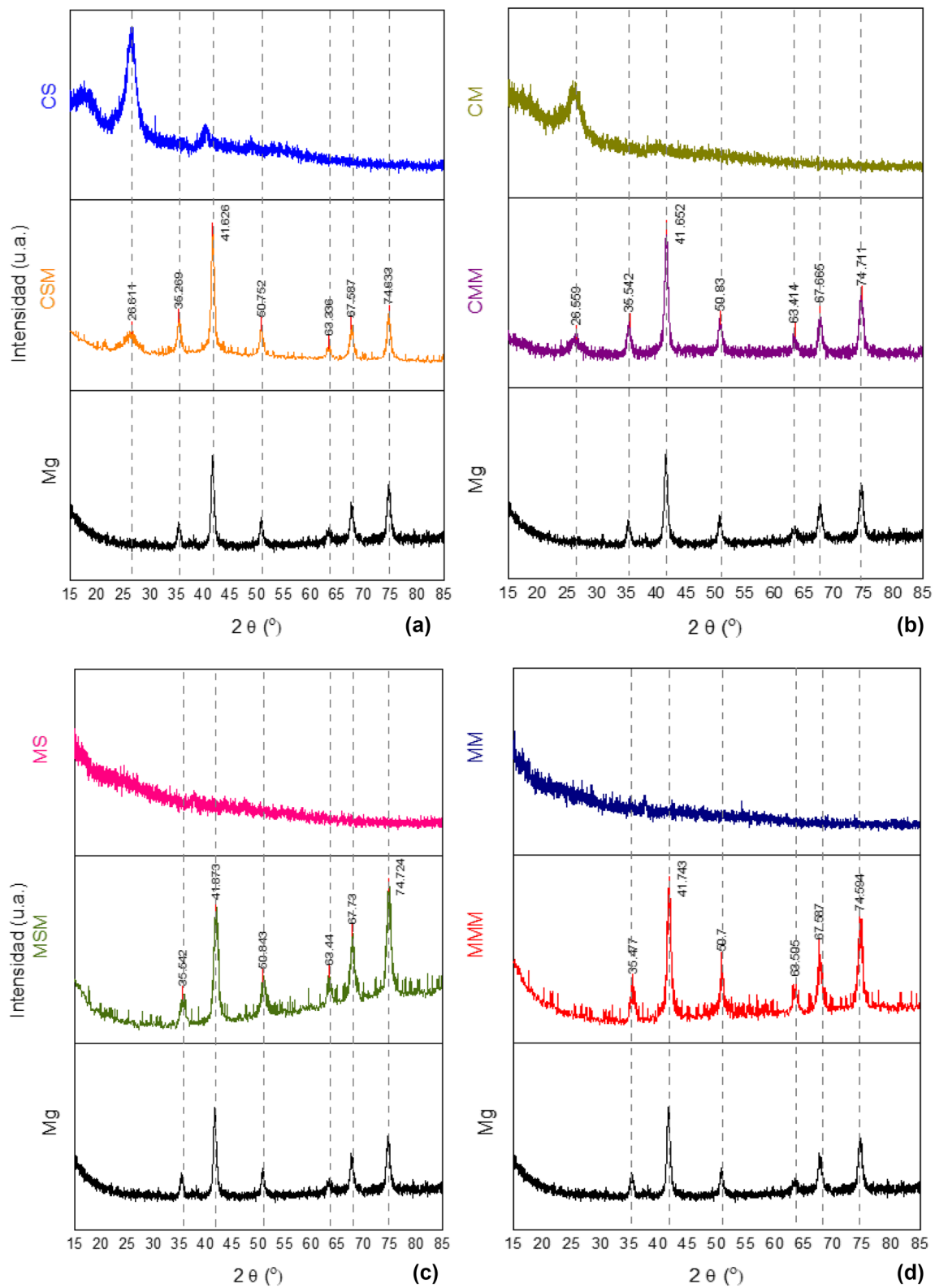
