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Original Research

A Novel Hazelnut Shell/Magnetite Bio-Nanoadsorbents for Enhanced Oil Removal From Oil-Contaminated Waters

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Abstract:

The primary objective of this study is to evaluate the feasibility of using hazelnut shells as a biosorbent for the cleanup of oil and petroleum product spills in water bodies. The effects bioadsorbent, nanoparticle concentrations, oil concentration, temperature (10 – 400 °C), and pH on oil adsorption were investigated to determine the optimal conditions for maximum purification efficiency. The highest oil removal efficiency achieved with the biosorbent was 61.25% at a pH of 7.5. To enhance the sorption capacity of the biosorbents were synthesized by incorporating Fe_3O_4 superparamagnetic nanoparticles at concentrations of 1%, 3%, 5%, and 10%. Characterization of both the biosorbents was conducted using SEM, TGA, and FTIR analyses to assess the interaction between Fe_3O_4 nanoparticles and the plant-derived surface of the biosorbent. For an adsorbent such as hazelnut shell + Fe_3O_4 bio-nanoadsorbent, the pHzpc indicates the point at which the surface of the bio-nanoadsorbent changes from negatively charged to positively charged as the pH of the solution increases or decreases. The zero-charge pH point of the bio-nanoasorbent was determined to be pH = 8 by Boehm titration. The optimal adsorption conditions for the bio-nanosorbent, composed of hazelnut shell and 10% Fe_3O_4 nanoparticles, were found to be 92.5% oil removal within 12 minutes at a pH of 7.5.

Keywords: Adsorption; Bio-nanoadsorbent; Biosorbent; Hazelnut shell; Oil and oil products; Superparamagnetic nanoparticles

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1. Introduction

The water obtained from the discharge of a large amount of oil waste into the aquatic environment during oil extraction and product production is referred to as produced water. The composition of this type of produced water varies depending on many factors [1]. The composition of produced water varies, particularly depending on the type of hydrocarbon extracted, the extraction method used, and the type of oil [1]. Pollution of the aquatic environment with oil and petroleum products has a devastating impact on the environment. Oil spreads in a thin layer on the surface of the water, preventing oxygen from entering the plants and animals living in the water. For example, petroleum products soak into the fish's gills, preventing them from breathing and inhibiting the respiration process, resulting in their mass destruction [2].

Oil-polluted waters are considered particularly favorable environments for the growth of bacteria (Alcanivorax and Pseudomonas specie) and pathogens. Diseases such as cholera, dysentery or hepatitis can spread if the water is not treated in areas where polluted water is spread [1, 2]. Water contaminated with oil and oil products contains dissolved substances, a complex mixture of organic and inorganic compounds, inorganic salts, radioactive substances, toxic gases, various minerals, and large amounts of organic substances (benzene, toluene, ethylbenzene, and xylene, etc.) and hydrocarbons such as PAH (polycyclic aromatic) to come across. Discharges of oil and oil products into the aquatic environment have increased recently, resulting in significant environmental problems [1, 2]. To prevent or eliminate ecological problems, several cleaning technologies and methods have been developed to remove oil and oil products from water [2]. Among these methods, activated carbon as an adsorbent adsorbs highly porous oil with a large surface area. There are also membrane-based filtration methods for removing petroleum products from water, such as oil-water separation membranes. This method can either adsorb oil directly or act as a physical barrier to prevent oil from passing through. Zeolites are also effective at adsorbing oil due to their high surface area, like activated carbon adsorbents. Graphene oxides exhibit adsorption effects similar to adsorbents such as zeolite and activated carbon. Recent studies have reported, it is known that the use of nanocellulose (NC) as an adsorbent material for the treatment of aquatic environments is also prevalent in modern times [1, 3]. Among these methods, the biosorption method is also preferred for cleaning the aquatic environment. The process of biosorption utilizes low-cost biological materials. Currently, agricultural waste and plant waste are employed [3].

Many products are developed to produce adsorbents with high adsorption efficiency at a low cost from final agricultural wastes. Additionally, transforming agricultural waste into a valuable adsorbent with high adsorption capacity is proposed as an innovative approach to agricultural waste treatment from the environment [1, 2, 3]. Furthermore, converting agricultural waste into a cost-effective, highly adsorbent material offers a novel approach to treating agrarian waste. The primary reason for utilizing plant-of-origin waste as an adsorbent material is its high absorption capacity, low ash content, and well-developed porous structure. As a result, this technology is economical and environmentally friendly [3].

Hazelnut shells are the waste of the hazelnut tree. The primary producers of hazelnuts are Turkey, Azerbaijan, and the United States [4]. Hazelnut shells are used in Azerbaijan, primarily for furniture production and as a source of fuel. Utilizing hazelnut shell waste as an effective bioadsorbent for further environmental cleaning is also considered appropriate. The composition of hazelnut shells consists of cellulose fibers, with the following breakdown: approximately 30% hemicellulose, 26% cellulose, 43% lignin, and approximately 3.3% extractives [4]. According to research by numerous scientists, hazelnut shells comprise 25% hemicellulose, 37% cellulose, and 23% lignin. The primary purpose of choosing hazelnut shells is that they can be reused in the environment, biodegradable and environmentally benign and is not considered harmful to the environment [4]. M. Stjepanovi´c et al. (2022) synthesized a new adsorbent for nitrate removal from wastewater [5]. This adsorbent is made by chemically modifying hazelnut shells. They have found that a new adsorbent made by chemically modifying hazelnut shells is effective in removing nitrates from water. L.P. Cruz Lopes et al. (2012) found that hazelnut shells have a good effect in removing several toxic ions and organic compounds [6]. The chemical composition of chemically modified hazelnut shells consists of lignin, cellulose, hemicellulose, amines, thiols, etc. On the other hand, they also found that hazelnut shells are a source of natural and effective phenolic antioxidants.

Sadeghi et al. (2011) introduced a simple method for synthesizing environmentally friendly nanosorbents based on a gold/hydroxypropyl cellulose hybrid nanocomposite for various applications [7]. Viviane G. P. Ribeiro et al. (2019) synthesized new hybrid nanomaterials based on a magnetite core, resulting in stable and magnetic hybrid nanomaterials through the Van der Waals interaction between alkyl chains protruding from the Fe_3O_4 surface [8]. Mubarak et al. (2024) developed a novel MoS_2-carbon quantum dot (CDs@MS-NF) nanocomposite designed for eco-friendly mitigation of calcite scaling [9]. They discovered that plant-of-origin waste with a porous structure contains carboxyl, hydroxyl, and other active groups, which can better clean pollutants. Hydrothermal and co-precipitation methods were used to control the size of the synthesized nanoparticles [10]. The electronic, optical, magnetic, and catalytic properties of these nanoparticles depend mainly on their size, structure, magnetic stability, and purity, which makes their use possible in the electronics, audio recording, and medical industries. Fe_3O_4 nanoparticles synthesized using the hydrothermal method are pure and have high magnetization capability. The preparation of new environmentally friendly chemicals derived from renewable, natural sources and recyclable plant-based waste has generated significant interest [10, 11]. In our research, the synthesis of plant-based nanoadsorbents is described as an environmentally friendly chemical. In our study, we provide information on the synthesis of new magnetite Fe_3O_4 nanoparticles. Magnetite Fe_3O_4 nanoparticles, due to their high surface-to-volume ratio, exhibit high absorption and reaction capabilities, making them practical for the absorption and separation of oil and petroleum products from water [9, 10, 11]. Because magnetite Fe_3O_4 nanoparticles can be easily separated using external magnetic fields, their use as adsorbents in water purification is more practical. In our research, the presence of magnetic nanoparticles also helps to increase the surface area and improve the interaction between the adsorbent and the oil molecules. Magnetic nanopar-

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ticles are effective in the adsorption of oil molecules. The Fe_3O_4 superparamagnetic nanoparticles we used increase their adsorption efficiency due to the presence of an external magnetic field without maintaining the magnetization constant [10]. The presence of superparamagnetic properties of Fe_3O_4 nanoparticles and the fact that they are a reusable nanoparticle led us to choose this nanoparticle in our research [10]. Magnetite, a ferromagnetic material, is often used in adsorbent composites to enable easy recovery of the adsorbent via magnetic fields. Ensuring the mechanical and chemical stability of the composite adsorbent is essential for its long-term effectiveness and environmental sustainability.

