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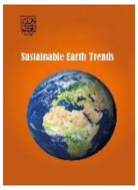

Synthesis of zinc-reinforced carbon nanotubes for degradation of erythromycin antibiotic

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ABSTRACT

The growth of urban communities, the expansion of industrial activities, and the adverse effects of human activities on limited water resources have endangered the lives of humans and other living organisms. In recent years, in addition to common pollutants introduced into nature by humans, emerging pollutants have also appeared as a serious challenge. Among these pollutants are pharmaceutical residues. The presence of these types of pollutants and their increasing quantities, given the inability of conventional purification methods to decompose them, has led researchers to focus their attention on new and efficient methods. Therefore, this research aimed to remove the pharmaceutical pollutant erythromycin from aquatic environments using zinc-reinforced carbon nanotubes. The properties of these carbon nanotubes were determined by FESEM, VSM, FTIR, and XRD analyses. A pH of 3 and a catalyst dose of 0.075 g/L with a concentration of 10 mg/L for 60 minutes were the optimal parameters obtained in the experimental process. The fabricated photocatalyst was investigated in contact with the pollutant erythromycin at different concentrations and at different times in the presence of UVC light, and the results of the photodegradation tests showed the remarkable performance of zinc-reinforced carbon nanotubes in eliminating Erythromycin by 94%. Also, at a concentration of 10 mg/L of Erythromycin, after 4 consecutive cycles, the degradation efficiency of this zinc-reinforced carbon nanotube was 70%. Therefore, the use of this photocatalyst can be suggested as a useful method with acceptable efficiency for removing antibiotics from wastewater.

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1. Introduction

Due to the scarcity of water resources and the increasing expansion of industrial and commercial units, the increase in the production of industrial wastewater and the contamination of water resources are considered social and economic problems (Sayadi et al., 2019). Widespread pollutants, including organic and inorganic compounds, cause water pollution. Extensive contaminants, including organic compounds and minerals, also cause contamination. Medications are one of the most important environmental pollutants because some are weakly soluble or insoluble, which makes them resistant to degradation in water treatment processes. These drugs are present in the urine samples of those who use them and can enter other environments (Fatta et al., 2007). Among them, pharmaceuticals and personal care products have created an important class of emerging pollutants due to their high consumption and increased release into wastewater (Chaturvedi et al., 2021). The reasons for the spread of drugs in water include the therapeutic use of drugs by humans and animals, personal and health products, pharmaceutical industry wastewater, and hospital wastewater (Khan et al., 2021). The widespread presence of various types of drugs in small amounts in aquatic environments has increased public concerns because the long-term effects of pharmaceutical waste on humans and the ecosystem are still unknown (Rosi-Marshall et al., 2015). In recent years, various types of pharmaceutical antibiotics, such as erythromycin, have been widely used to prevent and treat infectious diseases (Daghrir and Drogui, 2013). Antibiotics are widely used in the treatment of infections caused by viral diseases, chronic diseases, and infectious diseases (Babu et al., 2013; Yang et al., 2020).

Erythromycin (ERM) is a macrolide that is widely used as an antibacterial antibiotic and is produced by the gram-positive bacterium *Saccharopolyspora erythraea* (Omura, 2002). Erythromycin antibiotics are widely used in veterinary medicine and aquaculture due to their low cost and high antimicrobial activity; they are also used as feed additives to promote growth in animals (Schafhauser et al., 2018). Over the past few decades, various methods have been used to degrade erythromycin from wastewater, including biological treatment, adsorption, coagulation, and flocculation methods, membrane processes, and advanced oxidation, all of which have limitations (de Ilurdoz et al., 2022).