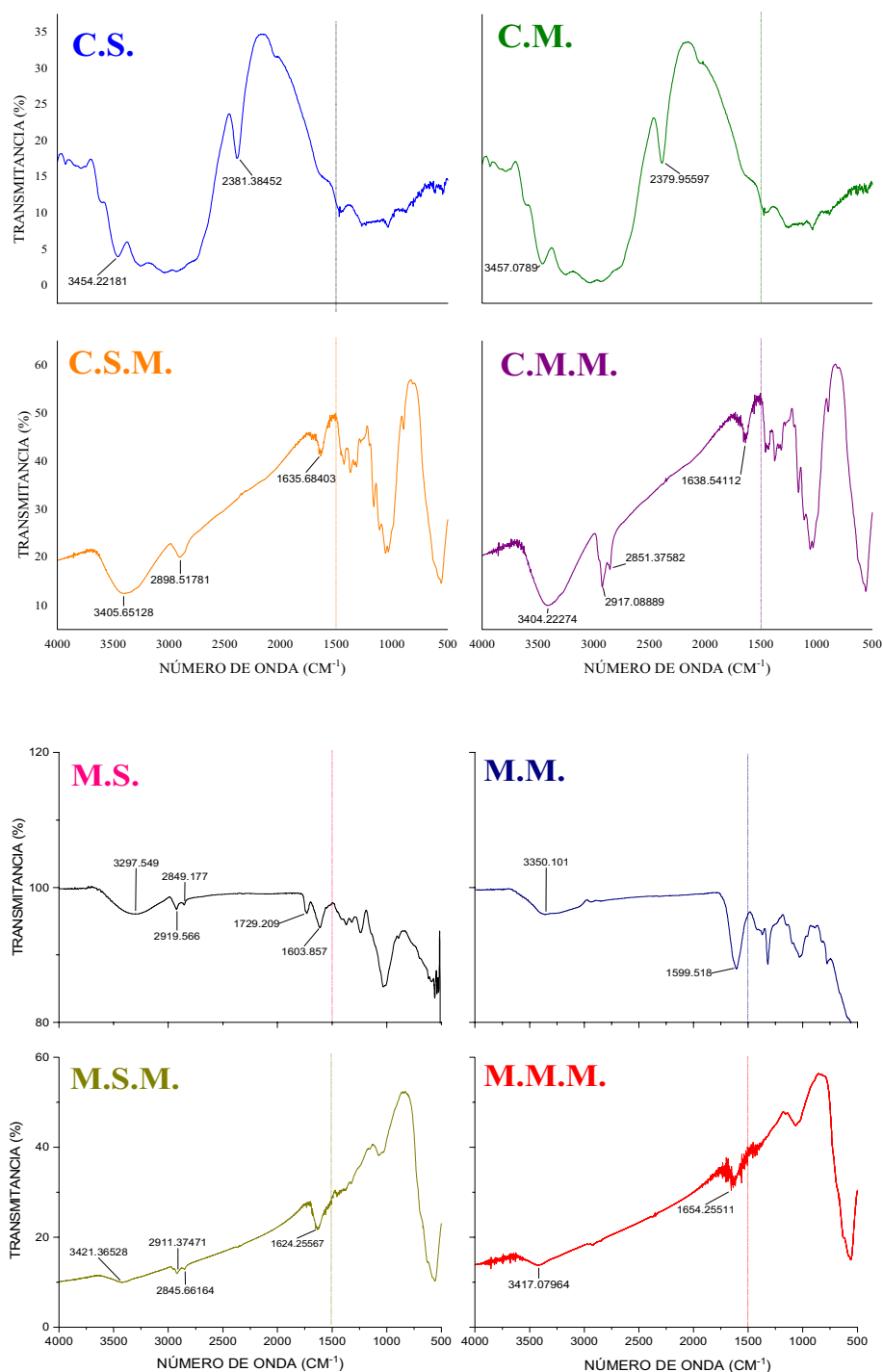