A new bio-nanoadsorbent was synthesized by impregnating Fe_3O_4 superparamagnetic nanoparticles on hazelnut shells. An adsorption experiment used the synthesized biosorbent and bio-nanoadsorbent to treat oil-contaminated waters.

The novelty of this work lies in the application of low-waste and zero-waste technology and the cost-effectiveness and cheapness of synthesized adsorbents for oil purification.

The primary objective of the presented paper is to synthesize new bio-nanoadsorbents based on hazelnut shells and Fe_3O_4 nanoparticles and to investigate their application in the purification of oil-contaminated waters.

2. Experimental, materials, and methods

2.1 Materials and methods

Iron sulfate heptahydrate ($FeSO_4 \cdot 7H_2O$, 98% chemically pure, PLC 141362), ferric chloride hexahydrate ($FeCl_3 \cdot 6H_2O$, 98% chemically pure, PLC 141358), ammonium hydroxide (NH_4OH, 99.9% chemically pure, 25% PLC 141129), polyethylene glycol-6000 ($H(OCH_2CH_2)nOH$, 99.4% chemically pure PEG-6000, PLC 163325), were laboratory-grade chemicals and used without further purification [11, 12].

2.2 Preparation of hazelnut shell

Hazelnut shells from the Gabala region have been used as a biosorbent. Hazelnut shells are first obtained through the hazelnut harvesting process. Harvesting was carried out in September, when the average air temperature was approximately 22 °C and the relative humidity was 65%. First, the shells are washed several times with distilled water to clean them, then dried at 105 °C for 48 hours (Muffle furnace), and crushed, ground, and powdered to obtain a particle size of less than 0.3 mm.

2.3 Synthesis of bionanosorbent based on hazelnut shell and Fe_3O_4

Firstly, Fe_3O_4 magnetic nanoparticles, stabilized with PEG, were synthesized by co-precipitating Fe^{3+} and Fe^{2+} ions in an alkaline medium. Specifically, a 0.5% polyethylene glycol solution was added to 100 mL of Fe^{3+} and Fe^{2+} (molar ratio Fe^{3+}:Fe^{2+} = 3:2) solution, under vigorous stirring for several minutes. Subsequently, 100 mL of a 25% ammonium solution was quickly added. The precipitation process was carried out at a temperature of 70-80 °C for 1 hour. The resulting black precipitate is washed several times with distilled water using the decantation method and is subsequently allowed to air-dry over several days. To prevent agglomeration, the precipitated particles are separated using a centrifuge, then washed with water and poured into a Petri dish for drying after decantation steps [12, 13, 14, 15].

Next, the brown shell of the hazelnut is heated and crushed for synthesis. For synthesizing bio-nano adsorbents based on hazelnut shells and Fe_3O_4 nanoparticles, 1 gram of hazelnut shell is mixed with 1%, 3%, 5%, and 10% Fe_3O_4 nanoparticles and stirred intensively for several minutes (400 rpm and no temperature). Then, 1 mL of a 25% ammonium solution is added, stirring the mixture using a magnetic stirrer for 1 hour [16]. The resulting black precipitate is separated by centrifugation, washed with water and then transferred to a Petri dish, where it is dried. The sorbents are crushed into powder form to enhance the adsorption capacity of the dried bio-nano adsorbents [16, 17].

2.4 Characterization

The X-ray diffractogram of iron nanoparticles was acquired at room temperature employing a Rigaku Mini Flex 600 diffractometer (Rigaku, Japan) equipped with a copper anode X-ray tube (Cu-Kα radiation, 30 kV, and 15 mA). The samples were scanned in a 2θ range of 30° to 80° and step size is 0.005°. Infrared analysis of bio-adsorbent materials based on hazelnut shell and Fe_3O_4 was conducted at room temperature in the wavelength range of 400-4000 cm^{-1} using a Varian 640-IR spectrometer (Agilent Technologies, U.S.) with a spectral resolution of 4 cm^{-1}.

Scanning Electron Microscopy and Energy-Dispersive X-ray Spectroscopy were employed to analyze the structure, size, and elemental composition of hazelnut/Fe_3O_4 adsorbents. The analysis of Fe_3O_4 nanoparticles was carried out using a JEOL JSM 7600F scanning electron microscope (Jeol, Japan) equipped with an X-Max 50 EDS detector (Oxford Instruments, UK). Images of magnetite nanoparticles were acquired using an accelerating voltage of 15 kV and a working distance of 4.5 mm. For the evaluation of the morphology and coverage of iron oxide nanoparticles on bio-adsorbents derived from hazelnut shell, an SEM-EDS analysis was carried out using a field-emission Mira3 electron microscope (Tescan, Czech Republic), operating at a voltage of 15-20 kV and a working distance of 15 mm, along with an EDS detector (EDAX). Before examination, all specimens were sputter-coated with gold (the thickness of the coating is 2-5 nm, and the sputtering time is 1.5 minutes).

An SDT Q600 thermoanalyzer (TA Instruments, New Castle, DE (USA)) was used to determine the thermal properties of hazelnut/Fe_3O_4 adsorbents. Thermogravimetric analysis was conducted in air from room temperature to 900 °C with a heating rate of 10 °C/min.

2.5 Adsorption experiments

Oil recovery from oil-contaminated waters typically involves evaluating the various methods used to recover

clean water. These methods must meet environmental standards and aim to reduce or eliminate oil contamination of the water. When analyzing the performance of oil cleaning methods, the preferred method is evaluated based on their effectiveness (cleaning efficiency, recovery rate), operational feasibility, and environmental impact. This method is the adsorption method, which takes into account the duration of oil cleaning, the amount of cleaning, etc. during the experiment [17, 18, 19].

For the adsorption of oil and petroleum products from the water environment, 10 mL of oil is mixed with 100 mL of H_2O. Then, 0.25, 0.5, 1, and 1.5 grams of hazelnut shells were added to the mixed solution. The resulting mixture is stirred for 30 seconds, and then each sample is left at room temperature for 1 minute, 15 minutes, 30 minutes, 1 hour, 2 hours, 24 hours, and 72 hours (Qravimetric method). After this, the samples are filtered through filter paper.

The filtered papers are dried, and the amount of the filter paper is measured [20]. The optimal hazelnut shell adsorption amount was determined to be 0.5 g. This experiment allows us to determine the biosorbent's optimal concentration and absorption time for absorbing oil (Fig. 1).

3. Results and discussion

3.1 XRD analysis

The X-ray diffraction (XRD) analysis of the synthesized magnetite Fe_3O_4 nanoparticles was performed (figure 2). The XRD analysis of the magnetite nanoparticles reveals that the prominent peaks at 2θ angles of 30.360° (220), 35.680° (311), 43.30° (400), 57.360° (511), and 62.950° (440) correspond to the spinel-structured Fe_3O_4 particles, as indicated by card number N000-001-1111. The diffraction peaks indicate that they belong to Fe_3O_4, as confirmed by the card database. The crystallite sizes were calculated using the Debye-Scherrer formula based on the peak with the highest intensity, and it was determined that the average size of the nanoparticles was 10 nm [18].

The average crystallite size, calculated using the Debye-Scherer formula and derived from the peak with the maximum intensity, was found to be 10 nm [18].

$$D = \frac{K\lambda}{\beta \cos\theta} \quad (1)$$

Here, K is a unitless quantity. $K = 0.94$, λ is the wavelength of the incident X-ray, with a value of $\lambda = 1.5406$ Å (CuKα). The angle corresponding to the θ-diffraction maximum is half the width of the β-diffraction maximum.

3.2 SEM, TEM and EDX analysis

Figure 3 shows TEM image of Fe_3O_4 nanoparticles. According to TEM studies, the average size of Fe_3O_4 nanoparticles is up to 10 nm. In general, it can be said that the porous morphology determined by sorption experiments confirms the presence of pores [19].

