

Article

Low-Cost, Biodegradable, and Magnetic Biocomposite of *Luffa cylindrica* Fruit and Natural Magnetite by Removal of Microplastics

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Abstract

Techniques such as adsorption have been widely adopted to remove residual microplastics (MPs) because they are efficient at removing contaminants from aqueous environments. In this study, we report a novel, low-cost, biodegradable, and scalable biocomposite (LCMAG NaOH) derived from *Luffa cylindrica* fruit (LC) and micrometric natural magnetite (MAG), prepared without organic solvents, for the removal of MPs. The performance of LCMAG NaOH was evaluated for the removal of polystyrene (PS) and polyethylene terephthalate (PET) MPs, with particle sizes ranging from 75 to 600 μm in three distinct aqueous media: drinking water, simulated seawater, and water collected from the eutrophic lake of Dourados, MS, Brazil. The material was also evaluated for capture capacity and for reutilization in drinking water over three cycles, using a neodymium magnet. The biocomposite exhibited maximum removal capacities of 163 mg g^{-1} and 158 mg g^{-1} for PS and PET, respectively, in drinking water. Additionally, it demonstrated high magnetic recovery efficiency (>90% of the initial mass) and good reusability after immersion (10 and 20 min) and a dry step during the first cycle.

Keywords: *L. cylindrica*; magnetite; microplastics

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1. Introduction

Water pollution by microplastics (MPs) is a global problem that threatens not only humans [1] but also the entire planet (available at <https://www.unep.org/plastic-pollution>, accessed on 3 May 2026). About 9.2 billion tons of plastic were produced between 1950 and 2017, and approximately 7 billion tons were discarded as plastic waste [2,3]. The removal of microplastics (MPs) from drinking water and wastewater remains a significant environmental and technological challenge. Most plastics are slow to degrade and persist for over hundreds of years in the environment [3,4]. MPs are defined as plastic particles ranging from 0.0001 to 5 mm in size that originate from the fragmentation of larger plastics and from everyday products such as cosmetics and textile fibers [5].

The capacity to retain and quantify MPs varies globally due to unequal access to water treatment technologies across different regions. Developed countries, such as those in Europe, have higher efficiency in removing microplastics at wastewater treatment plants (WWTPs), with removal rates of 72% to 99% [6,7]. In contrast, developing countries face methodological, financial, and infrastructure challenges, resulting in removal efficiencies ranging from 15% to 79% [8–10]. In underdeveloped countries in Asia, Latin America, and Africa, plastic pollution is more noticeable due to inadequate collection services and limited landfill capacity [1,11].

Brazil is one of the world's largest producers and consumers of plastic, generating around 12 to 14 million tons of plastic waste per year, with most of the plastic consumed used for single-use packaging. Only about 21% of plastic waste generated in the country is recycled. More than 60% of plastic packaging produced lacks a monitored final destination, being discarded in landfills, dumps, or improperly disposed of in the environment (<https://www.abiplast.org.br/publicacoes/indices-de-reciclagem-mecanica-de-plasticos-pos-consumo-no-brasil-2025/>, accessed on 3 May 2026). Concomitantly, there is no legislation limiting microplastic concentrations in drinking water, and the Brazilian sanitation companies do not carry out any analysis to detect MPs in drinking water intended for human consumption [12].

The removal efficiency of MPs in conventional water treatment processes typically ranges from 42 to 82% [13]. For complete removal, a new separation step is necessary. Among the available techniques, adsorption has been widely employed for the removal of residual MPs, as it is an effective and cost-efficient method capable of achieving removal efficiencies of up to 99.9% [14]. The main adsorbents reported in the literature include silica, zeolites, activated carbon, metal–organic frameworks (MOFs), and magnetic materials, such as magnetic biochars [14–18]. Biochar magnetic materials have stood out in numerous studies across different groups of adsorbents used for MPs removal, owing to their use of renewable resources and accessibility [14,19]. Biochar can be obtained from natural materials and residues, such as bamboo [20] and coconut fiber, *Luffa cylindrica* [20,21], sugarcane bagasse [22], and magnetite. Magnetic biochar is produced by incorporating iron oxide phases, such as magnetite or natural magnetite, into a biochar composite [23]. Natural magnetite at the micrometric scale offers the advantages of high stability and the use of a residue from other mineral exploration [24]. The incorporation of magnetic properties into biochar increases its capacity to adsorb MPs and facilitates their separation from the aqueous environment. The adsorbed MPs on the magnetic biochar can be easily separated from the aqueous environment using magnets or an external magnetic field [25,26].

Although the use of magnetic biochar is a promising technique for removing MPs from water, it still faces economic, environmental, and technical limitations that hinder its large-scale application. The synthesis of these materials generally involves chemical substitution and pyrolysis, which require high energy inputs, thereby significantly increasing operational and production costs [14,19,26]. Furthermore, pyrolysis can

generate toxic byproducts and requires emission control systems, which increases operational complexity. Regeneration of magnetic adsorbents often requires intensive thermal treatments, such as high-temperature pyrolysis (500–600 °C), to restore adsorption capacity and degrade retained microplastics. The recovery of the adsorbent under laboratory conditions has been efficient, but it faces limitations in scaling up for industrial use [27,28]. As an alternative to the thermal process, the use of naturally derived materials has been investigated [3]. The use of *Luffa* sponge (unspecified species) as a bioadsorbent is reported in the literature. A sponge coated with beeswax and palm wax exhibited excellent adsorptive properties for 5 µm MPs and oil. The use of vegetable sponge offers several advantages, including biodegradability and a three-dimensional structure with good mechanical resistance, elasticity, and robustness, thereby reducing production costs and facilitating regeneration when used as an adsorbent [29]. The adsorption capacity ranged from 381 to 569 mg of PS MPs per g of bioadsorbent after 30 min of agitation. The positive aspect of this article is the development of a bioadsorbent using biodegradable, low-impact (with minimal environmental impact) plant-based raw materials (composition: luffa, beeswax, and carnauba wax) and scalable materials. However, there is still a limitation: the solvent used (xylene), which is extremely toxic for wax coating and ethanol cleaning for reuse in MPs, and the long contact time, which would be impractical in large water treatment plants [3].

In this study, *L. cylindrica* sponges were impregnated with micrometric natural magnetite to obtain a magnetic adsorbent biocomposite, following the principles of green chemistry and sustainability, using a simple, low-step process with low operating cost at temperatures below 80 °C. The biocomposite was evaluated for the adsorption of PS and PET MPs ranging from 600 to 75 µm in three simulated aquatic environments: drinking water (AGF), seawater (AGS), and water collected in an aquatic environment (AGP) in Dourados, Mato Grosso do Sul.

2. Materials and Methods

Dichloromethane (Synth) and Sodium Hydroxide PA (Synth, São Paulo, Brazil) were used without further purification. Sea salt was purchased from Ocean Tech Reef Active. Phenol (VETEC, Rio de Janeiro, Brazil) was distilled using simple distillation before use. Natural Magnetite was obtained from Sirius Minerals (São Paulo, Brazil).

Simulated seawater (AGS) was prepared as described by Zeron et al. [30]. In a Erlenmeyer flask were dissolved 35 g of NaCl (Ocean Tech brand, Reef Active, São Paulo, Brazil; characteristics: pH 8.3, KH 8~9, Na⁺ 9300~9700 mg L⁻¹, Mg²⁺ 1400~1450 mg L⁻¹, K²⁺ 260~410 mg L⁻¹, Ca²⁺ 430~450 mg L⁻¹, Sr²⁺ 9.0~10.5 mg L⁻¹, Rb²⁺ 0.11~0.13 mg L⁻¹, Fe 0.06~0.2 mg L⁻¹ e Li²⁺ 0.14~0.16 mg L⁻¹) in 1000 mL of the distilled water.

Lake water samples (AGP) were collected between 7:00 and 9:00 a.m. Sampling was conducted following a minimum 72 h dry period after the last rainfall. The water was kept refrigerated at 20 °C until use, with daily collections performed for the experiments.

2.1. Collection and Preparation of *Luffa cylindrica* (LC) Samples

The fruits of *Luffa cylindrica*, popularly known as loofah, were obtained from a rural property in the Dourados/MS region, Brazil. The fruits were peeled and the seeds removed. The research was cataloged in the SISGEN (number: AE1B402). Then, the fruits were washed with tap water and distilled water and dried at room temperature. Part of the sample of clean, dried fruits was ground in a Willey-type knife mill, model S-31, and sieved through a steel sieve with a diameter of 8 cm and a pore size of 4750 mm. Part of the sample was used without grinding.

2.2. Preparation of LC HCl and LC NaOH Bioadsorbents

Fiber samples of approximately 4 mm (1.0 g) were immersed in 25.0 mL of 0.1 mol L⁻¹ hydrochloric acid (HCl, sample designated LC HCl) or 0.1 mol L⁻¹ sodium hydroxide (NaOH, sample designated LC NaOH) solutions for 30 min. The samples were then removed from the solutions and dried in an oven at 70 °C for 30 min. The procedure was performed in triplicate for both LC NaOH and LC HCl.

2.3. Preparation of LCMAG IN, LCMAG HCl, and LCMAG NaOH Magnetic Biocomposites

Three biocomposites were prepared using *Luffa cylindrica* (LC) and magnetite (MAG): (1) LC and MAG biocomposite without prior treatment (LCMAG IN), (2) LC HCl and MAG biocomposite (LCMAG HCl), and (3) LC NaOH and MAG biocomposite (LCMAG NaOH). In 250 mL Erlenmeyer flasks, 5.0 g of LC IN, 5.0 g of MAG (Fe₃O₄), and 100 mL of deionized water were added. The samples were heated on a hot plate at 80 °C with mechanical stirring until the solvent had completely evaporated. After cooling, the samples were washed with distilled water to remove unincorporated magnetite, using a Büchner funnel and qualitative filter paper in a vacuum filtration system. Subsequently, the cleaned samples were dried in Petri dishes at 80 °C for 50 min. Then, the samples were placed in a desiccator containing silica under vacuum. The LCMAG NaOH and LCMAG HCl biocomposites were prepared using the same procedure described above, with LC IN substituted for LC NaOH or LC HCl. The procedure was performed in triplicate for each sample (LCMAG IN, LCMAG NaOH, and LCMAG HCl).

2.4. Preparation of PS and PET MPs

The polystyrene (PS) and polyethylene terephthalate (PET) plastic packaging was collected at Antenor Martins Park in the municipality of Dourados-MS, on 7 May 2023, at 4:04 p.m., with a recorded temperature of 20 °C, during the winter season. A second collection was carried out on 4 September 2025, at 1:35 p.m., also in winter; the characteristic Köppen climate classification is Cwa (humid mesothermal climate, hot summers and dry winters) [31]. The map (Figure S1) shows the collection points. Plastics in different states of degradation were identified using industry-standard symbols and transferred to a properly sanitized and identified glass container. Plastics in an advanced state of degradation, without industrial identification or with no visual identification, were discarded.

The collected packaging was washed with drinking water to remove soil and any adhered plant material. Then, the packaging was washed again with distilled water until all residues were removed, and it was dried at room temperature for 96 h. The properly classified, dry, and clean packaging was cut with scissors into sizes of 10 to 15 cm. Then, the PET and PS packaging was shredded in an industrial forage shredder. The polymers were sieved using a set of stainless-steel vibrating sieves with an 8 cm diameter and square mesh sizes ranging from 75 to 600 µm. The size distribution of PS fractions resulting were 600 µm (11.8%), 355 µm (55.9%), 150 µm (9.5%) and 75 µm (22.8%) and size distributions of the PET fractions were 600 µm (39.5%), 500 µm (12.3%), 355 µm (34%), 150 µm (11.6%) and 75 µm (2.6%).