Fig. 5 DRX Diffractograms: **a** CS, CSM and Mg, **b** CM, CMM and Mg, **c** MS, MSM and Mg, **d** MM, MMM and Mg

Fig. 6 Fourier Transform Infrared Spectroscopy (FTIR) analysis of Biofloculants



cellulosic materials, persisted, though with slight reductions in intensity. This observation indicated that the structural properties of the magnetized cellulosic materials were not completely altered. Additionally, the presence of the peaks at 2917 and 554 cm^{-1} of the prepared composites confirmed successful incorporation.

Following this, M.S. Without magnetization, it presents a signal at approximately 1729.2 cm^{-1} , which corresponds to the C=O tension bond, which after being modified, the material is no longer visible. This may be the result of magnetization and coverage by glutaraldehyde, as well as reported by [41].

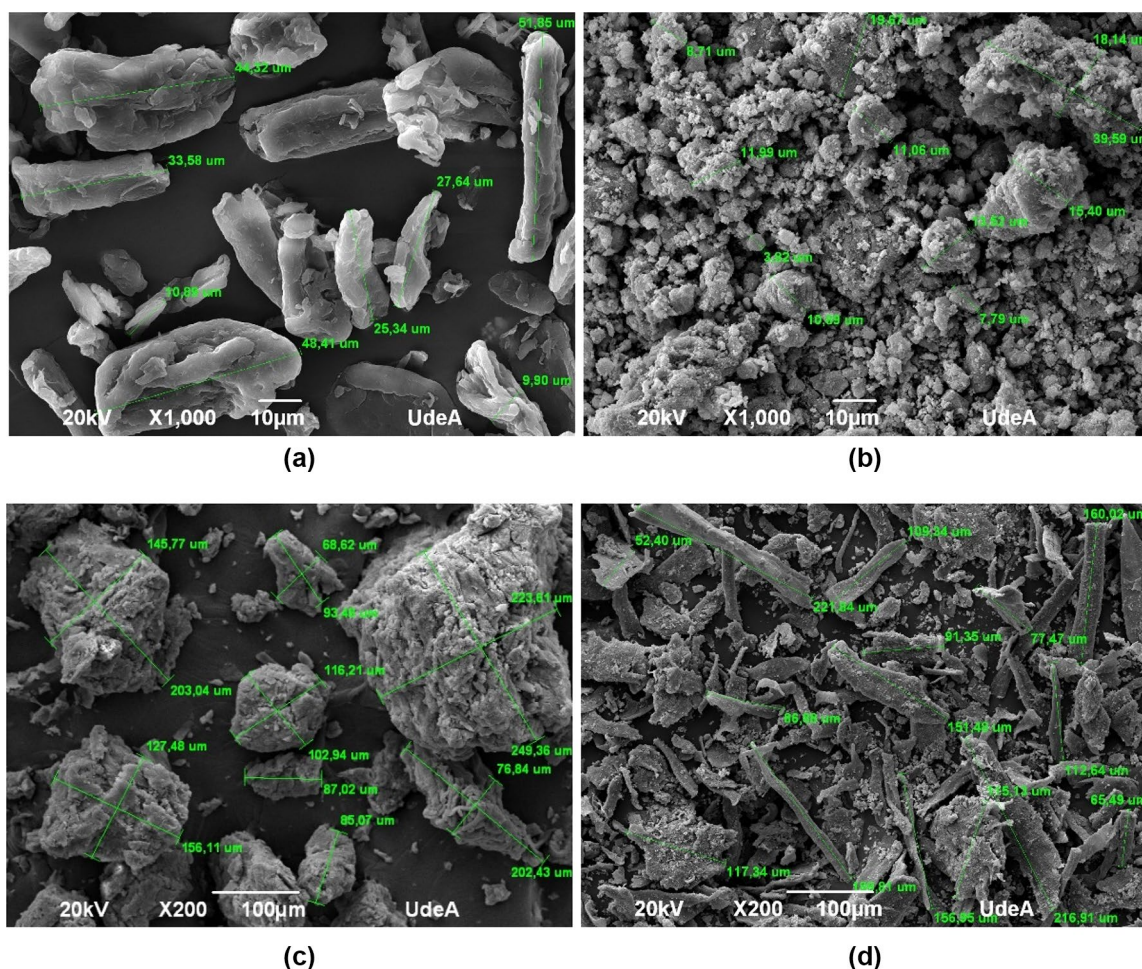


Fig. 7 SEM images of Biofloculants: **a** C.S., **b** C.S.M., **c** C.M., **d** C.M.M

Finally, the signal at approximately 580 cm^{-1} , is due to the contribution mainly of the Fe–O functional group of the magnetite during the reaction [41].

SEM Analysis

Regarding the morphology of the cellulose and mucilage biofloculants, a difference was evident between CS/CM and MS/MM, Fig. 7. This is because the origin and species of the cactus does influence the final morphology of the biopolymer. The surface morphology of the obtained BFM were different from that of the Cellulose, suggesting that the introduction of Fe_3O_4 particles in the cellulosic did affect the surface morphology of the cellulose composites. Despite having the same molecular components and being cellulose or mucilage respectively, the chains can be longer or shorter, depending on their origin, longevity of the plant, among others. This observation is slightly opposite to some reported magnetized celluloses coming from different origins [42, 43].

In addition, the size of the composites can vary depending on the type of extraction, so a morphological difference in the biopolymers initially obtained was to be expected. This issue is confirmed in Fig. 7, section (a) and (b).