Figures 4 and show the SEM images of Fe_3O_4 nanoparticles (a, b), 0.5 g pure hazelnut shell (c), and 0.5 g hazelnut shell + Fe_3O_4 -based nanosorbent (d), respectively. From SEM analysis of the nanoparticles (Fig. 4 (a,b)), we have determined that the distribution of Fe_3O_4 nanoparticles is monodisperse, with an average size ranging from 5 to 8 nm. The SEM analysis of the bio-nanoadsorbents reveals that the addition of Fe_3O_4 nanoparticles to the hazelnut shell biosorbent successfully incorporates the iron oxide nanoparticles onto the biosorbent's surface [19]. Based on the EDX analysis of pure hazelnut shell, it has been determined that the presence of cellulose, hemicelluloses, lignin, tannins, and proteins characterizes the hazelnut shell. Characterizing the hazelnut shell by cellulose, hemicelluloses, lignin, tannins, and proteins reveals that the surface structure of the hazelnut shell consists of voids and exhibits an irregular surface [19, 20]. SEM images also revealed the surface morphology and porosity of the pure and modified biosorbents, providing insight into their adsorption capabilities. Moreover, the structural characterization indicates that the hazelnut shell possesses an irregular surface morphology with numerous voids and pores.

This porous and heterogeneous texture increases the surface area available for adsorption and facilitates mass transfer of adsorbates into the biosorbent matrix. The combined effect of the natural porous structure and the magnetic Fe_3O_4 nanoparticles results in a composite nanosorbent with enhanced adsorption efficiency and ease of separation from aqueous solutions. Overall, the SEM and EDX analyses not only confirm the successful synthesis of the Fe_3O_4/hazelnut shell nanosorbent but also provide insights into its physicochemical properties that underlie its potential application in environmental remediation. SEM imaging was performed on dried samples.

Figures 5 (a) and (b) show the EDX spectra of pure 0.5 g hazelnut shell and 0.5 g hazelnut shell + Fe_3O_4 -based nanosorbent, respectively. According to the EDX analysis, it was determined that the hazelnut shell used as a biosorbent consists of key elements, including carbon (6.69%), chlorine (25.93%), oxygen (29.85%), and sodium (10.71%). With the addition of Fe_3O_4 nanoparticles to the hazelnut shells, it was determined that the main elements of the bio-nanoadsorbent are oxygen (9.87%) and iron (81.34%) [19, 20, 21]. The main stable elements of the bio-nanoadsorbent are considered to be oxygen and iron [21].

Table 1 and Table 2 combine standard values of mass, weight, and atomic. The standard deviation was observed to be higher in elements such as oxygen and chlorine. Standard deviation was learned through the digital filtering method or ZAF algorithm.

Based on the results shown in Table 1, it can be determined that although the chemical composition of the hazelnut shell surface contains chlorides, oxygen is the dominant element.

Based on the results shown in Table 2, it can be determined that although there are chlorides in the chemical

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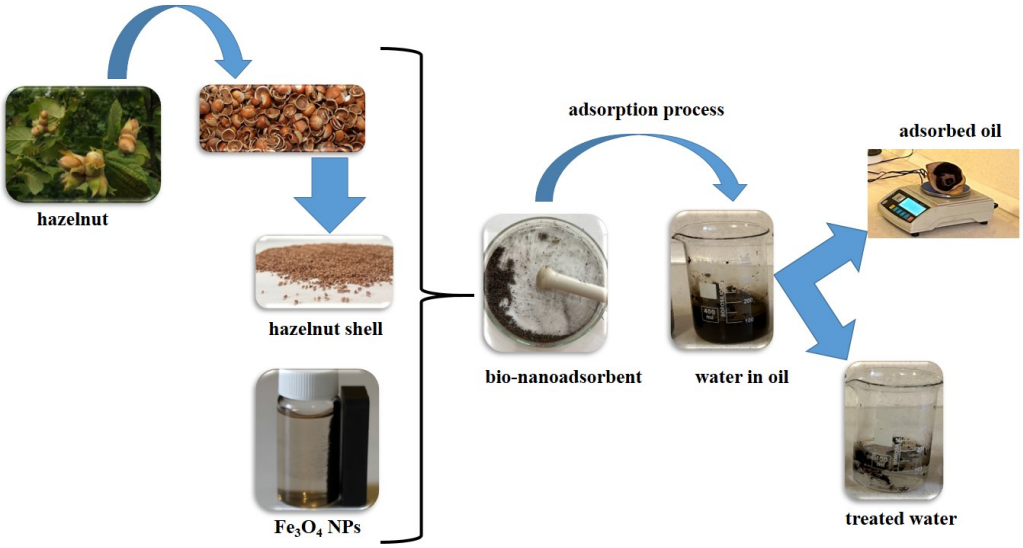

Figure 1. Schematic of Hazelnut shell/magnetite bio-nanoadsorbents for oil removal.

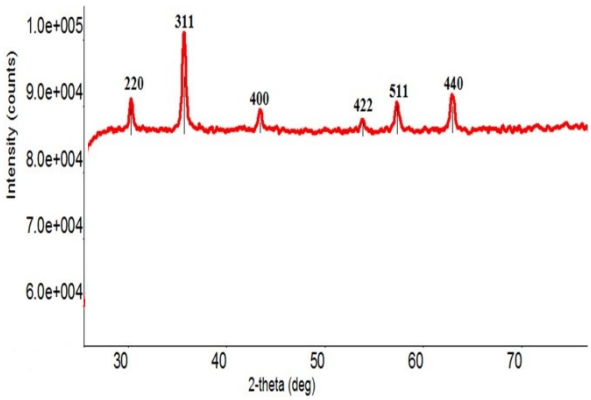

Figure 2. XRD diffractogram of magnetite nanoparticles.

Table 1. Results of EDX analysis of hazelnut shell.

Element	Weight %	Atomic %	Standard deviation (or Error %)
C K	34.95	49.47	±0.8
O K	40.66	43.20	±1.2
NaK	0.00	0.00	±1.0
AuM	10.55	0.91	±0.8
ClK	9.43	4.52	±1.2
K K	3.16	1.38	±1.0

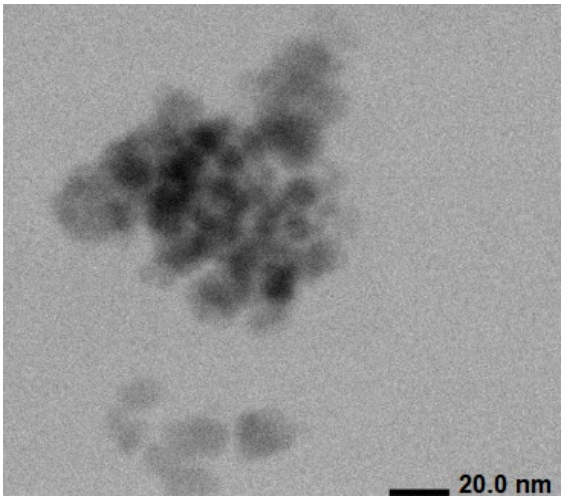

Figure 3. TEM image of Fe_3O_4 nanoparticles.

Table 2. Results of EDX analysis of newly synthesized bio-nanoadsorbent based on hazelnut shell + Fe_3O_4 nanoparticles.

Element	Weight %	Atomic %	Standard deviation (or Error %)
C K	23.03	45.43	±0.8
O K	18.26	27.04	±1.2
NaK	2.48	2.55	±1.0
AuM	9.03	1.09	±0.8
ClK	15.77	10.54	±1.2
FeK	31.44	13.34	±1.0

composition of the surface of the bio-nanoadsorbent, Fe is observed as the dominant element.

The magnetic behavior of Fe_3O_4 nanoparticles was analyzed using a Vibrating Sample Magnetometer (VSM) (Tokyo, Japon). Figure 6 shows the temperature dependence of the magnetic moment of the nanoparticles. As seen in the figure 6, the nanoparticles exhibit a net positive magnetization, a distinct peak in the Zero-Field-Cooled (ZFC) curve, and a reversible region between the ZFC and Field-Cooled (FC) curves above 200 K. The samples sit in a stream of heated air, not a vacuum or inert gas. These characteristics in the m(T) curve indicate superparamagnetic behavior, with a blocking temperature of approximately 100 K. Compared to bulk magnetite, which exhibits ferrimagnetic ordering and a much higher blocking temperature (well above room temperature), the significantly lower blocking temperature observed here reflects the particles' nanoscale size.