2.5. Characterization of Biocomposites and Bioadsorbents

The X-ray Fluorescence (XRF) technique (Epsilon 3X X-ray spectrometer, Waltham, MA, USA) was used to determine the mineral oxide composition of the samples. Infrared spectroscopy (FTIR) was performed using a Shimadzu (IR Prestige-21, Kyoto, Japan) spectrophotometer equipped with an ATR accessory. FTIR spectra were obtained at room temperature, between 4000 and 300 cm⁻¹, 52 scans with a resolution of 2 cm⁻¹. Morphological characteristics of the samples were obtained using scanning electron

microscopy (SEM) with a Vega3-TESCAN microscope (20 kV, 15 mm, Warrendale, PA, USA) equipped with an integrated X-ray Dispersive Energy Spectrometer. Thermogravimetric analyses were performed using the DTG-60H equipment (Shimadzu, Kyoto, Japan). Approximately 5 mg of each sample was transferred to an alumina crucible and heated from 25 °C to 900 °C in a compressed-air atmosphere (flow rate 50 mL min⁻¹) at a heating rate of 10 °C min⁻¹. Zeta potential analyses (ζ) were recorded in triplicate at 25.0 °C for freshly prepared in AGF (Zetasizer Nano ZS equipment, Waltham, MA, USA).

Nitrogen adsorption/desorption analyses were performed using an Autosorb iQ (Quantachrome Instruments, Boynton Beach, FL, USA) at −196 °C, over the relative pressure range of 0.005–1.0, with samples previously degassed at 150 °C for 12 h under vacuum. The amount of material used was about 300 mg, and the analysis time was 8 h. The surface area of each material was estimated by the BET method (Brunauer–Emmett–Teller), and the pore size distribution was estimated by the BJH method (Barrett–Joyner–Halenda). Experimental data were acquired and processed using ASiQwin version 5.21 software.

The crystalline phases of these samples were determined by X-ray diffraction (XRD) analysis (Rigaku Geigerflex D/Max, C Series; Cu K α radiation; 2 θ angle range 20–70°; step 0.02 s⁻¹), with reference to standards compiled by the International Center for Diffraction Data. The experimental XRD data were compared with diffraction patterns from crystallographic structure databases. For this purpose, QualX 2 [32] was employed to identify the qualitative phase, using data from the Crystallography Open Database (COD) [33]. The software Nimloth v. 1.76 [34], a free and flexible program for X-ray diffraction calculations, was used to perform theoretical modeling for quantitative phase analysis of well-crystallized phases and peak-area analysis of the semicrystalline contributions associated with the partially ordered domains of the natural fiber. For the MAG sample, a conventional Rietveld refinement was performed to determine the weight fractions of the crystalline phases, as is typically required for natural multiphase materials. The background was modeled using a ninth-order Chebyshev polynomial. The peak profiles were described using a pseudo-Voigt function, with the peak width parameterized as a function of diffraction angle, following the formalism proposed by Caglioti et al. [35], to account for instrumental broadening. Standard intensity corrections appropriate for Bragg–Brentano geometry without a monochromator were applied. For the LC–NaOH and LCMAG–NaOH samples, the well-crystallized magnetite phase was refined using the same procedure described for the MAG sample. The broad reflections associated with the semicrystalline phase of the natural fiber were modeled using general peak functions. In this case, the peak profiles were described by pseudo-Voigt functions in which the full width at half maximum (FWHM) and the mixing parameter (η) were refined independently, aiming to obtain accurate peak positions and integrated areas for the semicrystalline contributions.

The crystallinity index (I_c) of the LC NaOH was calculated from the intensity of the peaks from the relation between crystalline and amorphous regions of the XRD curve (Equation (1)) [36]:

$$CI(\%) = \frac{I_{002} - I_{am}}{I_{002}} \times 100 \quad (1)$$

In Equation (1), CI represents the relative degree of crystallinity, I_{002} is the area of the maximum intensity (a.u.) of the (002) lattice diffraction between 21 and 24°, and the I_{am} is the amorphous diffraction area (a.u.) at 2 θ = 18°.

Biochemical Oxygen Demand was determined using the methodology described in the literature (<https://www.standardmethods.org/doi/epdf/10.2105/SMWW.2882.102>; <https://www.sidneyseckler.com/manuais>; accessed on 2 March 2026).

2.6. Determination of the Capture Capacity of the Biocomposites Using a Neodymium Magnet

The biocomposites formed by LC and MAG: LCMAG IN, LCMAG HCl, and LCMAG NaOH were evaluated for their ability to be captured in drinking water, using the magnetic force between the biocomposites containing magnetite and a neodymium magnet at 25 °C. The procedure was adapted from the literature [37]. In 500 mL glass test tubes, 400 mL of drinking water (pH 7.86) and 250 mg of LCMAG NaOH were added. The LCMAG NaOH sample was kept in contact with water for 5 min inside the test tube. At the designated time, the biocomposite was captured with a 35 mm diameter neodymium magnet attached to a string. The magnet was introduced into the test tube at a constant, uniform speed until it reached the bottom [37]. The biocomposites adhered to the magnet were collected and dried in an oven at 45 ± 1 °C for 24 h. The mass of the biocomposite was measured before immersion and after drying in the oven. The procedure was repeated for each biocomposite in triplicate.

2.7. Determination of the Sedimentation Rate of the Biocomposite and Bioadsorbents

The procedure for determining the sedimentation rate was adapted from the literature [38,39]. In 500 mL graduated cylinders, 400 mL of drinking water and 300 mg of LCMAG NaOH were added in triplicate. Then, the samples were observed at 1, 3, 5, 10, 15, 30, and 60 min by visually assessing the amount of sedimented biocomposite and the height of the supernatant in the graduated cylinder. The procedure was repeated for the bioadsorbents, LC NaOH (LC treated with 0.1 mol L^{-1} of NaOH) and LC IN (LC *in natura*), with modification of the separation process from the aqueous medium, where the LC NaOH and LC IN samples were separated from the water by filtration through voile fabric. The procedure was repeated for each biocomposite in triplicate.

2.8. Removal of PS and PET Microplastics from Water Using Magnetic Biocomposites

Short contact times (5, 10, and 15 min) were selected to evaluate the feasibility of rapid microplastic removal, aiming at potential applications in water treatment processes where short hydraulic retention times are desirable.

The procedure for determining the removal content of MPs was adapted from the literature [37]. In 500 mL glass test tubes, 400 mL of drinking water (pH 7.86), 50 mg of microplastics [mixture of PS or PET in the sizes of 75–600 μm], and 250 mg of LCMAG NaOH were added. The biocomposite and the MPs (PS or PET) were kept in contact in the test tube for 5, 10, or 15 min. After the established time, the biocomposite and the MPs adhering to it were captured using a 35 mm diameter neodymium magnet attached to a string [37]. The magnet was introduced into the test tube at a constant, uniform speed until it reached the bottom. The biocomposite + MPs adhered to the magnet (Figures S8 and S9) were collected and dried in an oven at 45 ± 1 °C for 24 h. Residual MPs present in the drinking water were filtered through voile fabric and dried in an oven at 45 °C for 24 h. The masses of the biocomposite and MPs before immersion, the biocomposite + MPs after oven drying, and the filtered and dried residue were weighed. The procedure was repeated for each biocomposite, time point, or MPs in triplicate. The same procedure was realized by AGS and AGP, with 10 min of contact between the MPs and LCMAG NaOH. The dried solid samples were used for gravimetric analysis, whereas the supernatant obtained after removal of the solid samples was analyzed by ultraviolet-visible (UV-Vis) spectrophotometry. The procedure was repeated for each biocomposite in triplicate.

The capacity of adsorption of the MPs by the biocomposite was indirectly determined by calculating the % MPs of the PS or PET in aqueous media using the equation:

$$\% \text{ MPs of the PS or PET in solution} = \frac{m}{M} \times 100\% \quad (2)$$

where m = the mass of MPs measured in aqueous media by gravimetry or UV/Vis, and M = the total mass measured (~50 mg per sample). A higher residue content in aqueous media indicates a low adsorption capacity.

2.9. Determination of the Removal Capacity of the PS or PET MPs on LC IN in Drinking Water

Washed and dried LC IN samples (without magnetite) were cut into approximately 23 mm lengths, weighed 250 mg, and fixed to a cotton thread. The procedure was performed in triplicate, as described by LCMAG NaOH experiments, except that the magnet was replaced with a cotton thread and a glass rod to ensure that the bioadsorbent moved up and down inside the test tube.

2.10. Determination of Water Absorption and Recovery Rate by the Neodymium Magnet of LCMAG NaOH

The water absorption behavior and magnetic recovery efficiency of the LCMAG NaOH biocomposite were evaluated to assess its physical stability and operational performance under aqueous conditions. Since the material is intended for use as a magnetic adsorbent, these properties are essential to determine its ability to withstand prolonged contact with water, maintain its structural integrity, and be efficiently recovered by an external magnetic field for subsequent reuse.

Briefly, 300 mg of LCMAG NaOH was dispersed in 400 mL of drinking water contained in 500 mL graduated cylinders. The suspensions were maintained under static conditions for 10, 20, 30, 40, and 50 min, and each experiment was performed in triplicate.

At the end of each immersion period, the biocomposite was recovered using a neodymium magnet by applying a single downward movement along the outer wall of the graduated cylinder. Immediately after magnetic separation, the wet mass of the recovered material was recorded to evaluate water uptake. The samples were subsequently dried in an oven at 45 °C for 24 h, and the dry mass was determined. The immersion–magnetic recovery–drying procedure was repeated for three consecutive cycles to investigate the effect of repeated use on both water absorption and magnetic recovery performance.

The magnetic recovery efficiency was calculated according to Equation (3):

$$\text{rate of recovery of the LCMAG NaOH} = \frac{w}{W} \times 100\% \quad (3)$$

where w is the dry mass of LCMAG NaOH recovered after immersion and oven drying, and W is the initial dry mass of the biocomposite before immersion.

Water absorption was determined from the difference between the wet and dry masses of the recovered material.

2.11. Determination of PS and PET MPs Content by Ultraviolet and Visible (UV/Vis) Spectroscopy

The non-adsorbed PS and PET MPs residues in aqueous medium (AGP, AGS, and AGF) were filtered through voile fabric, dried in an oven at 45 °C, and stored in glass vials previously cleaned with dichloromethane (PS residue samples) or a solution of dichloromethane and phenol (PET residue samples).

The PS MPs residues were dissolved in 20 mL of dichloromethane and then analyzed by UV/Vis in 3.0 mL quartz cuvettes. UV/Vis measurements were performed on a Shimadzu scanning spectrophotometer (UV-2600), in the range of 190 to 600 nm. The quantification of PS MPs residue was performed at 260 nm using standard PS curves constructed over concentrations ranging from 0.50 to 1.5 mg mL^{−1}, corresponding to the PS samples used in the adsorption experiments. The least squares method was used, and

a correlation coefficient (R) above 0.99 was considered acceptable ($y = 0.153(\pm 0.09645) + 1.954(\pm 0.0995)$, $R^2 = 0.992$).

The PET MPs residues were heated with 1.0 g of previously distilled phenol until complete solubilization (at a temperature below 100 °C), then diluted with 20 mL of dichloromethane and analyzed by UV/Vis in 3.0 mL quartz cuvettes. UV/Vis measurements were performed on a Shimadzu scanning spectrophotometer (UV-2600) over the range 190–500 nm. The quantification of PET MPs was performed at 310 nm using standard PET curves constructed over concentrations ranging from 0.68 to 4.48 mg mL⁻¹ of the PET samples used in the adsorption experiments. The least squares method was used, and a correlation coefficient (R) above 0.99 was considered acceptable ($y = 0.1952(\pm 0.04) + 0.87389(\pm 0.014)$, $R^2 = 0.999$).