The micrographs show white and black surfaces uniformly distributed on the composite surface, which indicates that the iron oxide nanoparticles were well dispersed on the composite. It exhibits a morphology in the form of disaggregated rods with an irregular surface and measurements that are in the range of $10.89\text{ }\mu\text{m}$ and $51.85\text{ }\mu\text{m}$. Some agglomerates are present as well, mainly in mucilage composites. From the SEM image (Fig. 7 b and Fig. 7 d), large and fractal-like flocs denote the development of Al species clusters. Figure 7a and Fig. 7c, reveal that the Al species influenced the aggregation kinetics, which reduced the alum aggregated structures to a smooth surface. When the magnetization of the material is carried out, microstructural changes appear. Biopolymers without magnetization exhibit a typical cellulosic and mucilage morphology with rounded structures, some elongated

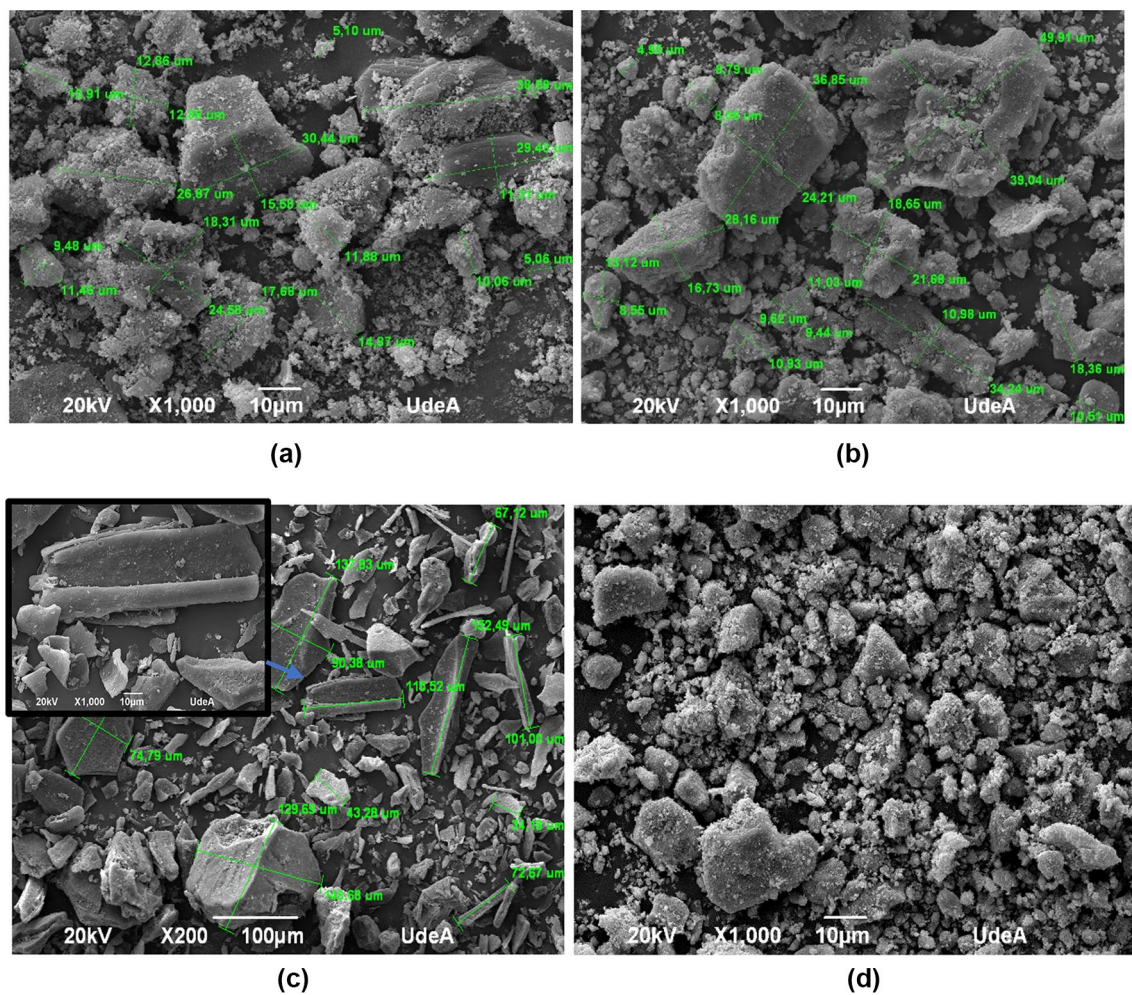


Fig. 8 SEM micrographs: **a** M.S., **b** M.S.M., **c** M.M., **d** M.M.M

considerably, agglomerated and with non-homogenic measurements. According to some authors [20], is probable that the magnetic characteristics of the functionalized coagulants allow alignment of all magnetic moments towards the applied magnetic field, whereas in the absence of a magnetic field, magnetic nanoparticles lose the affinity to retain a magnetisation network. This characteristic could permit different answer of magnetic and non-magnetic compounds when applied to wastewater samples.