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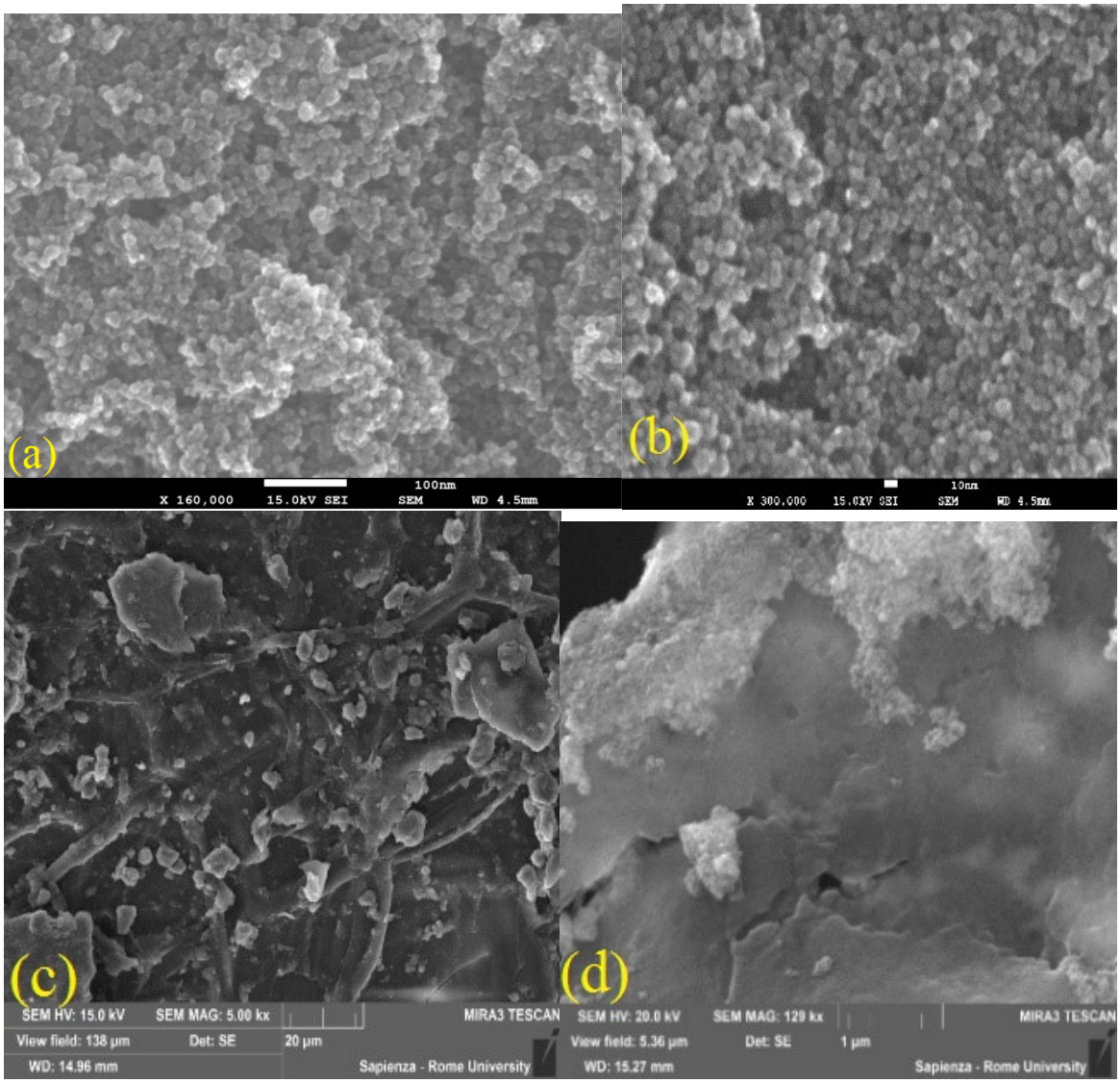

Figure 4. SEM analysis of Fe_3O_4 nanoparticles (a,b), hazelnut shell biosorbent (c), and hazelnut shell + Fe_3O_4 bio-nanosorbent (d).

In such small particles, thermal energy is sufficient to randomly flip the magnetization direction, suppressing long-range magnetic order above blocking temperature and resulting in superparamagnetic behavior [22].

These results are consistent with literature reports on Fe_3O_4 nanoparticles of similar size, where superparamagnetism dominates due to finite-size effects and surface spin disorder. The observed magnetic behavior confirms that the synthesized nanoparticles maintain the fundamental magnetic characteristics of magnetite while exhibiting distinct size-dependent phenomena [22]. VSM analysis has shown that the coercivity (Hc) in the main hysteresis parameters is close to zero (e.g. < 50 Oe) and the remanent magnetization is negligible compared to the saturation magnetization. These values are characteristic of superparamagnetic behavior, as they indicate remagnetization and insensitivity.

Figure 7 presents the magnetization curve of Fe_3O_4 nanoparticles. As the intensity of the applied magnetic field increases, the specific magnetization of the nanoparticles rises, reaching a saturation value of approximately 60 emu/g. It is known from the literature that the saturation value for bulk magnetite is 92-98 emu/g [22]. The absence of hysteresis in the magnetization curve confirms the superparamagnetic nature of the nanoparticles. Additionally, the nanoparticles exhibit a rapid magnetic response. The combination of high saturation magnetization, superparamagnetic behavior, and fast response indicates that Fe_3O_4 magnetic nanoparticles are well-suited for applications in magnetic separation and recovery processes.

3.3 IR analysis

The IR spectra of the hazelnut shell and hazelnut shell + Fe_3O_4 -based bio-nanoadsorbent are shown in figure 8.

In the infrared spectrum of the plant-based bioadsorbent, hazelnut shell (figure 8 (a)), the absorption bands at 619 cm^{-1} and 774 cm^{-1} correspond to the C-Cl bond, indicating the presence of alkyl groups. The absorption bands at 1001 cm^{-1} and 1063 cm^{-1} correspond to the =C-H bond, indicating the presence of alkenes, and the C-H bending band suggests the presence of aromatic

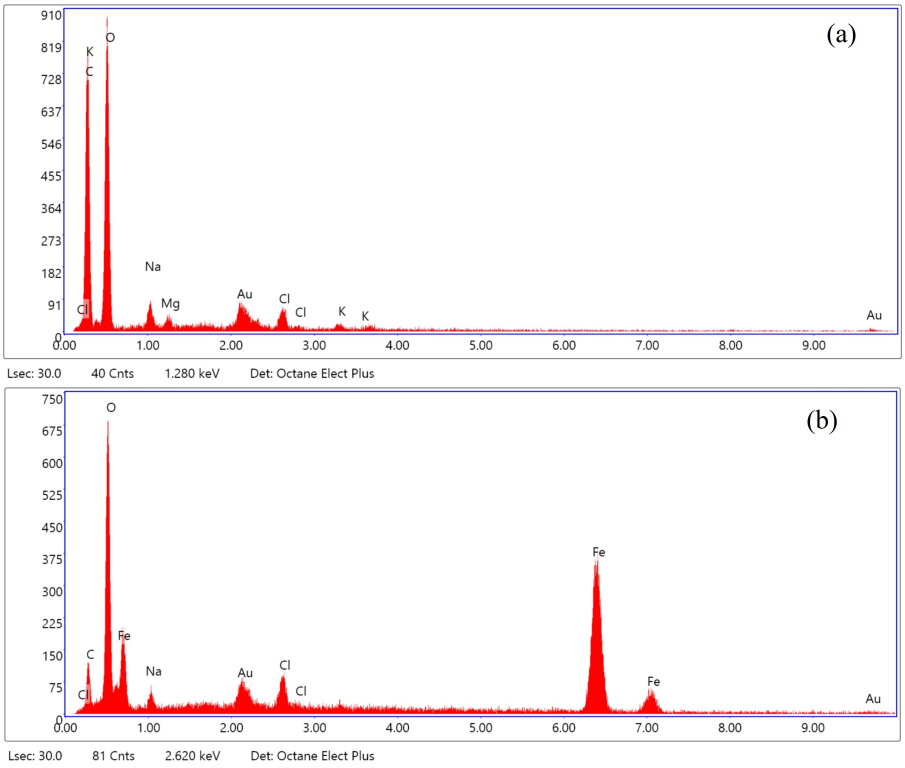

Figure 5. EDX analysis of hazelnut shell biosorbent (a) and hazelnut shell + 10%Fe_3O_4 bio-nanosorbent (b).