3. Results and Discussions

The samples LC IN, LCMAG HCl, LCMAG NaOH, and MAG (Figure 1) were evaluated by FTIR to determine their characteristic functional groups and magnetite phase, and compared with those reported in the literature. The broadband at 567 cm⁻¹ confirms the presence of the Fe-O bond overpositions of the magnetite and hematite phases in MAG [24,40,41]. The same band is observed in the LCMAG HCl and LCMAG NaOH samples, indicating the stability of the magnetite phase in the biocomposites, which can be attributed to its micrometric size and natural origin, thereby increasing stability [23–25]. The bands at 1440 and 874 cm⁻¹ are typical of the asymmetric stretching and out-of-plane bending, respectively, of carbonate ions in calcite and dolomite [42,43]. The weak band attributed to quartz is visible at 465 cm⁻¹. The presence of albite is indicated by bands in the 1300–400 cm⁻¹ range. The two bands at 480 cm⁻¹ are due to the bending vibration of Si–O–Si and the coupling between the O–Si–O deformation [44,45]. The band at 805 cm⁻¹ is attributed to Si–O–Si bending vibrations, and the band at 1090 cm⁻¹ is characteristic of the Si–O–M symmetric bond stretching bands, characteristic of the presence of Na, K, Mg, Al, and Mg [46–51].

The FTIR spectra of the samples LC IN, LCMAG NaOH, and LCMAG HCl showed cellulose and pectin characteristic bands, with only small differences in their positions and intensities after the respective treatments, consistent with the literature. The 1200 and 950 cm⁻¹ regions are the fingerprint regions of carbohydrates of each polysaccharide [52,53]. The bands at 662 and 895 cm⁻¹ correspond to the OH out-of-plane bending and the β -glycosidic linkages, respectively. The band at 1025 cm⁻¹ is attributed to ring vibrations and C–OH bending. The band at 1150 cm⁻¹ is attributable to the C–O and C–O–C stretching. The C–H bending vibration is observed in the bands at 1237 and 1372 cm⁻¹. The band at 1320 cm⁻¹ is due to OH in-plane bending or CH bending. The band at 1421 cm⁻¹ is due to CH₂ bending. The characteristic band at 1646 cm⁻¹ is the –O– tensile vibration band adjacent to the H group. The absorption band at 1732 cm⁻¹ is attributed to the C=O stretching of the methyl ester (COOCH₃) and the undissociated carboxylic acid (COOH) groups in pectin. The band at 2917 cm⁻¹ is indicative of the C–H asymmetric and symmetric tensile vibration [54–58]. The FTIR of the LCMAG IN, PET, and PS samples is presented in the supplementary material. The FTIR spectra of these samples are consistent with those reported in the literature.

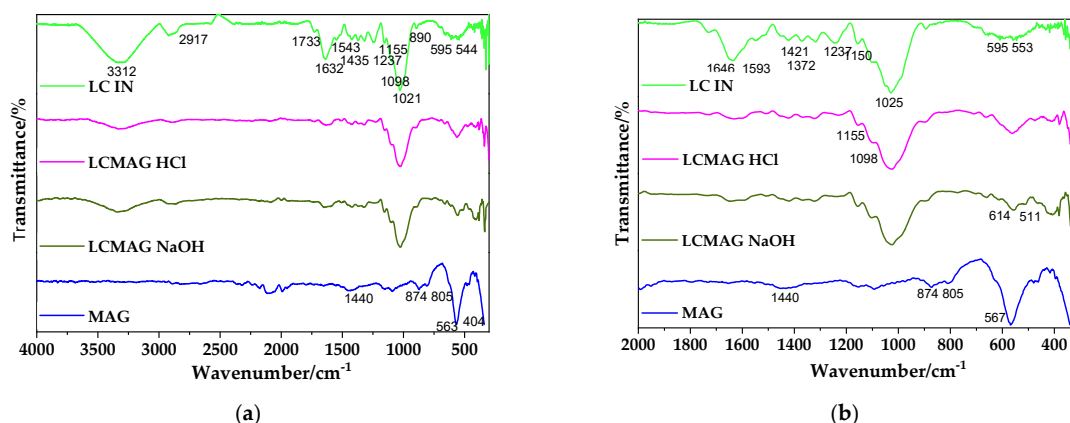


Figure 1. FTIR spectra of the samples: LC IN, LCMAG HCl, LCMAG NaOH, and MAG. (a) show the full spectra, while (b) present the magnified region (2000–400 cm^{−1}) of the same samples.

3.1. Determination of the Sedimentation Rate of the LCMAG NaOH Biocomposite and Bioadsorbents

Obtaining a low-density adsorbent system that remains suspended on the aqueous surface without complex, energy-intensive equipment can reduce the cost of adsorbing MPs. Therefore, the sedimentation times of LCMAG NaOH, LC NaOH, and LC IN were evaluated. The bioadsorbents LC IN and LC NaOH, and the LCMAG NaOH biocomposite, showed stability on the surface throughout the experiment, with small variations in initial sedimentation rates, followed by a return to the surface and floating over time. The low sedimentation rate (buoyancy of the samples) can be explained by the low density of the sponge LC, as reported in the literature, ranging from 385.46 to 468.70 kg m^{−3}. The incorporation of MAG (>5 kg m^{−3}) does not significantly affect the density of the biocomposite [59].

3.1.1. Determination of the Capture Capacity of Magnetic Biocomposites Using a Magnet and Characterization of the LCMAG NaOH

The pH of the measured drinking water (AGF) was 7.86, with a conductivity of 88.85 $\mu\text{S cm}^{-1}$ at 25 °C, whereas the simulated seawater had a pH of 8.3 and a conductivity of 41.63 mS cm^{−1}. The lake water from the park (AGP), due to its natural origin, was further characterized by dissolved oxygen ($2.31 \pm 0.15 \text{ mg L}^{-1}$), conductivity (149.5 $\mu\text{S cm}^{-1}$), and BOD₅ ($156 \pm 31 \text{ mg O}_2 \text{ L}^{-1}$), with a measured pH of 7.66.

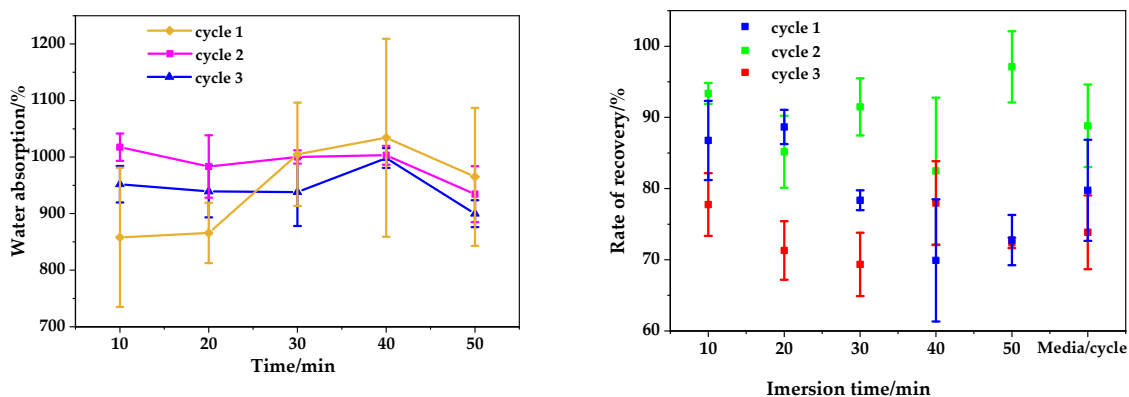
The biocomposites from LC and MAG: LCMAG IN, LCMAG HCl, and LCMAG NaOH were evaluated in relation to their capacity to be captured in drinking water using the magnetic force between the biocomposites and the neodymium magnet at 25 °C. The sample denominated LCMAG NaOH was captured from the aqueous medium with an efficiency exceeding 95%. The LCMAG IN and LCMAG HCl samples were captured with efficiencies of 10% and 50% (*w/w*), respectively. The LCMAG IN and LCMAG HCl biocomposites initially interacted with the magnet in water; however, when the magnet approached the surface, the samples did not maintain the magnetic attraction required for removal from the aqueous medium.

The low magnetic force observed in the LCMAG HCl sample could be attributed to magnetite instability in acidic media [60]. However, as observed in the FTIR (Figure 1), magnetite was not oxidized in the LCMAG HCl sample; therefore, this does not explain the magnet's low sample capture. The low magnetic interaction of the LCMAG IN and LCMAG HCl samples with the magnet may be associated with greater interactions

between magnetite and the sponge components, and between the sponge and water. The raw sponge (LC IN) is composed of cellulose (65.69%), hemicellulose (19%), and lignin (12.0%). Hemicellulose contains pentoses, hexoses, and uronic acids, which can interact strongly with magnetite and water through electrostatic interactions or hydrogen bonds. Treatment with sodium hydroxide solution reduces the concentration of hemicellulose (X-ray and SEM) and, therefore, can reduce the electrostatic interactions between magnetite and sponge, and the hydrogen bonds between the sponge and water, resulting in a greater interaction between the magnetite of LCMAG NaOH and the magnet, and, consequently, greater capture of this biocomposite. In a study on the influence of LC sponge immersion time in aqueous sodium hydroxide solutions ranging from 0 to 16% for up to 8 weeks, it was observed that low concentrations of sodium hydroxide (0.5% *w/w*, $\sim 0.1 \text{ mol L}^{-1}$) increased strength and toughness and reduced fiber density. The authors attributed the improvements in characteristics to bond breaking and the decomposition of lignin and hemicellulose. The reduction in density may be associated with the release of inorganic salts from the fiber, which can reduce the electrostatic interactions between water and the sponge, and between magnetite and the sponge [61–63]. Cellulose is still classified as diamagnetic. Diamagnetic materials are characterized by having all their electrons paired and, in the presence of an external magnetic field, these electrons align in the opposite direction to the field, reducing the field's magnitude [64].

3.1.2. Determination of the Water Absorption Capacity and Recovery Rate by the Neodymium Magnet of the LCMAG NaOH

Another relevant factor for an efficient hydrophilic bioadsorbent is its water-absorption capacity. The potential uses of LCMAG NaOH as a floating adsorbent biocomposite for capturing MPs are limited by the rate and capacity of water adsorption. The ideal system involves hydrophobic bioadsorbents that reduce the volume of water removed from the medium during the removal of adsorbents + adsorbed MPs [3]. However, obtaining this property requires energy-intensive thermal processes, such as biochar formation or the use of toxic organic solvents. Therefore, in this work, hydrophobicity was not considered a limiting factor. But to ensure the efficiency of the bioadsorbent, the water absorption capacity was evaluated to enable potentially large-scale application, determine the final mass to be collected, and establish the conditions and methodologies for scalable application. The rate of drinking water absorption, measured over 50 min in three cycles (Figure 2a), reached up to 1050% of the initial mass of LCMAG NaOH. The high amount of absorbed water, however, did not alter the biocomposite's fluctuation. The capacity of the floating can be explained by the sponge LC's low density (discussion above).



(a) (b)

Figure 2. (a) Determination of the water absorption capacity of LCMAG NaOH versus contact time. Measurements determined in triplicate, for three 50 min cycles, with drying at 45 °C for 24 h after each immersion in potable water; (b) determination of the recovery rates of the LCMAG NaOH versus immersion time. Measurements determined in triplicate, for three cycles of 50 min, with drying at 45 °C for 24 h after each immersion in drinking water.

In the first cycle, it was observed (Figure 2a) that the 10 and 20 min times had an approximately constant average water absorption rate, with an increase in the average absorption rate in the first 30 min, followed by maintenance of this average rate at 40 and 50 min, indicating a saturation of the biocomposite in relation to the water absorption capacity. The average absorption deviations observed in this first cycle were greater than those observed in cycles 2 and 3. This result can be explained by a lower recovery rate of LCMAG NaOH (Figure 2b) compared with the second and third cycles, and consequently, a greater experimental error, as evidenced by variation in water absorption capacity. In the second cycle, the average water adsorption rate remained approximately constant at 10, 20, 30, and 40 min, with a small reduction at 50 min. However, the average water absorption rates were generally similar to or higher than those of the first cycle. In the third cycle, the average water absorption rate remained approximately constant, with intermediate values between the first and second cycles at 10, 20, 30, and 40 min.