Figure 8, in its upper left section (a) and (b), shows the morphology of M.S. and M.S.M. respectively, with a magnification of 1000.

The microstructure of mucilage differs from that of cellulose. The micrographs in Fig. 8 show irregularly shaped rough surfaces and mesoporosity, with a massive heterogeneity that may favour the agglomeration of large flocs.

It is noticeable that after the magnetization reaction, agglomeration occurs in the morphology of the material,

accompanied by some rounded structures and with the absence of the initial rods and the particles aggregate to varying degrees, which could be due to the interaction of the magnetic dipole moment between the particles. [21].

Finally, in Fig. 8, in section (c) and (d), with a magnification of 1000, it is evident that the polymer (section c) presents rounded structures. Their measurements range from 9.48 μm to 30.44 μm and agglomerated sticks with lengths from 29.43 μm to 38.59 μm .

On the other hand, in section (d), larger irregular structures are evident, from 13.12 μm to 49.9 μm , with more agglomerated morphology [28]. Fe_3O_4 -mucilage composite flocculant has poor dispersibility and partial agglomeration because mucilage is coarser and less adsorbent than cellulose, which results magnetic particles collide with each other to cause agglomeration under the action of mucilage adhesion[44].

Flocculation Tests

The influence of flocculant dosage on the percentage removal of pollutants was investigated by varying the biopolymers dosage from 12 to 16 mg/L. These doses are significantly lesser than those reported in different coagulation/flocculation tests, however, with the added advantage of a lower sedimentation time due to the functionalization of Fe_3O_4 with the cacti components [18, 34]. In this work, MMM and CSM recorded the highest percentage reduction of some parameters: 98.15% 80.5%, 60.8% for Turbidity, TSS, and COD respectively. Probably, the major limitation of using MN-C/Fs is their relatively low coagulation/flocculation efficiency compared to synthetic chemicals, when synthetic water samples are analysed. This is due to the lower charge density and smaller size of natural materials, which can result in lower binding capacity with waterborne contaminants. On the other hand, some authors have reported coagulation efficiency with different coagulants, such as ferro magnetite (F) and eggshells (E). As coagulants showed high potential for water and wastewater treatment as alternatives to conventional alum. This work has demonstrated that those coagulants could be applied in lower and almost nil doses and magnetized flocculants reach similar or better results, when natural wastewater is treated.

Additionally, the magnetic properties of these systems make them easy to separate from the treated water, which reduces the need for additional treatment steps and minimizes the conventional potential of synthetic coagulants/flocculants for producing harmful by-products.

Conclusions

The methodology of modification of celluloses o mucilage showed in the present study, can also be easily adjusted to promote the incorporation of different hydrophobic nanoparticles in the cellulose or mucilage matrix, conferring new properties such as optical and electronic ones.

Results showed here underscore the advantages of water treatment approach that encompasses magnetic particles with natural flocculants, which offers a novel water treatment. Besides, the usage of chemical coagulants traditionally employed in treatment plants, has been reduced by more than 60% during the water treatment process, which is convenient for drinking water consumers.

Plant-based coagulants/flocculants are readily available and inexpensive, but their effectiveness may vary depending on the source and the quality of the material. Functionalized and magnetized cellulose and mucilage from cacti, have demonstrated being sustainable, innocuous, and safe flocculants.

According to these results, the stability of MN-C/Fs under various environmental conditions, such as pH, temperature, and salinity, may be a challenge to be addressed. Further research is recommended on the mechanisms of action of MN-C/Fs, which can improve their efficiency and increase their applicability for a wide range of wastewater treatment applications in rural areas, where technical treatment is often scarce.

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Data Availability The set of data related to this work has been presented in the manuscript and is available upon reasonable request from the corresponding author Oscar Medina.

Declarations

Competing Interests The authors declare no competing interests.

Ethical Approval All the authors have agreed to submit this manuscript to Environmental Science and Pollution Research, and this manuscript contains material that is the authors' own original work and has not been published elsewhere. Furthermore, this paper is not currently being considered for publication elsewhere. The paper reflects the authors' own research and analysis in a truthful and complete manner. The paper properly credits the meaningful contributions of co-authors and coresearchers. The results are appropriately placed in the context of prior and existing research. All sources used are properly disclosed (correct citation).

Consent to Participate Not applicable.

Consent for Publication Not applicable.

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