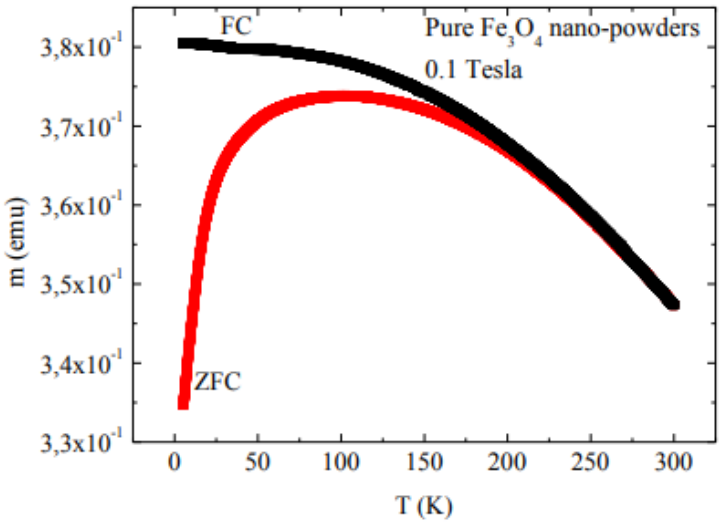

Figure 6. Dependence of magnetic moment of Fe_3O_4 nanoparticles from the temperature, at the induction of magnetic field 0.1 Tesla.

compounds in the hazelnut shell [22, 23]. The absorption band at 1354 cm^{-1} indicates the presence of NO_2 from nitro compounds. At 1350–1330 cm^{-1}, aromatic nitro compounds are present, and at 1390–1350 cm^{-1}, aliphatic nitro compounds are present. Thus, the presence of the n –NO_2 group at 1354 cm^{-1} is clearly visible in figure 8 (a). The absorption bands in 2035, 2373, and 3620 cm^{-1} correspond to O-H, C-H, and N-H groups, respectively, which explain the presence of cellulose, hemicelluloses, lignin, tannins, and proteins [23].

According to literature data, C≡C stretching is observed in the range of 2100-2260 cm^{-1}, –C≡N 2200-2250 cm^{-1} (strong), and C–H stretching (in the aldehyde group) at 2725 cm^{-1} [24].

From the IR spectrum of the hazelnut shell and Fe_3O_4 -based nanosorbent (figure 8 (b)), it is evident that the absorption band at 617 cm^{-1} indicates the presence of characteristic Fe–O bond bands. The absorption band at 1618 cm^{-1} allows the identification of the O-H bond. This suggests the presence of hydroxyl groups on the surface of Fe_3O_4 nanoparticles in the bio-nanoadsorbent's infrared spectrum. The hydroxyl groups are formed during the synthesis process, which is carried out in an aqueous environment. Additionally, the absorption bands at 617 cm^{-1} and 773 cm^{-1} indicate the presence of C-Cl bonds [23, 24]. The absorption bands between

1356-1610 cm^{-1} correspond to the stretching of aromatic C=C bonds. In figure 8 (a) and 8 (b), the weight ratio is 0.5 g for both adsorbents.

3.4 TGA analysis

Figure 9 shows the TGA curves of the pure plant-based hazelnut shell bio-sorbent and hazelnut shell + (3.5%) Fe_3O_4 nanoparticle-based synthesized bio-nanosorbent [24, 25, 26]. According to the TGA curve, a sharp mass loss of the hazelnut shell is observed from 34 °C to 600 °C. The TGA curve indicates that the 3% and 10% bio-nanosorbents synthesized with Fe_3O_4 nanoparticles have improved thermal stability compared to the pure hazelnut shell after 600 °C. The thermal stability is observed from 600 °C to 991 °C [26, 27, 28]. During TGA analysis, the gas atmosphere was conducted under N_2 atmosphere to study the thermal stability [28].

Thus, the pure hazelnut shell exhibits a 32.6% mass loss, the bio-nanoadsorbent synthesized from hazelnut shell + 3% Fe_3O_4 nanoparticles shows a 24.4% mass loss, and HN + 3% Fe_3O_4 nanoparticles shows a 50.59% mass loss between 991 °C and 600 °C. Thermal stability is observed at 600-800 °C. Inert gases (usually nitrogen or argon) are used to prevent oxidation during TGA analysis. The use of these gases is very important for materials that are prone to decomposition or oxidation at high temperatures [29].

The increased thermal stability of the bio-nanosorbents is attributed to the interaction and contact between the iron oxide nanoparticles and the surface of the hazelnut shell. Based on the TGA analysis, it has been determined that the mass loss of the plant-based bioadsorbent (hazelnut shell) during the experiment is related to its structural decomposition and the evaporation of water content [30, 29, 31].

The percentages of the composition of the bio-nanoadsorbent are indicated because the 10% bio-nanoadsorbent synthesized based on hazelnut shell + Fe_3O_4 nanoparticles contains more Fe_3O_4 nanoparticles than the 3% bio-nanoadsorbent [32].

3.5 Adsorption experiments

First, in the adsorption experiment, it was determined that 100 mL of water was used, and the purity of the water was 99% (distilled water). As can be seen from the time-dependent graph of 0.5 g hazelnut shells, the biosorbent can absorb 51.3% of oil in 1 minute, 51.6% in 5 minutes, 57.6% in 10 minutes, 58.6% in 12 minutes, 60.62% in 20 minutes, and 61.25% in 30 and 60 minutes (figure 10). The optimal condition for the hazelnut shell is the absorption of 61.25% of oil in 30 minutes [36]. The standard deviation is 0.15 g/L in our sorption process.

Adsorption experiment of Fe_3O_4 nanoparticles oil from water environment was also conducted. In particular, during the experiment, the optimal absorption time was determined as a function of the oil absorption time. The nanoparticle has the ability to adsorb 84% of oil in 1 minute, 84.2% in 5 minutes, 85% in 10 minutes, 85.1% in 12 minutes, 85.6% in 15 minutes, 85.58% in 20 and 30 minutes, and 85.56% in 60 minutes.

A bio-nanoadsorbent synthesized with hazelnut shell and Fe_3O_4 nanoparticles in different concentrations (1, 3, 5, 10%) is synthesized. New bio-nanoadsorbents have been synthesized to increase the efficiency of adsorption experiments.

According to the experiment's results, as shown in figure 10, to compare the obtained results, a bio-nanoadsorbent synthesized based on hazelnut shell and 1, 3, 5, 10% Fe_3O_4 nanoparticles is added to the water medium mixed with oil, and the experiment is repeated as described above [36, 37]. This experiment allows us to determine the optimal concentration and absorption time of a bio-nanoadsorbent for absorbing oil.

As shown in figure 10, the 1% bio-nanosorbent absorbs 51.25% of the oil in 1 minute, 52.5% in 5 minutes, 53.75% in 10 minutes, 54.25% in 12 minutes, 57.75% in 20 minutes, 58.5% in 30 minutes, and 58.75% in 60 minutes. As seen from the time dependence graph

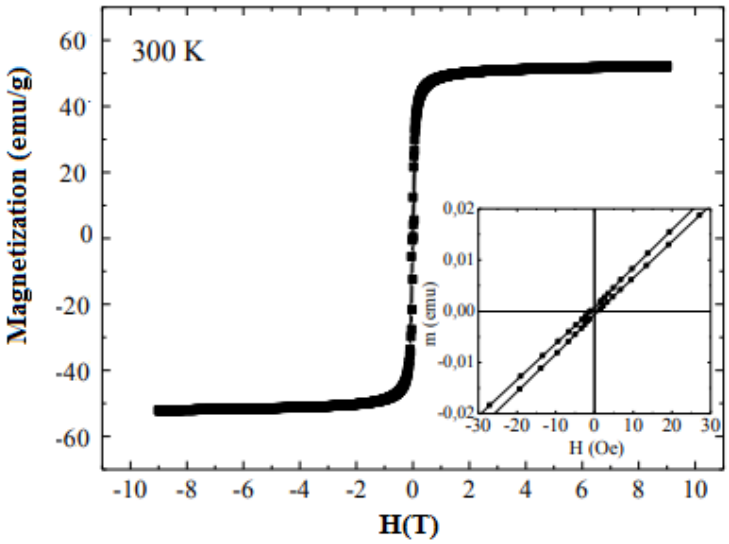

Figure 7. Magnetization curve of Fe_3O_4 nanoparticles.