The recovery rate (Figure 2b) evaluated the neodymium magnet's capacity to capture LCMAG NaOH as a function of biocomposite exposure time in aqueous media (AGF). The 10 min time showed a higher recovery rate across three cycles (immersion, dry, and gravimetric measurements). The higher recovery rate in cycle 2 can be attributed to the biocomposite, which exhibits greater homogeneity in magnetite distribution than in cycle 1. The increase in the percent loss in cycle 3 is attributed to a reduction in magnetite adsorbed in the LC.

3.2. Characterization of LCMAG NaOH

As the LCMAG NaOH sample exhibited the highest magnetic recovery efficiency among the synthesized biocomposites, it was selected for further characterization and adsorption studies. LCMAG NaOH and its precursors (LC NaOH and MAG) were characterized using XRF, SEM, SEM-EDX, TGA, and XRD (Figures 2 and 3). Subsequently, LCMAG NaOH was employed in the adsorption experiments with PS and PET microplastics (MPs).

The elemental composition of the MAG, LCMAG NaOH, and LC NaOH, determined by XRF, is shown in the Supplementary Material (Table S1). The MAG sample is predominantly composed of iron, corresponding to 79 wt.% as iron oxide, with less than 6 wt.% of oxides of other metals typically found in natural materials, such as calcium, sodium, and magnesium. In contrast, the metallic composition of the LC NaOH sample comprises sodium, potassium, and calcium oxides as the major components (approximately 20 wt.% each), with iron oxide accounting for 9 wt.%. The LCMAG NaOH sample, corresponding to the impregnated natural fiber, exhibits iron as the predominant element (80 wt.% as iron oxide) in the metallic components. A significant decrease in the content of other metallic elements, such as calcium, sodium, and potassium, is observed. Compounds containing these elements generally exhibit relative solubility in water and may have been partially leached during the impregnation process.

The SEM and SEM-EDX images of the MAG, LC NaOH, and LCMAG NaOH are displayed in Figures 3, 4, and 5, respectively. The morphological features of the MAG (Figure 3) and LC NaOH (Figure 4) are characteristic of the samples. The irregular surface of the MAG is derived from grinding natural rocks. The particle size of the MAG sample

used in the impregnation was measured using the sieve and ranged from 30 to 150 μm . SEM-EDX elemental maps of the three samples confirm the elements identified in the XRF, with excellent distribution of all the biocomposite LCMAG NaOH (Figure 5) and absence of particles with a size larger than 150 μm .

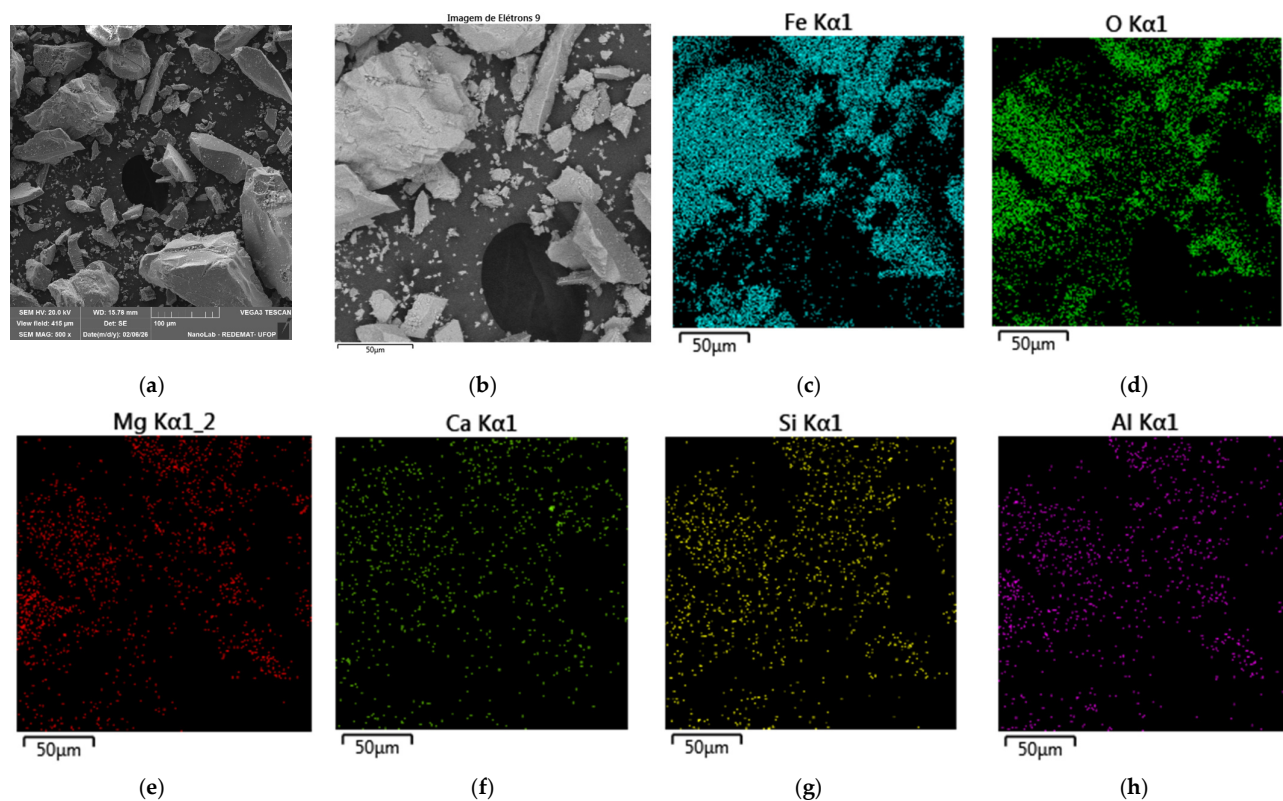
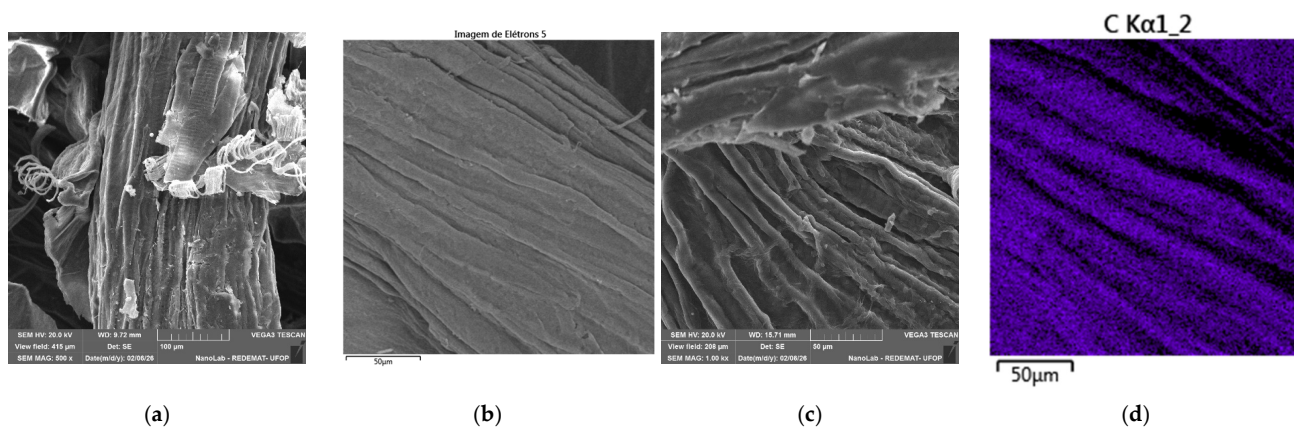


Figure 3. SEM (a,b) and EDX elemental mapping images (c–h) of MAG.



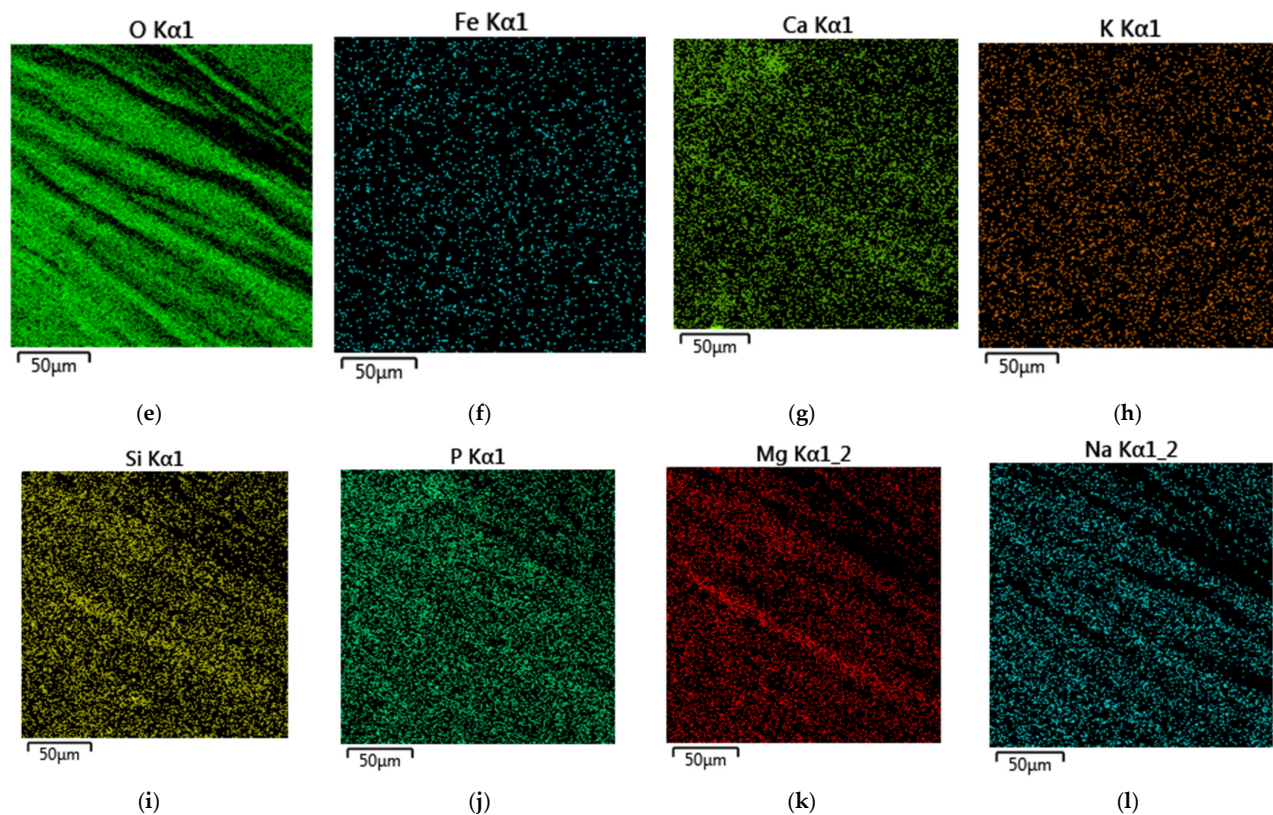


Figure 4. SEM (a–c) and EDX elemental mapping images (d–l) of LC NaOH.