Advanced oxidation is also used to degrade various types of pollutants such as pharmaceuticals (Rivera-Utrilla et al., 2013). Numerous studies in the field of water and wastewater treatment using nanotechnology have been increasing due to the extraordinary properties of these materials. Therefore, recent advances in the field of nanotechnology have enabled the further development of the next generation of water supply systems and have led to the provision of the required water by less expensive and new treatment methods, which in turn can overcome the problems encountered by conventional treatment methods (Mauter et al., 2018). One of the nanotechnology methods for removing water pollution is the use of nanophotocatalysts. This method, as one of the advanced oxidation methods, has shown very high potential for material degradation (Sillanpää et al., 2018). The most important advantages of using photocatalysts include the absence of multi-cycle metabolite formation, complete advanced oxidation of pollutants within a few hours, availability of catalysts with high activity, practicality in environmental conditions, ability to use sunlight, and cost-effectiveness and efficiency (Sillanpää et al., 2018; Liu et al., 2020). Khalatbary and colleagues conducted chemical oxidation of MWCNTs under treatment with different reagents. According to the results, after the oxidation treatment, various functional groups such as carboxylic (-COOH), carbonyl (-C=O), and hydroxyl (-OH) groups were formed on the surface of MWCNTs (Khalatbary et al., 2022 and 2024). According to the study by Rosca and his colleagues, the oxidation of MWCNTs in nitric acid and the effect of various process parameters on the solubility of MWCNTs were investigated. The experimental results showed that the solubility was mediated by the functional groups on the surface of MWCNTs during the degradation of the nanotubes (Rosca et al., 2005). Lu and his colleagues oxidized MWCNTs using different types of chemical agents, and the results showed that these modifications resulted in better performance for the adsorption of Zn^{2+} on MWCNTs (Lu and Chiu, 2008).

Therefore, due to the necessity of removing drug pollutants from water resources, nanotechnology and photocatalytic methods are considered to be the most well-known and successful technologies in this field. This method works faster, easier, and cheaper, without destroying the molecular structure. Therefore, in this study, the photocatalytic destruction of the antibiotic erythromycin in a blue solution using modified carbon nanotubes was investigated.

2. Materials and methods

2.1. Materials

Sulfuric acid and nitric acid, iron nitrate ($Fe(NO_3)_3.9H_2O$) (Purity 98%), sodium hydroxide (NaOH) (Purity 99%), sodium dodecylbenzene sulfonate (Purity 98%), and distilled water are also the chemicals used in the synthesis of zinc-reinforced carbon nanotubes. In this study, all chemicals used except distilled water were obtained from Merck, Germany. Erythromycin manufactured by Iran Darou was purchased. A solution of H_2SO_4 and NaOH was used to adjust the pH.

2.2. Synthesis of magnetic carbon nanotubes

First, 2g of multi-walled carbon nanotubes were weighed using a digital balance (Kern, ABJ220-4M, Japan) and added to a solution of sulfuric acid and nitric acid with a volume ratio of 3 to 1. The resulting suspension was placed in an ultrasonic bath (Parsonic 7500 S, 220 VAC, Iran) for 3 hours at room temperature, then the resulting suspension was diluted to 50% and filtered through a 0.45 µm filter using a vacuum (Hailea, ACO-5504, China) device.

The carboxylate functionalized multi-walled carbon nanotubes were washed several times with double-distilled water until reaching neutral pH (pH meter: Istek, 915PDC, Korea) and dried under vacuum conditions at 50°C for 12 hours. To make magnetized carbon nanotubes, first 0.65 grams of $Fe(NO_3)_3.9H_2O$ was dissolved in 20 ml of 100% pure ethanol solution. Then, the resulting carbon nanotubes were sonicated in a mass ratio of 1 to 4 in the resulting iron salt solution for 3 hours. 20 ml of 1.2 mM sodium dodecylbenzene sulfonate solution was added to the iron salt and carbon nanotube solution and mixed under a magnetic stirrer (Mtops Ms300hs Magnetic Stirrer, Korea) for 30 minutes. The resulting mixture was placed in an oven (Shimaz Electric, Iran) at 100°C for 3 days. The resulting powder was washed several times with ethanol and dried at 50°C (Hoa, 2018).

2.3. Modification of magnetized carbon nanotubes with zinc

In order to prepare the photocatalyst (zinc modified carbon nanotubes), a mass ratio of 2:1 of magnetized carbon nanotubes to $ZnSO_4$ was used. Some of the magnetized carbon nanotubes were dissolved in distilled water, then the pH was kept constant in the range of 11 to 12 using a mixed solution of H_2SO_4 and NaOH. The temperature of the solution was kept constant in a hot water bath at 60°C, and while it was being mixed with a stirrer at a constant speed of 400 rpm (Centurion Ltd, K2042, England), $ZnSO_4$ was added to the solution for 30 minutes at a rate of one drop per second. Then, the solution was left at the same temperature for another 30 minutes (Mahmoodi, 2014).