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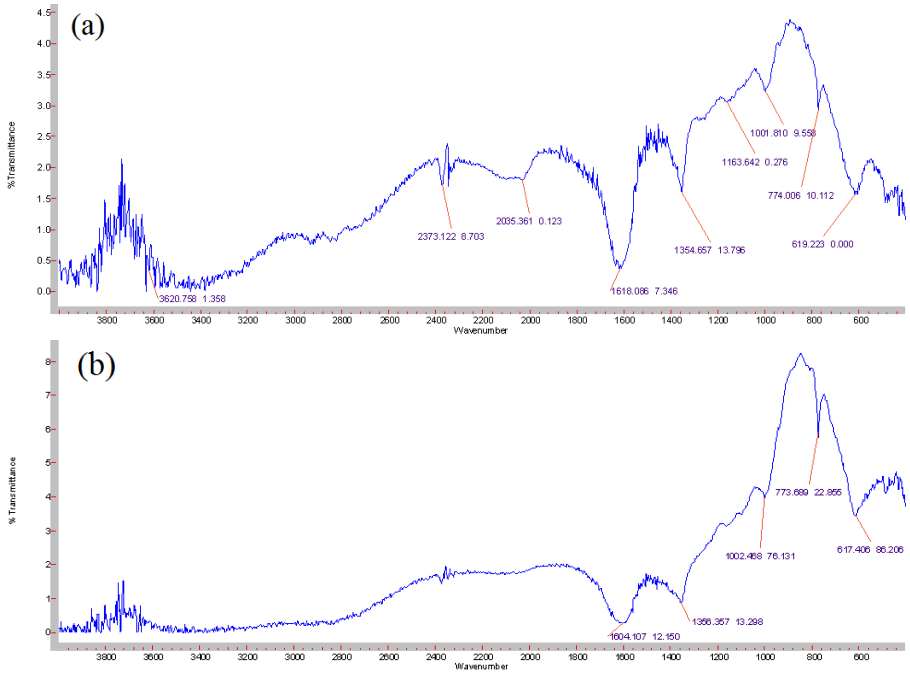

Figure 8. IR spectra of pure hazelnut shell (a) and hazelnut shell + 10%Fe_3O_4 (b) based sorbent.

(Fig. 10) of the bio-nanosorbent, the 3% bio-nanosorbent has an oil absorption capacity of 75% in 1 minute, 76.25% in 5 minutes, 77.5% in 10 minutes, 78.12% in 12 minutes, 81.25% in 20 and 30 minutes, and 81.37% in 60 minutes. Then, the experiment was performed with 5% bio-nanoadsorbent, and it absorbed 81.25% of the oil in 1 minute, 83.5% in 5 minutes, 85% in 10 minutes, 85.37% in 12 minutes, 87.5% in 20 minutes (also 60 minutes), and 87.6% in 30 minutes. For comparison, the experiment was continued with 10% bio-nanoadsorbent, and 90.75% of the oil was adsorbed at 1 minute, 91.5% at 5 minutes, 91.87% at 10 minutes, 92.5% at 12 minutes,

Table 3. Physical-chemical parameters of oil.

Name of the oil	Density (kg/m^3)	H_2O (%)	Cl (mg/L)	S (%)	T (°C)
Shirvan oil	863.16	0.03	40.9	0.21	29.32

Table 4. Comparison of oil removal from oil-contaminated waters on different adsorbents.

Name of the adsorbents	Adsorption Capacity, %	Environmental Impact	Advantages	Disadvantages
Cellulose	70.5	Low (biodegradable, renewable)	Biodegradable, renewable, environmentally friendly	May require modification for higher efficiency, slower adsorption rate
Coconut Shell Biochar	65	Low (biodegradable)	Biodegradable, abundant, renewable, low cost [33]	Lower adsorption capacity, slower adsorption
Mushroom-based Bioadsorbents [34]	70	Moderate (biodegradable)	Renewable, high surface area, effective for large-scale treatment [34]	May have lower efficiency compared to synthetic adsorbents [34]
Rice Husk [34]	67	Low (biodegradable)	Low-cost, abundant, environmentally friendly [34]	Moderate adsorption capacity, less efficient at high oil concentrations [34]
Pomegranate peel [34]	71.5	Low (biodegradable)	Low cost, renewable, environmentally friendly	High absorption capacity [34]
PP + Fe_3O_4	99.75	Low	Low cost, renewable, environmentally friendly	High absorption capacity
Hazelnut shell [34]	61.25	Low (biodegradable)	Low cost, renewable, environmentally friendly	Medium to good absorption capacity [34]
HN + Fe_3O_4 (Hazelnut shell + Fe_3O_4)	92.5	Low	Low cost, renewable, environmentally friendly [35]	Medium to good absorption capacity

92.1% at 20 minutes, 92% at 30 minutes, and 60 minutes.

Figure 10 shows that the bio-nanoadsorbent with the most optimal absorption capacity is the one synthesized using hazelnut shells and 10% Fe_3O_4 nanoparticles, achieving 92.5% absorption at 12 minutes [37]. Based on the experiment, the optimal adsorption amount of the bio-nanoadsorbent was 0.5 g, and the optimal concentration was 10%.

To conduct the experiment, the physical and chemical parameters of Shirvan oil were first determined (Table 3). The absorption experiment of biosorbent and bio-nanoadsorbent of oil with a density of 863.16 kg/m^3 was conducted.

An experiment was conducted to investigate the dependence on pH to determine the optimal conditions. Experiments were conducted in the pH range of 1-10 to determine the pH dependence. The experiment continued over time, varying the bio-nanoadsorbent and temperature [38, 39].

While investigating the oil adsorption of our newly synthesized adsorbents, the literature data on oil sorption of various adsorbents were reviewed [33]. Table 4 shows the treatment of oil-contaminated water on multiple adsorbents.

It is also clear from the table that plant-based adsorbents remove oil from water with their high absorption capacity, causing less harm to the environment. The adsorption capacity is greater in pure pomegranate peel and cellulose adsorbents.

The optimum pH value of the hazelnut shell adsorbent was measured using a pH meter. The pH_{pzc} was determined by mixing 1.5 g of HN adsorbent with 50 mL of a 0.01 N NaCl solution (400 rpm). The initial pH was determined by titrating with NaOH or HCl solutions from 1 to 12. The solution is stirred for 24 hours [33, 34, 35, 40]. The initial pH and the final pH value of the obtained solution were measured using a pH meter. Based on the obtained results, pH curves were plotted (Fig. 11 a). As shown in figure 11 (a), it is possible to determine the pH zero charge value. According to the obtained results, the surface of the hazelnut shell is positively charged at a pH lower than the zero-charge value, and the surface of the hazelnut shell is negatively charged at a higher pH. Based on the obtained result, it is determined that the pH point of zero charge is 8. As seen from figure 11 (b), the optimal pH value of the adsorption process is 7.5 [40, 41]. These results agree with the results obtained by Boehm titration, which is used to determine the active acid and base groups on the adsorbent surface. The adsorption efficiency of the Fe_3O_4/hazelnut shell nanosorbent is significantly influenced by both pH and temperature, as these factors directly affect the surface charge of the adsorbent, the ionization state of the adsorbate, and the overall thermodynamics of the adsorption process. The pH of the solution plays a crucial role in determining the adsorption capacity by influencing the surface charge of the biosorbent and the speciation of adsorbates. At low pH values, the biosorbent surface tends to be protonated, acquiring a positive charge, which can enhance the adsorption of negatively charged species via electrostatic attraction. Conversely, at higher pH values, the surface may become negatively charged due to deprotonation of functional groups (e.g., hydroxyl and carboxyl), leading to repulsion of anionic adsorbates and reduced adsorption capacity. Temperature-dependent adsorption behavior reveals the energetic nature of the process, supporting chemical adsorption mechanisms. For adsorbents, t-tests were performed to compare the optimal pH and contact time values between biosorbent and bio-nanosorbent. Statistical significance was considered at the $p < 0.05$ level.