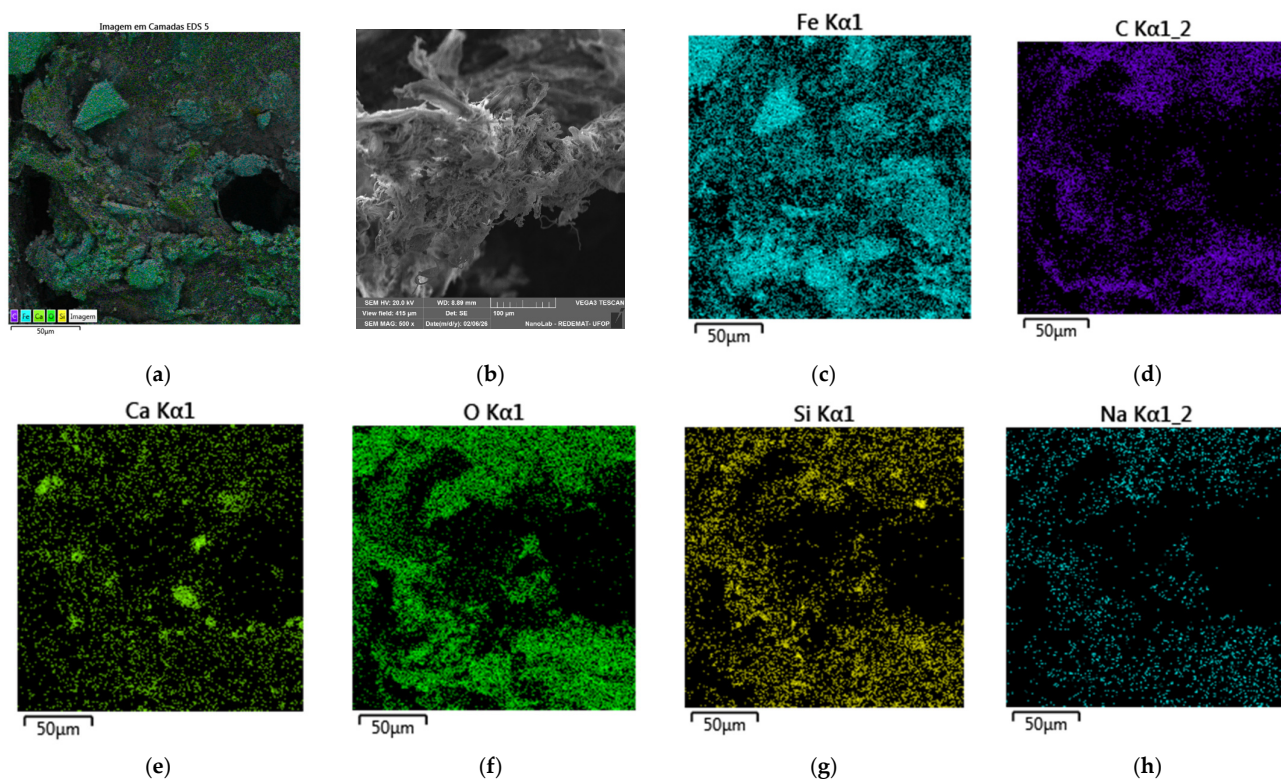
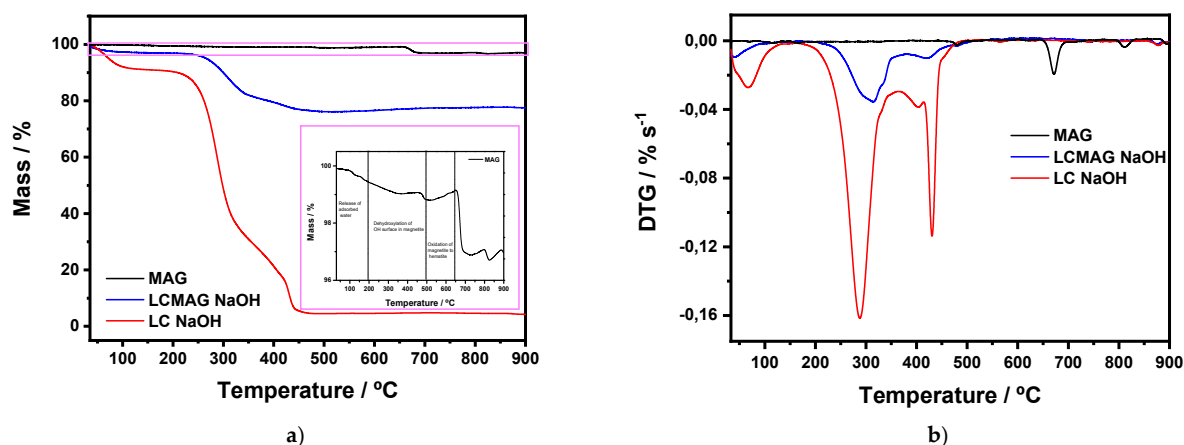


Figure 5. SEM (a,b) and EDX elemental mapping images (c–h) of LCMAG NaOH.

The TGA and DTG curves of MAG, LC NaOH, and LCMAG NaOH were used to evaluate their thermal stability and to determine the percentage of residual inorganic content (at 900 °C) in the samples. The thermogram of MAG (highlighted in Figure 6) shows an initial mass loss of 0.27% (*w/w*) between 25 °C and 150 °C, attributed to the release of adsorbed water and volatile components. This interpretation is consistent with the FTIR spectrum of MAG (Figure 4a,b), which does not exhibit noticeable absorption bands in the OH stretching region ($\sim 3400\text{ cm}^{-1}$), indicating that these species are present only in very small amounts. Between 200 and 500 °C, a small mass-loss event occurs, corresponding to 0.69% of the initial mass, which is associated with the dehydroxylation of surface OH groups of magnetite. In the range of 500–650 °C, a mass gain is observed, attributed to the oxidation of magnetite (Fe_3O_4) to hematite (Fe_2O_3) [9,23,25]. Above 650 °C, additional mass-loss and mass-gain events are detected, which are attributed to the decarbonation of the dolomite and calcite phases present in the sample, as previously reported by Zhu et al. [65]. These findings are also consistent with the results obtained from XRF and XRD analyses (Supplementary Material and Figure 6c–e).

The LC NaOH sample exhibits three main mass-loss events. The first event, occurring between 30 and 150 °C, corresponds to the removal of physically adsorbed water from the material's surface. The second mass-loss event, between 155 and 360 °C, is associated with the thermal degradation of hemicellulose and the cleavage of glycosidic bonds in cellulose, with a maximum decomposition rate at 290 °C [66]. The third event, observed between 365 and 463 °C, with a maximum rate at 428 °C, is attributed to the thermal degradation of lignin fractions present in the biomass [67].

The LCMAG NaOH biocomposite exhibits a mass-loss profile similar to that of LC NaOH during biomass degradation. However, the biocomposite exhibits higher thermal stability, as evidenced by a shift in the maximum decomposition temperature toward higher values. This behavior indicates that incorporating magnetite improves the thermal stability of the biocomposite matrix. Additionally, a mass gain above 600 °C is observed, corresponding to the oxidation of magnetite to hematite. At the end of the thermal process under an air atmosphere, the residual mass obtained for LCMAG NaOH was 77% wt., indicating a high inorganic content in the composite material, which is corroborated by the results observed in the XRF (Supplementary Material, Figure S10).



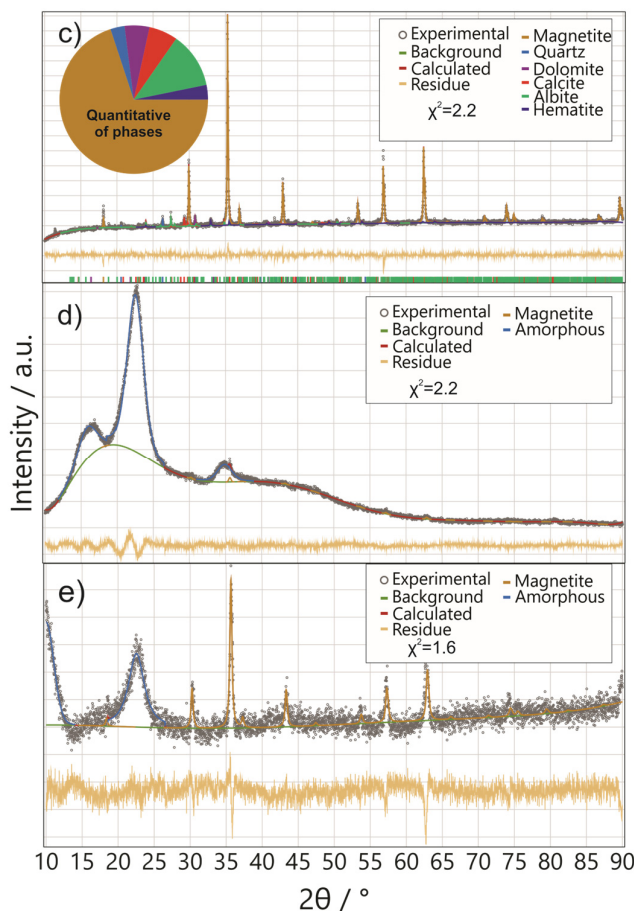


Figure 6. TG (a) and DTG (b) of the LC NaOH, MAG, and LCMAG NaOH; experimental and theoretical diffractograms obtained from *Rietveld* refinement of (c) MAG, (d) LC NaOH, and (e) LCMAG NaOH samples.

The specific surface area, pore diameter, and pore volume of the MAG, LC IN, LCMAG NaOH, and LC NaOH were determined by N_2 adsorption–desorption at 77 K using the BET method (Figure 7, Table 1). Alkali treatment increased the specific surface area of LC NaOH to approximately three times that of LC IN. The specific surface area of LCMAG NaOH was intermediate between those of LC IN and LC NaOH, indicating that magnetite incorporation partially preserved the increase in surface area promoted by the alkaline treatment. The specific surface area, pore diameter, and pore volume obtained for LC IN and LC NaOH are consistent with values reported in the literature [68,69]. Despite the relatively low BET surface area observed after alkaline treatment, magnetite incorporation may improve the accessibility of adsorption sites. The specific surface area of LC IN is comparable to that reported in the literature [68], confirming the reproducibility of the alkali-treated lignocellulosic precursor.

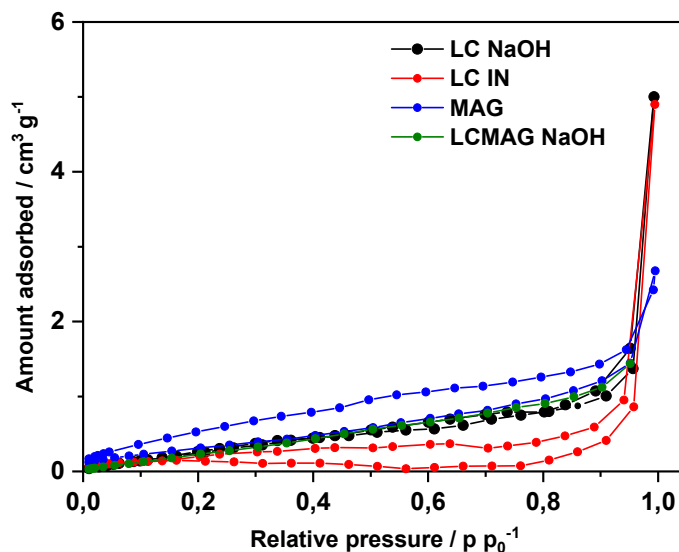


Figure 7. Nitrogen adsorption–desorption isotherms at 77 K of LC IN, LC NaOH, MAG, and LCMAG NaOH.

Table 1. BET specific surface area, average pore diameter, and total pore volume of LC IN, LC NaOH, MAG, and LCMAG NaOH.

Sample	$S_{\text{BET}}/\text{m}^2 \text{ g}^{-1}$	$D_p^a/\text{cm}^3 \text{ nm}^{-1} \text{ g}^{-1}$	$V_{\text{tp}}^b/\text{cm}^3 \text{ g}^{-1}$
LC NAOH	1.436	3.132	0.002
MAG	1.317	3.082	0.002
LC IN	0.468	4.262	0.008
LCMAG NaOH	1.174	3.442	0.002

Where ^a—obtained by the BJH method, ^b—total pore volume obtained at $p/p_0 = 0.95$.