2.4. Characterizations of synthesized modified carbon nanotube

X-ray diffraction (XRD) is a rapid analytical technique used to identify the type of material, as well as its phase and crystalline properties. The XRD pattern is the intensity of the peaks versus the diffraction angle from which the arrangement of the atoms of the material and the formation of its crystal can be estimated. In this study, XRD analysis with X-ray radiation was used to examine the size of the particles and determine the phases (Rigaku MiniFlex 600). Field emission scanning electron microscope (FESEM, TE-SCAN MIRA3), this microscope is one of the most famous types of electron microscopes that uses electron beams to produce images of objects as small as 10 nanometers for study. The image of this microscope is the result of the collision of high-energy electrons with the surface and the production of a signal. It scans the surface of the sample and provides information about the type and size of the grain size of the sample particles and its morphology. Infrared spectroscopy (FTIR) is based on the absorption of radiation and the study of vibrational mutations of molecules and polyatomic ions using a Shimadzu FT-IR1650 spectrophotometer. This spectrum was carried out in the range of 400-4000 ($^{-1}$cm). Vibrating Sample Magnetometer VSM analysis is the main method for studying the magnetic properties of materials (Lake Shore 7403, Westerville, USA). The result of this analysis is to obtain a hysteresis curve or remanence loop of the material, which can be used to calculate data such as coercivity, saturation magnetization and magnetic permeability and to identify the magnetic classification of the material such as ferromagnetic, paramagnetic and super magnetic.

2.5. Photocatalyst experiments

The experiments were conducted in a 100cc cylindrical glass container that was completely insulated from light. The light source inside this photoreactor was 6-watt UVC lamps. To prevent direct contact of the solution with the lamps, the radiation source was placed in quartz tubes. The solution was homogenized and aerated using an air pump. Sampling from the photoreactor was possible using a syringe and through the opening at the top of the reactor. In order to investigate the ability of the synthesized nanoparticles to degradation pharmaceutical contaminants in the photocatalyst process, solutions with a specific pH, a specific concentration of the contaminant, and a known amount of nanoparticles were first poured into the reactor. Then the desired solution was placed in the dark for 30 minutes to determine the amount of surface adsorption. After that, the lamp was turned on and 5 cc of the solution was degradation from the reactor at intervals of 5, 15, 30, 60, and 90 minutes. When the sample was degradation from the reactor, its nanoparticles were separated and then the residual amount of the contaminant was read using a spectrophotometer. The percentage of contaminant degradation is calculated from the following Eq. 1:

$$\mathrm{R}\,(\%) = \frac{(\mathrm{C}_0 - \mathrm{C}_t)}{\mathrm{C}_0} \times 100 \qquad (1)$$

In this formula, R is the percentage of contaminant degradation, C_0 is the initial concentration, and C_t is the contaminant concentration at any time.

3. Results and discussion

3.1. Characterizations

3.1.1. X-ray diffraction (XRD) analysis

As shown in Fig. 1, the peaks (268) and (046) at θ2 = 13.46° and θ2 = 89.56° are the characteristic diffraction peaks of carbon nanotubes, respectively. The diffraction peaks at 78.44° and 77.69° indicate that magnetic iron oxides are coated on carbon nanotubes. All these peaks confirm the presence of iron, zinc, carbon and zinc carbonate elements. This graph is also consistent with the results of FTIR graph (Li et al., 2017).

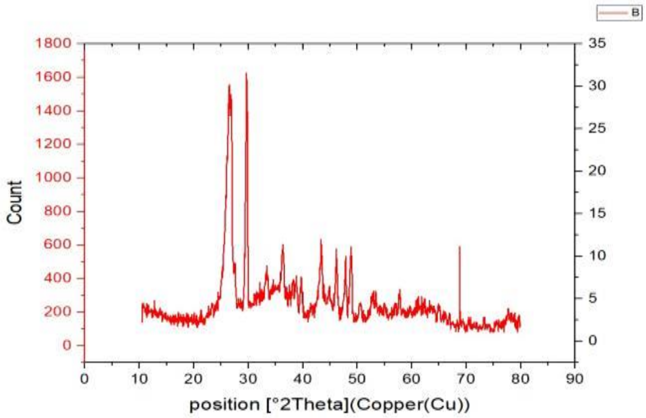

Fig. 1. XRD patterns of zinc-reinforced carbon nanotubes.