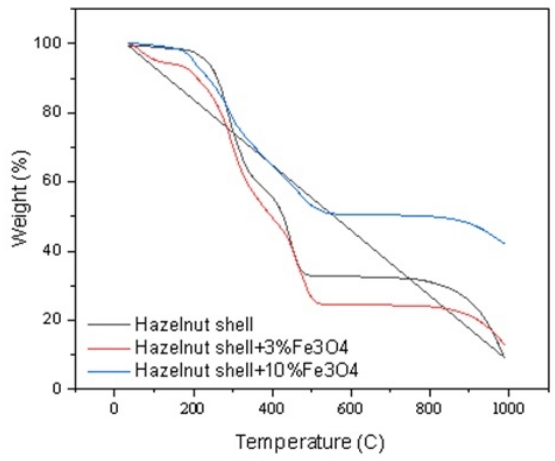

Figure 9. TGA curves of biosorbent (HN) and bio-nanoadsorbents (HN + 10%Fe_3O_4) obtained from pure hazelnut shell, hazelnut shell + 3% Fe_3O_4, and hazelnut shell + 10%Fe_3O_4.

To study the effect of temperature on oil sorption, sorption experiments were conducted using 0.5 g of hazelnut shell as a plant-based biosorbent and 0.5 g of hazelnut shell + 10% Fe_3O_4 as a bio-nanosorbent. An amount of 10 mL oil was mixed with 100 mL of oil-contaminated water at temperatures of 10 °C, 21.5 °C (room temperature), and 40 °C. It was determined that as the temperature increased, the sorption capacity decreased [42, 43]. So, statistically speaking, this means that as temperature increases, the pH environment changes, which affects the sorption environment.

As shown in figure 12, based on the obtained results, it was determined that 0.5 g of pure hazelnut shell absorbs 61.25% of 10 mL of oil at pH 7.5 and 21.5 °C. In comparison, 0.5 g of hazelnut shell combined with 10% Fe_3O_4 bio-nanosorbent has a maximum absorption capacity of 92.5% under the same conditions (pH 7.5 and 21.5 °C) [36]. In a 1:1 oil:water system, 0.5 g of hazelnut shell absorbs 54.1%, while 0.5 g of hazelnut shell + 10% Fe_3O_4 bio-nanosorbent absorbs 78.5% of the oil over the same period [44, 45, 46].

The experiment continued for 24 hours, depending on the amount of oil used. Specifically, after mixing 1, 2, 4, 6, 8, and 10 mL of oil with 100 mL of water, the experiment was repeated for 24 hours using hazelnut shell and hazelnut shell with 10% Fe_3O_4 bio-nanosorbent [47, 48, 49, 50, 51, 52]. As shown in figure 13, the conducted experiment demonstrates that the bioadsorbent achieves

Table 5. Langmuir isotherm model coefficients for HN and HN + 10%Fe_3O_4 adsorbent materials.

Model coefficients	HN	HN + 10%Fe_3O_4
b (L/g)	0.020	0.013
q_{max} (g/g)	3.26	4.9
R^2	0.988	0.992

an optimal absorption of 61.25% for 10 mL of oil over 24 hours, whereas the bio-nanosorbent absorbs 92.4%. By matching the obtained results with the statistical analysis data, it was determined that a 95% confidence interval was observed. This can be attributed to the bio-nanosorbent having a higher absorption capacity than the bioadsorbent. Also, the weight ratio used in figure 13 for the bioadsorbent is 0.5 g, and for the bio-nanoadsorbent, 0.5 g hazelnut shell + 10% Fe_3O_4. The experiments are repeated several times and the average value is found.

The Langmuir model is used to describe the adsorption process. The linear form of the Langmuir equation is as follows (Table 5) [52]:

$$q_e = \frac{q_{max} b\ \mathrm{Coil_e}}{(1 + b\ \mathrm{Coil_e})} \quad (2)$$

where, b (L/g) is the equilibrium constant of the Langmuir model correlated to the affinity of binding sites,

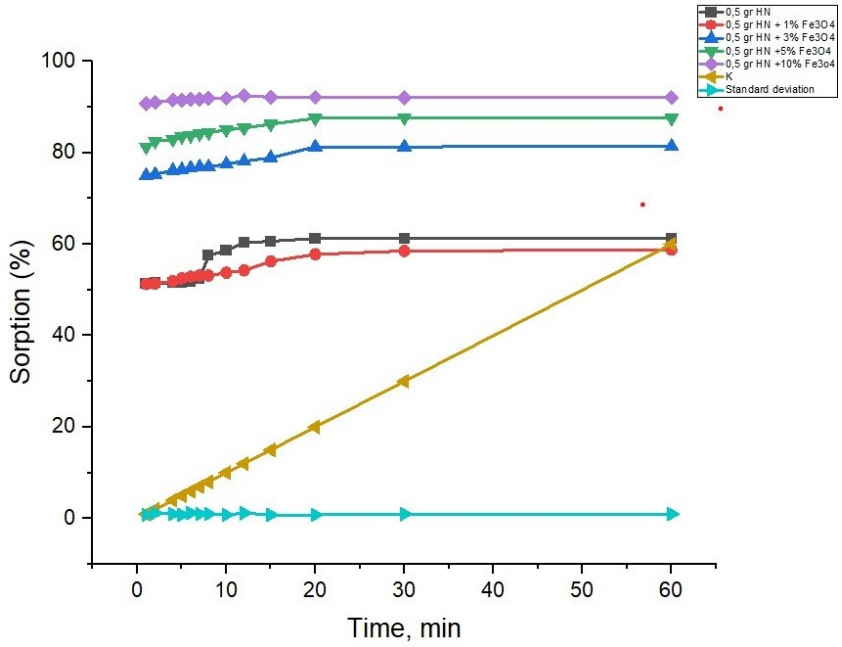

Figure 10. Graph of the time dependence of oil sorption by biosorbent and bio-nanosorbent (pH = 7.5).

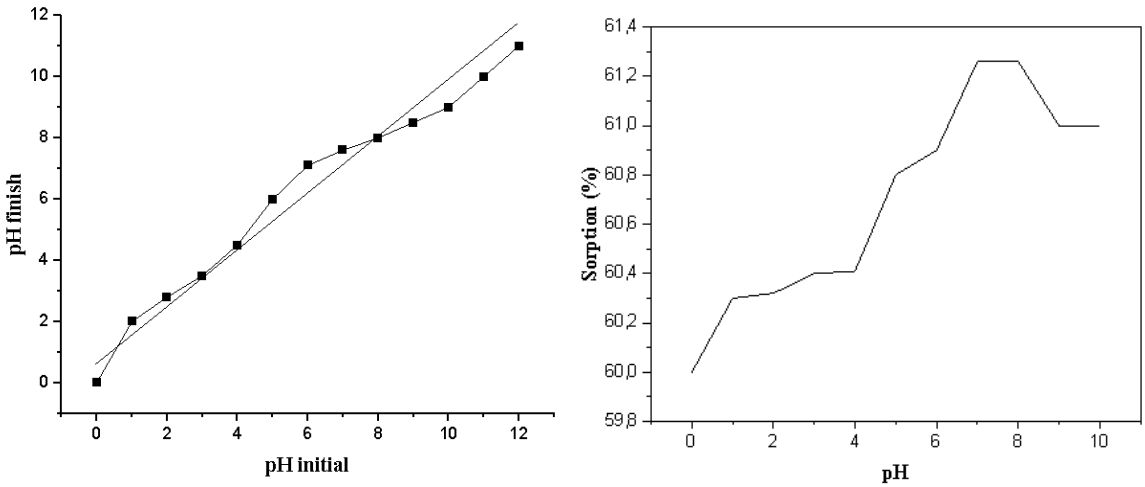

Figure 11. (a) Effect of pH on the adsorption of oil on HN + 10%Fe_3O_4 adsorbent and (b) zero charge pH point of HN + 10%Fe_3O_4.