The XRD patterns of sample MAG, LC NaOH, and LCMAG NaOH are presented in Figure 6c–e. The magnetite phase (Fe_3O_4) was identified in sample MAG. The most intense diffraction peaks were observed at 2θ angles of 18.14° , 30.11° , 35.47° , 37.10° , 43.10° , 53.48° , 57.01° , 62.60° , and 73.86° , corresponding to the (111), (220), (311), (222), (400), (422), (551), (440), and (620) crystallographic planes, respectively, in good agreement with the reference XRD data for magnetite (COD 1011032). Other minor peaks are attributed to minor phases: albite ($\text{NaAlSi}_3\text{O}_8$ —COD 2107372), calcite (CaCO_3 —2106394), dolomite ($\text{CaMg}(\text{CO}_3)_2$ —COD 1200014), hematite (Fe_2O_3 —COD 1546384), and quartz (SiO_2 —COD 1535066). For the LC NaOH (*Luffa cylindrica* fiber) sample, the X-ray diffraction pattern exhibits characteristic reflections in the 2θ range of 22 – 23° , which are associated with the (002) plane of cellulose I and contributions from cellulose II, indicating the coexistence of crystalline cellulose domains. In addition, a minimum in intensity is observed at $2\theta \approx 18.22^\circ$, which is attributed to the amorphous fraction of cellulose, in accordance with previous reports [70,71]. A minor peak at approximately $36^\circ(2\theta)$ was observed and tentatively attributed to magnetite (COD 1011032), which may have formed during the preparation of the bioadsorbents under alkaline conditions (NaOH). The X-ray diffraction pattern of the LCMAG NaOH sample reveals the coexistence of amorphous and crystalline features inherited from the LC NaOH sample, along with distinct diffraction peaks attributed to the magnetite phase present in the MAG sample (COD 1011032). This combination confirms the successful integration of the organic matrix with the magnetic

inorganic component. The LC NaOH sample exhibits three well-defined diffraction peaks associated with the semicrystalline phase at $2\theta = 15.79^\circ$, 22.49° , and 34.84° . However, in the LCMAG NaOH sample, only the reflection at $2\theta = 22.50^\circ$ remains, while the other two peaks disappear.

The crystallinity index of LC NaOH (56.93%) was higher than that of LCMAG NaOH (22.37%). However, this reduction should be interpreted with caution. The Segal method was originally developed for cellulose and assumes that the diffraction pattern arises exclusively from crystalline and amorphous cellulose domains. In the LCMAG NaOH composite, the incorporation of crystalline magnetite introduces additional diffraction peaks that modify the overall diffraction profile and influence the relative intensity of the cellulose reflections used in the calculation. Therefore, the lower crystallinity index obtained for LCMAG NaOH most likely reflects the combined effect of structural changes in the cellulose after processing and the contribution of the crystalline magnetite phase, rather than a direct increase in the amorphous cellulose content. Martínez-Pavetti et al. reported a decrease in the crystallinity of *L. cylindrica* fibers after alkaline treatment with sodium hydroxide, which was attributed to partial disruption of the ordered cellulose domains [72–74]. To gain deeper insight into the structural organization of the samples, Rietveld refinement was carried out using Nimloth software [34]. For all samples, the diffraction profiles were modeled using a pseudo-Voigt function. For the MAG sample (Figure 6c), the XRD pattern was refined with six crystalline phases to account for all reflections observed in the experimental diffractogram. (Fe_3O_4) was identified as the major phase, comprising 70(2) wt.%, followed by ($\text{NaAlSi}_3\text{O}_8$) with 12(1) wt.%. Minor phases included (CaCO_3 , COD 2106394) at 6(3) wt.%, ($\text{CaMg}(\text{CO}_3)_2$, COD 1200014) at 5(3) wt.%, (Fe_2O_3 , COD 1546384) at 1.6(1) wt.%, and (SiO_2 , COD 1535066) at 3(2) wt.%. The diffraction peaks corresponding to each phase in this sample are shown in Figure 6c. For the LC–NaOH sample (Figure 5d), three broad reflections were employed to model the semicrystalline phase of the natural fiber. In addition, the magnetite phase was included to account for the sharp and well-defined peak at $2\theta = 35.53^\circ$, corresponding to the main (311) reflection of magnetite. For the LCMAG–NaOH sample (Figure 5e), the refinement was carried out using two broad reflections to represent the semicrystalline phase, together with the magnetite phase, which is the dominant phase in the MAG sample. The contributions of the remaining crystalline phases are negligible in this sample and are not distinguishable in the diffractogram.

The zeta potential of the LCMAG NaOH, LC NaOH, LC IN, MAG, PS, and PET fractions is presented in Figure 8. The zeta potentials of the bioadsorbents, PS, and PET (Figure 8a) were negative, with the magnitude (negative) increasing from pH 2 to pH 10. The zeta potential of the bioadsorbents (Figure 8b) was -2.0 mV to -18.8 mV, and the PS and PET were -0.66 mV and -15 mV, respectively, over the pH range of 2 to 10. All materials exhibited increasingly negative zeta potential values as pH increased, indicating progressive deprotonation of surface functional groups. This behavior is characteristic of pH-dependent surface charging and is consistent with the acid–base properties of lignocellulosic materials, magnetite, and polymeric microplastics. The zeta potential values observed for PS, PET, and MAG were consistent with the literature [3,9,40,41,75,76].

Because both the adsorbents and the MPs exhibited negative zeta potentials, electrostatic attraction is not expected to be the dominant adsorption mechanism under the investigated conditions. Therefore, the experimentally observed adsorption is likely governed by other interactions, such as hydrophobic interactions, hydrogen bonding, van der Waals forces, and surface heterogeneity [3,41]. The relatively low absolute zeta potential values indicate weak electrostatic repulsion between the adsorbent surface and the MPs [9,40,41]. The low variation in PS and PET zeta potentials with size is also

reported in the literature by Viet Ha et al., Savaji et al., and Mummaleti et al. [3,76,77]. The pH-dependent trend observed for PS in the present work is consistent with that reported by Viet Ha et al., with increasingly negative zeta potential values as the pH increases [3]. Small differences in the absolute values reported in the literature are expected because zeta potential depends strongly on experimental conditions (solvent, ionic strength, and pH) [78,79].

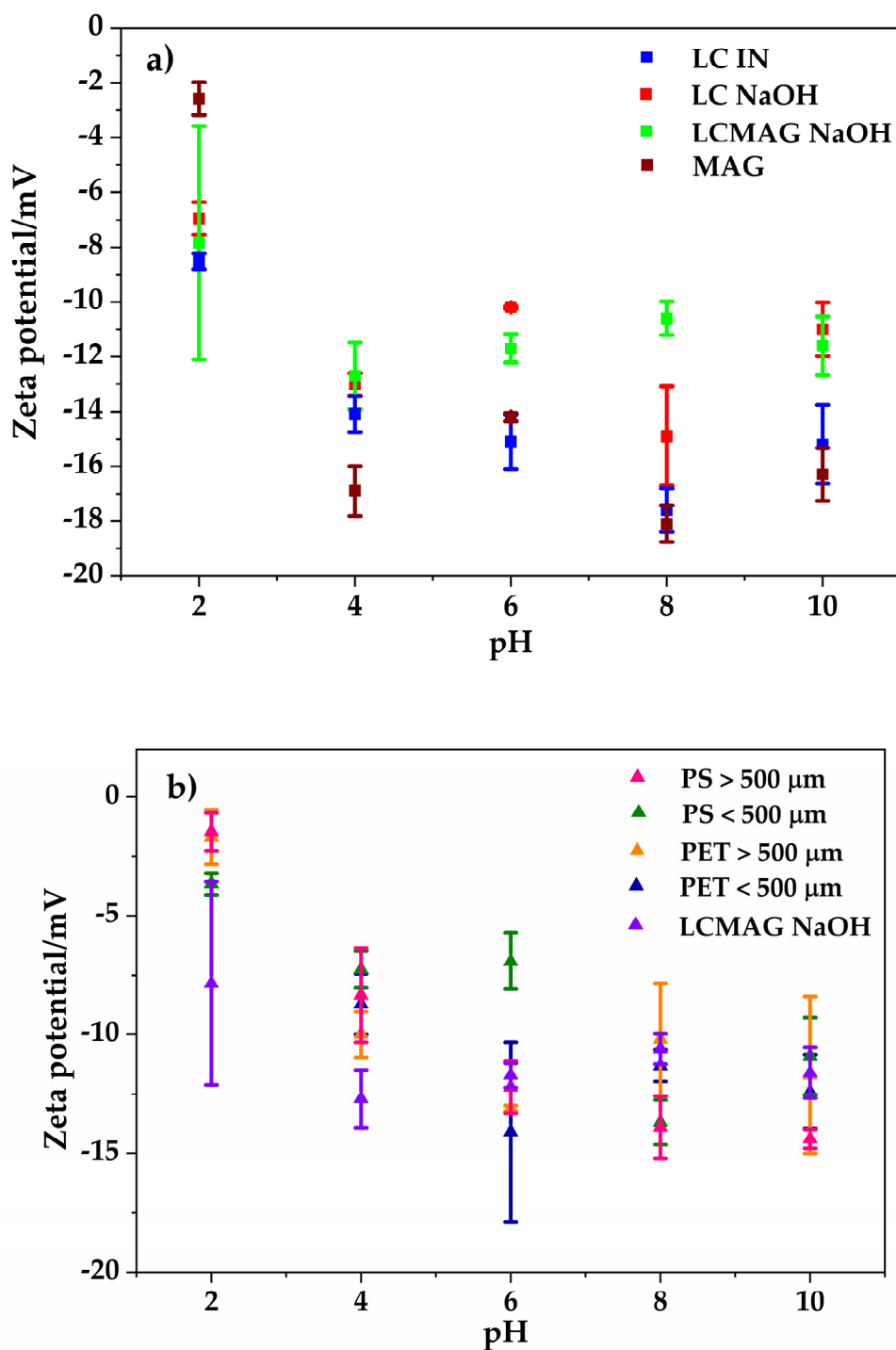


Figure 8. Zeta potential of the (a) LC IN, LC NaOH, LCMAG NaOH and MAG, and (b) MPs fractions, as a function of pH.

3.3. Determination of the Removal Efficiency of PS MPs on LCMAG NaOH in Drinking Water (AGF)

The removal capacity of PS MPs on LCMAG NaOH, LC NaOH, and LC IN was evaluated as a function of time, using the residual MPs content in the solution (Figure 9a), where lower residual content corresponded to greater adsorption capacity. The levels of residual MPs (not captured by the biocomposite) were determined by gravimetry and Ultraviolet-Visible Spectroscopy (UV/Vis) in triplicate. The lowest MPs content in solution was obtained for a contact time of 10 min between the biocomposite and PS MPs, with values of $18.4 \pm 0.5\%$ (*w/w*) and $38.5 \pm 0.89\%$ (*w/w*) by UV/Vis and gravimetry, respectively. The UV/Vis technique showed less dispersion, likely because it enabled differentiation of the PS residues from those of the adsorbents by solubilizing the PS residues in dichloromethane. In the gravimetric technique, the adsorbents not captured by the magnet were filtered and quantified along with the MPs. This theory corroborates the LCMAG NaOH recovery levels determined by gravimetry (93.9 ± 1.1 ; 92.8 ± 0.7 ; $93.9 \pm 1.4\%$ (*w/w*) for 5, 10, and 15 min, respectively), indicating that the bioadsorbent not captured from the aqueous medium was quantified.