3.1.2. Field emission scanning electron microscopy (FESEM) analysis

Using FESEM analysis, the size of the nanoparticles, their morphology and the way they are arranged on the surface of their bodies were shown. As shown in the Fig. 2, their shape is almost spherical and intertwined. It should be noted that the aggregation of the particles is due to their magnetic attraction and it is believed that these granular materials are magnetic iron oxide and zinc (Li et al., 2017). EDS analysis was used to identify the elements of the synthesized sample. The elements Fe, O, C and Zn were observed in the elemental map of the carbon nanotube magnetized with zinc (Table 1).

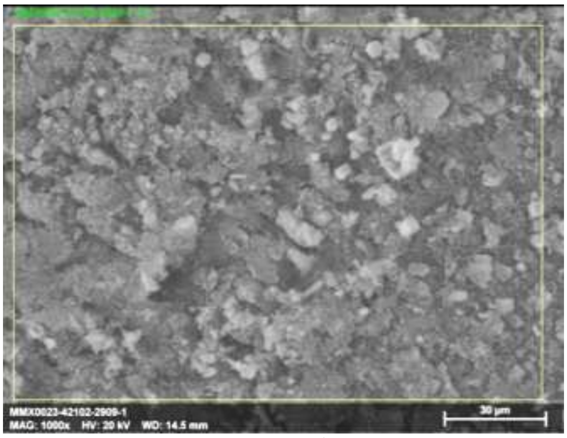

Fig. 2. Scanning electron microscope analysis for particle morphology identification.

Table 1. EDAX analysis.

Element	C	O	Fe	Zn
Percentage	41.20	29.75	17.49	11.56

3.1.3. Infrared spectrometer (FTIR) analysis

The FT-IR spectrum is shown in Fig.3. The wavenumber bands at 1571, 3433 and 1115 $^{-1}$cm for CNTs indicate the appearance of stretching of C=C, –OH and C-F groups, respectively (Tselepidou et al., 2012). After combining CNTs with Ca, the –OH peak shifts to a lower frequency (1415 $^{-1}$cm). The –C=C peak is at 1571 $^{-1}$cm (stretching vibration). In addition, there is a C-H bending vibration band at about 854 $^{-1}$cm, an O-H bond stretching vibration band at about 1415 $^{-1}$cm, a C=C group bending vibration band at about 966 $^{-1}$cm, and a C-H bending vibration band at about 643, 782, and 854, indicating that carbon nanotubes are present with iron oxides through hydrogen and carboxyl bonds in this compound (Fan and Xin, 2012).