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Table 6. Pseudo-first-order coefficients for HN and HN + 10%Fe_3O_4 adsorbent materials at different adsorption temperatures.

Temperature (°C)	HN		HN + 10%Fe_3O_4	
	k_1 (min^{-1})	R^2	k_1 (min^{-1})	R^2
10	0.36	0.75	0.35	0.98
20	0.31	0.88	0.32	0.92
40	0.16	0.88	0.13	0.93

Table 7. Langmuir separation factor (R_L) values.

Initial concentration (mg/L)	Langmuir constant b (L/mg)	$R_L = \frac{1}{(1+b\ \mathrm{Coil}_0)}$	Adsorption favorability
50 (HN)	0.02	0.5	Favorable
50 (HN + 10%Fe_3O_4)	0.013	0.606	Favorable

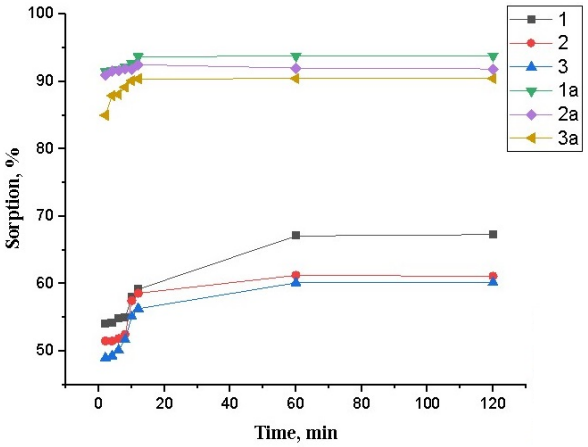

Figure 12. Temperature dependence of the oil sorption by hazelnut shell at different times: (1) 10 °C, (2) 21.5 °C, (3) 40 °C. Temperature dependence of the oil sorption by hazelnut shell + 10%Fe_3O_4 bio-nanosorbent at different times; (1a) 10 °C, (2a) 21.5 °C, (3a) 40 °C (pH = 7.5).

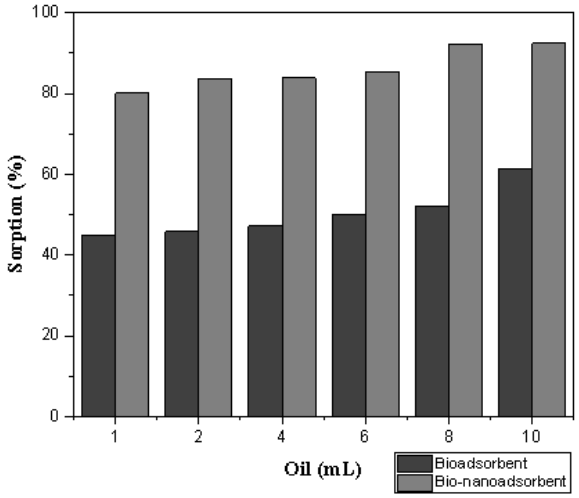

Figure 13. Graph of the oil quantity dependence in the adsorption process of biosorbent and bio-nanosorbent over 24 hours.

q_{max} (g/g) is the maximum adsorption capacity, q_e (g/g) is the equilibrium adsorption capacity and $Coil_e$ (g/L) is the equilibrium concentration of oil in the liquid phase [53]. R_L calculation formula:

$$R_L = \frac{1}{(1 + b\ Coil_0)} \tag{3}$$

The kinetic model is expressed as follows:

$$\frac{dq}{dt} = k_1(q_e - q) \tag{4}$$

where k_1 (min^{-1}) is the kinetic rate constant, and q is the amount of oil adsorbed per unit weight of adsorbent (g/g) at any time t or at the equilibrium (q_e) [53].

Adsorption isotherms were carried out at temperatures of 10, 20, and 40 °C. Considering the initial oil concentration, the dimensionless differentiation coefficient Coil, 0 (g/L), the coefficients for HN and HN + 10%Fe_3O_4 are 0.16 and 0.11, respectively. This indicates that spontaneous adsorption occurs in both cases, as confirmed by the RL calculation [52]. Regarding the temperature dependence, the tests were conducted using optimal adsorbent doses (0.5 grams of HN and HN + 10%Fe_3O_4, respectively) at controlled temperatures of 10 °C, 20 °C, and 40 °C. Figure 12 reports the removal trends over time (0-120 min) for HN and HN + 10%Fe_3O_4 (Table 6). It is clear that both materials reached a steady state within about 30 min and the nano-modified adsorbent exhibited higher oil removal capacity in the short term, even when adsorption was not favored (at 4 °C). In figure 12, the oil removal rate for HN + 10%Fe_3O_4 was similar to that measured with 0.5 g HN at 10 °C (Table 6). The initial values were well described by a pseudo-first-order kinetic model expressed as (4). The adsorption mechanism is primarily physisorption, supported by the pseudo-first-order kinetics and the lack of intraparticle diffusion as evidenced by the reduced adsorption capacity at higher temperatures (figure 12). It has the potential for efficient oil utilization [53].

The Langmuir isotherm is more suitable for modeling

monolayer and homogeneous surfaces, the Freundlich isotherm for heterogeneous surfaces and non-ideal adsorption, and the Temkin isotherm for modeling adsorption heat. The Langmuir isotherm is preferred because it is a homogeneous surface mixed with oil.
Table 7 also shows the adsorption efficiency. Since the results are in the range $0 < RL < 1$, adsorption is considered favorable.

4. Conclusions

It has been determined that the hazelnut shell, which we use as a plant-based waste for oil sorption, is a lignocellulosic biosorbent composed of 28.9% cellulose, 11.3% hemicellulose, and 30.2% lignin. To find the optimal conditions for sorption, the optimal amount of biosorbent, oil absorption mass, optimal absorption time, and the effect of pH were experimentally investigated. The optimal conditions for 0.5 g biosorbent sorption were pH 7.5 and 30 minutes, resulting in 61.25% adsorption. Thus, to increase the adsorption capacity of the biosorbent, we synthesized a new bio-nanosorbent based on Fe_3O_4 superparamagnetic nanoparticles and determined its significance and effectiveness in cleaning oil from water. The optimal conditions for the oil removal process were determined using a bio-nanosorbent synthesized from hazelnut shells and Fe_3O_4 superparamagnetic nanoparticles. The optimal adsorption conditions for the 0.5 g bio-nanosorbent were pH 7.5 and 12 minutes, with 92.5% adsorption, as observed in the experiment. It was concluded that the bio-nanosorbents synthesized from hazelnut shell + Fe_3O_4 nanoparticles are based on cost-effective, low-waste, and waste-free technology. The research work poses both technical and non-technical obstacles (the long drying time of the hazelnut shell, the subsequent crushing process of the shell, etc.) during its implementation, one of which is that agricultural waste such as hazelnut shells varies in chemical composition depending on the variety, region, and crop conditions, which affects the consistency of performance.

In conclusion, hazelnut shells are a promising, sustainable adsorbent option, especially for applications where biodegradability and low environmental impact are priorities. Hazelnut shells are high biodegradability, with organic components that naturally decompose in the environment over time, making them a good candidate for eco-friendly applications.

Several limitations can be considered in the study of the synthesis of hazelnut shell-based biosorbents and bio-nanoadsorbents for oil sorption from water, for example, oil-contaminated water may contain surfactants, detergents, or other chemicals that may interfere with the oil sorption process. These substances were not considered in the research process.

Authors contributions
Authors have contributed equally in preparing and writing the manuscript.

Availability of data and materials
The authors declare that the data supporting the findings of this study are available within the paper.

Conflict of interests
The authors assert that they do not have any identifiable conflicting financial interests or personal relationships that might be perceived to influence the work presented in this paper.

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