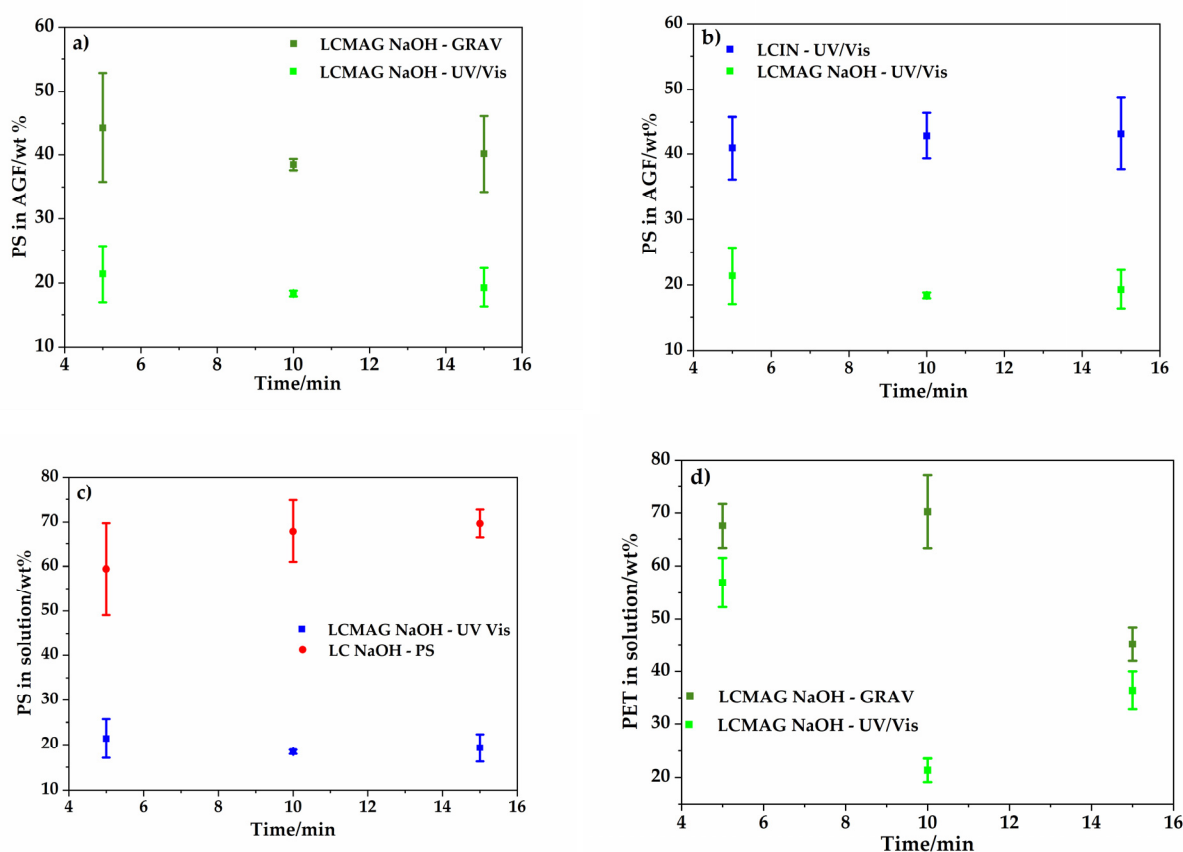
The experimental concentrations used in the present study (0.125 mg mL^{-1} of PS MPs and 0.625 mg mL^{-1} of LCMAG NaOH) are comparable to those reported by Babalar et al., who evaluated activated biochar and polymer-coated magnetic activated biochar-zeolite (PMACZ) composites using 0.1 mg L^{-1} of PS and 1 mg mL^{-1} of bioadsorbent in distilled water. PMACZ is prepared via multiple synthesis steps using activated biochar, magnetite, oleic acid, zeolite, polyethylene glycol (PEG), and polyethyleneimine, with processing temperatures up to 850°C . The modified composite exhibited a zeta potential of approximately +40 mV in the pH range of 6.0–8.0, whereas the 2 and $15 \mu\text{m}$ PS MPs presented zeta potentials of approximately −15 and −25 mV, respectively, within the same pH range (values estimated from the authors' graphs). Under these conditions, the opposite surface charges favor electrostatic attraction, which likely contributes to the approximately 70% adsorption achieved within 30 min [9].

The same study also evaluated activated carbon (AC), the precursor material used to prepare PMACZ. Activated carbon exhibited approximately 60% adsorption under the same experimental conditions despite presenting a zeta potential of approximately −40 mV [9], whereas the PS MPs also exhibited negative zeta potentials. This observation indicates that electrostatic attraction alone cannot explain the adsorption process. Instead, additional mechanisms, including hydrophobic interactions, van Der Waals forces, surface heterogeneity, pore entrapment, and possible ion-mediated interactions, are likely involved in MP adsorption. The negative zeta potential trend reported for AC and PS is also consistent with that observed for LCMAG NaOH and PS MPs in the present work, although the latter exhibited lower absolute zeta potential values.

From a practical standpoint, the approximately 10% increase in adsorption observed after the complex modification of activated carbon to produce PMACZ should also be interpreted in light of the additional synthesis steps, higher processing temperatures, and greater production costs. Therefore, the relatively modest gain in adsorption efficiency should be weighed against the increased process complexity in large-scale applications.

The use of *Luffa* sponge fruits coated with beeswax or palm wax has also been reported for microplastic removal. These coatings increase the hydrophobicity of the bioadsorbent surface, enhancing its affinity toward hydrophobic contaminants. Adsorption capacities of $381\text{--}569 \text{ mg g}^{-1}$ have been reported for wax-coated sponges, corresponding to more than 99% removal of $5 \mu\text{m}$ PS MPs after 30 min under continuous

stirring. This adsorption capacity is higher than that obtained for LCMAG NaOH (163 mg g⁻¹ under static conditions). However, direct comparison between the two studies should be interpreted with caution because the experimental conditions differ substantially. In the literature, the PS MPs were dispersed in an unspecified emulsification medium and diluted in a 1:1 ethanol-water mixture. In contrast, the present work employed drinking water with dissolved ions and a broader MP size distribution, conditions that more closely resemble those of natural aquatic environments. Furthermore, the adsorption experiments with LCMAG NaOH were performed without agitation and with a contact time three times shorter than that reported for the wax-coated sponges. Since continuous stirring substantially increases particle collision frequency and mass transfer, it generally favors adsorption. Static conditions were intentionally selected in the present work to better simulate the potential application of floating bioadsorbents in large aquatic environments [80,81].



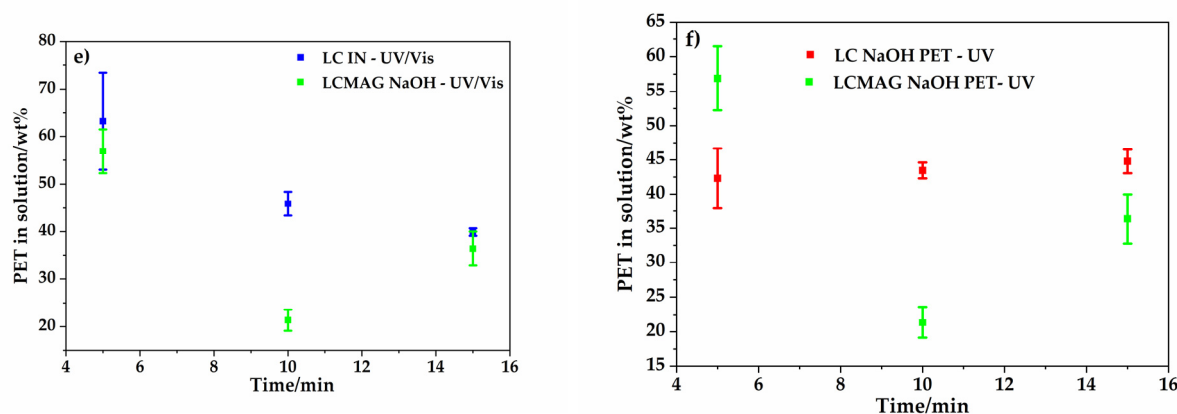


Figure 9. (a) Determination of residual PS MPs content versus time after LCMAG NaOH adsorption in drinking water (AGF) by gravimetry and spectroscopy in the ultraviolet and visible regions, after capture of the biocomposite + adsorbed PS, at times of 5, 10, and 15 min; (b) determination of residual PS content after LCMAG NaOH and LC IN adsorptions in drinking water (AGF) by ultraviolet-visible (UV/Vis) spectroscopy, after capture of the biocomposite or bioadsorbent + adsorbed PS, at times of 5, 10 and 15 min; (c) determination of residual PS content after LCMAG NaOH and LC NaOH adsorptions in drinking water (AGF) by ultraviolet-visible (UV/Vis) spectroscopy, after capture of the biocomposite or bioadsorbent + adsorbed PS, at times of 5, 10 and 15 min; (d) determination of residual PET MPs content in drinking water (AGF) by gravimetry and ultraviolet/visible spectroscopy (UV/Vis), after capture of the LCMAG NaOH + adsorbed PET, at 5, 10, and 15 min; (e) determination of residual PET content after LCMAG NaOH and LC IN adsorptions in drinking water (AGF) by ultraviolet-visible (UV/Vis) spectroscopy, after capture of the biocomposite or bioadsorbent + adsorbed PS, at 5, 10, and 15 min; (f) determination of residual PET content after LCMAG NaOH and LC NaOH adsorptions in drinking water (AGF) by ultraviolet-visible (UV/Vis) spectroscopy, after capture of the biocomposite or bioadsorbent + adsorbed PS, at 5, 10, and 15 min. GRAV—measurement performed by gravimetry; UV/Vis—measurement performed by spectroscopy in the ultraviolet and visible region. All measurements are performed in triplicate in drinking water (AGF) at 25 °C.

The lower absolute zeta potential of LCMAG NaOH led to a smaller difference in surface charge relative to the negatively charged MPs. Although zeta potential alone does not quantitatively determine interparticle interactions, this behavior suggests reduced electrostatic repulsion compared with LC IN and LC NaOH, which may facilitate particle approach and favor adsorption through additional interaction mechanisms. In the supplementary material of the same study, the authors reported the zeta potentials of the 5 μm PS MPs and wax-coated sponge bioadsorbents over the pH range of 2.0–10.0. Both materials exhibited a progressive decrease in zeta potential with increasing pH, reaching values below approximately +10 mV up to pH 5.0 (pH 6.0 for the bioadsorbent) and becoming negative above these values, approaching approximately −10 mV at pH 10. These results are consistent with those reported by Babalar et al. and with the trends observed in the present study, in which both the adsorbents and PS MPs exhibited predominantly negative zeta potentials under environmentally relevant pH conditions [9]. Although particles with similar surface charges are expected to experience electrostatic repulsion, the high adsorption efficiencies reported in these studies demonstrate that electrostatic attraction is unlikely to be the sole mechanism governing adsorption. Instead, non-electrostatic interactions, including hydrophobic interactions, van Der Waals forces, surface heterogeneity, and pore entrapment, probably play an important role in the adsorption process.

It should also be emphasized that direct comparison of zeta potential values across studies requires caution, as these measurements are strongly influenced by the dispersion medium, ionic strength, pH, and measurement protocol. As discussed by Sun et al. [75] and Honary et al. [78,79], zeta potential measurements are only directly comparable when performed under similar experimental conditions or in well-defined media, such as distilled water. Therefore, although zeta potential provides valuable information regarding particle surface properties, it should not be interpreted as the sole predictor of adsorption performance.

3.4. Removal Capacity of PET MPs on LCMAG NaOH, in Drinking Water (AGF)

The removal capacity of PET MPs on LCMAG NaOH is shown as a function of the MPs concentration in solution in Figure 10. The residual PET content (not captured by the biocomposite) was determined by gravimetry and Ultraviolet-Visible Spectroscopy. The lowest residual PET concentration in AGF, determined by UV/Vis, was obtained after 10 min of contact, reaching $21.4 \pm 2.2\%$ (*w/w*). The residual PET contents after 5 and 15 min were $56.9 \pm 4.6\%$ and $36.4 \pm 3.6\%$ (*w/w*), respectively. As observed for PS MPs, the 10 min contact time provided the highest removal efficiency. The magnetic recovery efficiencies of LCMAG NaOH were 88.2 ± 2.0 , 96.8 ± 2.5 , and $93.9 \pm 3.4\%$ (*w/w*) after 5, 10, and 15 min, respectively. These results explain the higher residues obtained by gravimetry, since the small fraction of unrecovered biocomposite remaining in suspension contributed to the measured mass.

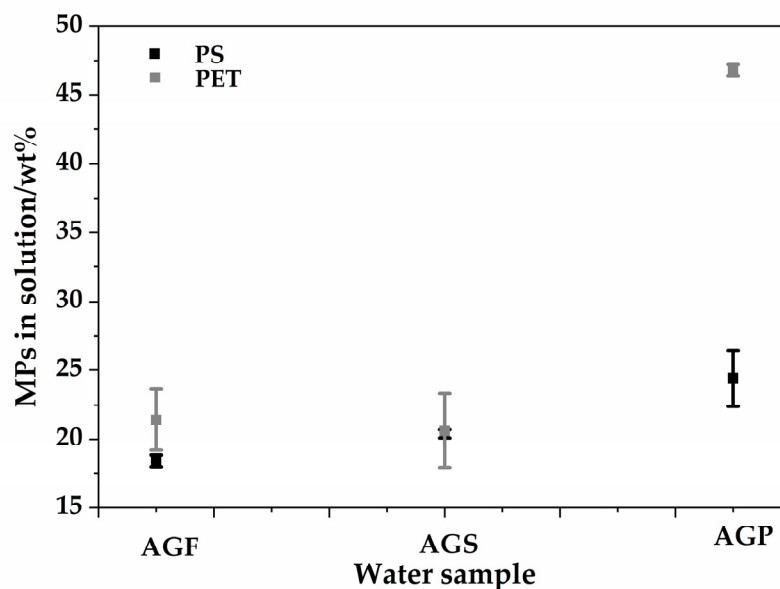


Figure 10. Determination of residual content of PS and PET MPs after LCMAG NaOH adsorptions in different simulations of aquatic media by ultraviolet and visible (UV/Vis) spectroscopy, after capture of the biocomposite + adsorbed PS, at a time of 10 min. Measurements were taken in triplicate, in lake water from the park (AGP), drinking water (AGF), and simulated seawater (AGS) at 25 °C.

The removal capacity of PET on LCMAG NaOH was compared with that of PET MPs on LC IN (fresh *L. cylindrica* sample) and LC NaOH at 5, 10, and 15 min (Figure 5d). The residual PET contents for LCMAG NaOH at 5, 10, and 15 min (56.9 ± 4.6 ; 21.4 ± 2.2 ; $36.4 \pm 3.6\%$ (*w/w*), respectively) were lower than those for LC IN. The 10 min time (discussed

previously) showed the lowest MP content in the PET in the water ($21.4 \pm 2.2\%$ (w/w)), with less dispersion of measurements across the evaluated contact times compared to the three samples. The higher removal efficiency of PET MPs on LCMAG NaOH compared to adsorption on LC IN and LC NaOH indicates that incorporating magnetite into the alkali-treated lignocellulosic matrix substantially improved PET removal. The same result was observed for the removal of the PS MPs, and the enhanced performance can be attributed to the combined effects of the increase in specific surface area induced by the alkaline treatment and the effects of surface chemical modification, increased surface heterogeneity, and the presence of magnetite, which synergistically contributed to the adsorption process.

The enhanced performance of LCMAG NaOH relative to LC IN and LC NaOH suggests that magnetite incorporation contributes not only to magnetic recovery but also to the formation of additional adsorption sites. Therefore, the adsorption mechanisms discussed previously, including hydrophobic interactions, van der Waals forces, surface heterogeneity, and the limited contribution of electrostatic interactions, as inferred from zeta potential measurements, are also expected to govern PS and PET adsorption.

The adsorption of phenol by lignocellulosic materials has been extensively investigated and may provide useful insights into the adsorption of PET and PS, since phenol represents the aromatic structural unit present in both polymers [82,83]. Abdelwahab and Amin reported that phenol adsorption on *Luffa cylindrica* decreased with increasing temperature, indicating a spontaneous and exothermic adsorption process ($\Delta G < 0$) [83]. Although PET is a polymer with physicochemical properties distinct from phenol, the presence of aromatic rings in both compounds suggests that similar π - π interactions and hydrophobic interactions may contribute to adsorption onto lignocellulosic surfaces. The superior adsorption performance of LCMAG NaOH compared with LC IN can be attributed to the combined effects of magnetite incorporation and alkali treatment. Magnetite provides additional hydroxylated active sites and may interact with the aromatic moieties of PET, whereas alkali treatment removes soluble components, increases surface area, exposes cellulose-rich domains, and modifies the surface chemistry of the fibers, thereby increasing the availability of adsorption sites [82]. The slight decrease in adsorption observed after 15 min may indicate the establishment of adsorption-desorption equilibrium or the progressive occupation of the most energetically favorable adsorption sites. In addition, hydration of the magnetite surface and competitive interactions with water molecules may partially reduce the affinity between PET MPs and the adsorbent surface during longer contact times.

3.5. Determination of the Removal Capacity of PS and PET Microplastics on LCMAG NaOH in Simulated Seawater (AGS) and Park Lake Water (AGP)

The removal capacity of LCMAG NaOH toward PS and PET MPs was evaluated separately in simulated seawater (AGS) and Park Lake water (AGP, collected from the same site where residual plastics were obtained for the production of simulated MPs, Dourados, Mato Grosso do Sul, Brazil) using a contact time of 10 min (Figure 10). Residual MPs concentrations were determined by both gravimetry and UV/Vis Spectroscopy. As observed in the previous experiments, UV/Vis measurements exhibited lower variability than gravimetry, reflecting this technique's higher selectivity for polymer quantification. The higher gravimetric residues are attributed to the small fraction of LCMAG NaOH that was not recovered magnetically after adsorption. This interpretation is supported by the magnetic recovery efficiencies obtained for LCMAG NaOH in AGF, AGS, and AGP (92.8 ± 0.7 , 93.0 ± 0.7 , and $91.7 \pm 0.7\%$, respectively), indicating that unrecovered biocomposite particles contributed to the gravimetric residue.

The physicochemical characteristics of the aqueous media differed substantially. Drinking water (AGF) had a pH of 7.86 and a conductivity of $88.85 \mu\text{S cm}^{-1}$, whereas simulated seawater (AGS) had a pH of 8.30 and a conductivity of 41.63 mS cm^{-1} due to its high ionic strength. Park lake water (AGP) had a pH of 7.66, conductivity of $149.5 \mu\text{S cm}^{-1}$, dissolved oxygen of $2.31 \pm 0.15 \text{ mg L}^{-1}$, and a BOD_5 of $156 \pm 31 \text{ mg O}_2 \text{ L}^{-1}$, indicating a considerably higher organic matter content.

The lowest residual PS concentration after adsorption was observed in drinking water ($18.4 \pm 0.5\%$), followed by simulated seawater ($20.4 \pm 0.3\%$). Likewise, the residual PET concentrations in AGF and AGS were very similar (21.4 ± 2.2 and $20.6 \pm 2.7\%$, respectively). Despite the high concentrations of dissolved ions present in simulated seawater (Na^+ , Mg^{2+} , K^+ , Ca^{2+} , Sr^{2+} , among others), no significant differences were observed in the adsorption efficiencies of either PS or PET. Similar behavior has also been reported for PS adsorption in electrolyte solutions [84]. These results indicate that, under the experimental conditions evaluated, the increase in ionic strength alone did not substantially affect the adsorption performance of LCMAG NaOH. In the experimental conditions reported by Savaji et al., pH did not significantly affect the adsorption kinetics at a constant ionic concentration [77].

In contrast, adsorption in park lake water was noticeably less efficient. The residual PS and PET concentrations increased to $24.4 \pm 2.0\%$ and $46.8 \pm 0.4\%$, respectively, with the residual PET concentration approximately twice that of PS. Since the pH values of AGF and AGP were very similar (7.86 and 7.66, respectively), pH is unlikely to be the main factor responsible for the reduced adsorption observed in AGP. Instead, the principal difference between these media is the high organic matter content of the lake water, as indicated by its elevated BOD_5 .

The influence of pH on adsorption processes has been extensively reported because it affects the ionic strength of the medium, the surface charge density of adsorbents and MPs, and consequently the zeta potential [25,84]. Sun et al. demonstrated that the surface charge of magnetite varies with pH owing to protonation and deprotonation of surface hydroxyl groups [75]. According to these authors, magnetite exhibits zeta potential values close to zero at approximately pH 6, whereas predominantly negative values (approximately -5 to -40 mV , depending on the magnetite source and measurement conditions) are observed near pH 8 [75]. Given that the pH values of AGF, AGS, and AGP were all within the neutral-to-slightly alkaline range, only minor differences in magnetite surface charge would be expected. Therefore, the marked reduction in adsorption efficiency observed in AGP is more likely attributable to dissolved and suspended organic matter than to pH effects.

Organic matter can compete with MPs for adsorption sites or form a coating on the adsorbent surface, reducing the accessibility of active sites and limiting contact between the MPs and the LCMAG NaOH surface. This physical barrier may be particularly important for PET, whose higher density than PS [84] may reduce particle mobility and increase the probability of interactions with natural organic matter before reaching the adsorbent surface. Consequently, PET adsorption was more strongly affected than PS adsorption in park lake water.

4. Conclusions

In this study, a low-cost biocomposite was produced from renewable, readily available raw materials obtained from *L. cylindrica* and natural magnetite, using a fast, scalable process under brand-new conditions and temperatures limited to 80°C . The incorporation of magnetite in *L. cylindrica* after treatment with sodium hydroxide solution (LCMAG NaOH) increases the capacity of the biocomposite to capture MPs of PS and PET by more than 50%, as compared to LCMAG IN and LC IN.

LCMAG NaOH was efficient at rapidly removing MPs from PS and PET in three simulated aqueous media: drinking water (AGF), seawater (AGS), and lake water (AGP).

LCMAG NaOH was an excellent bioadsorbent, capable of remaining on the aqueous surface, being reusable, and maintaining magnetic properties, with a biocomposite mass recovery greater than 90%.

Supplementary Materials: The following supporting information can be downloaded at <https://www.mdpi.com/article/10.3390/magnetochemistry12080086/s1>. Figure S1: Map on the left: Dourados/MS; on the right: Antenor Martins Park (Lake Park), showing the main water entry and exit points in the park, including the plastic collection point; Figure S2: LC IN sample (LC in natura) fixed to a thread (a), adsorbent measurement using a caliper (20 to 23 mm) (b); Figure S3: Beakers containing park lake water and PS MPs for LCMAG NaOH adsorption experiments; Figure S4: Typical UV/Vis spectra used in the determination of the concentration of PS (a) and PET (b) in the standard solution; Figure S5: The standard curve for the determination of the concentration of PS (a) and PET (b) in solution; Figure S6: FTIR spectra of (a) MAG; (b) LC IN; (c) LCMAG NaOH; and (d) LCMAG IN; Figure S7: FTIR spectra of (a) PS; (b) PET; and (b) LCMAG NaOH; Figure S8: SEM image of the LCMAG NaOH after removal of PET MPs; Figure S9: Optical microscopy image of the LCMAG NaOH after removal of PET MPs. Table S1: Elemental composition of the samples MAG, LC NaOH, and LCMAG NaOH by X-ray fluorescence. Figure S10. Experimental and theoretical diffractograms obtained from Rietveld refinement of (a) MAG samples and (b) mass percentage of phases.

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