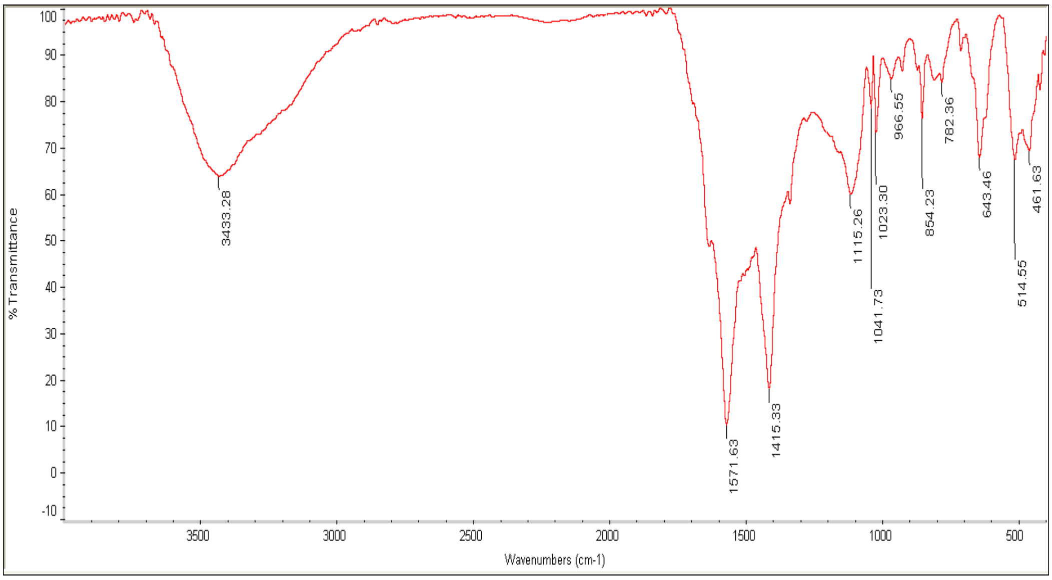

Fig. 3. Infrared spectrometer (FTIR) analysis.

3.1.4. Vibrating sample magnetometer (VSM) analysis

As shown in Fig. 4, hysteresis loop is one of the important curves that reflect the magnetic properties of materials. This magnetic curve shows that this carbon nanotube is paramagnetic. Its saturation magnetization was 1.25 emu/g at room temperature, which proves that magnetic iron oxides are coated on carbon nanotubes (Li et al., 2017).

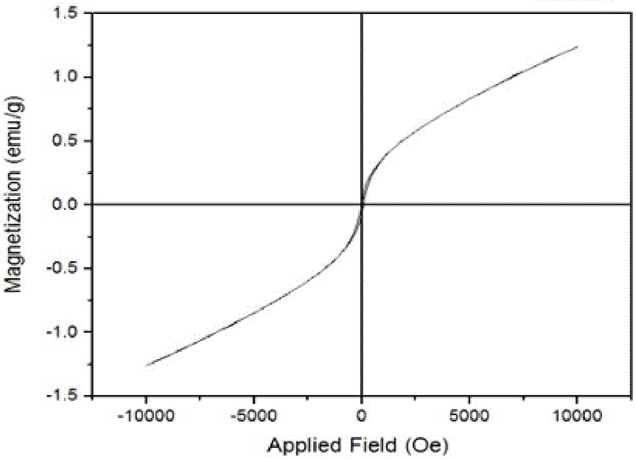

Fig. 4. Hysteresis loop and VSM analysis.

3.2. Photocatalytic experiments

3.2.1. The effect of pH

pH is one of the parameters that has a great influence on chemical processes. Among the effects of this factor, solubility, surface charge and production of hydroxyl radicals can be mentioned. Considering that hydroxyl radicals play a major role in the efficiency of the photocatalyst process, the acidic to alkaline range was investigated in this study. (All optimization experiments were initially kept in the dark for 30 minutes). For this purpose, the amount of this parameter was set to 3, 5, 7, 9, 11, and then the degradation rate of Erythromycin with a concentration of 0.02 g/L and a nanoparticle amount of 0.05 g/L was measured at time intervals of 5, 15, 30, 60 and 90 minutes.

Fig. 5 shows the effect of pH changes on the percentage of Erythromycin degradation. As can be seen in the figure, the highest degradation efficiency occurs at pH = 3. The maximum efficiency is 88% according to the experimental conditions. This value occurs after 90 minutes from the start of irradiation. The degradation rate after one hour is 86%, meaning that only 2% is added to the degradation efficiency after half an hour. At neutral pH, the lowest degradation rate of Erythromycin (77%) is observed after 90 minutes. In general, the degradation rate of the contaminant is higher in the acidic range than in the alkaline range. As mentioned, pH plays a very important role in the efficiency of chemical processes. The main effect of this parameter is on the surface charge of the particles in the reaction medium. Considering that in the photocatalytic process, the radicals produced, especially the hydroxyl radical, play a fundamental role, therefore, according to the results, it can be concluded that in these neutral and alkaline pHs, the rate of production of this radical is low and as a result, the percentage of degradation decreases. The high efficiency in acidic pH can be related to the following reactions (Eqs. 2-5):

$$2HO_2^{\circ} \rightarrow O_2 + H_2O_2 \quad (2)$$

$$H_2O_2 + O_2^{\circ-} \rightarrow OH^{\circ} + OH^- + O_2 \quad (3)$$

$$e^- + O_2 \rightarrow O_2^{\circ-} \quad (4)$$

$$O_2^{\circ-} + H^+ \rightarrow HO_2^{\circ} \quad (5)$$

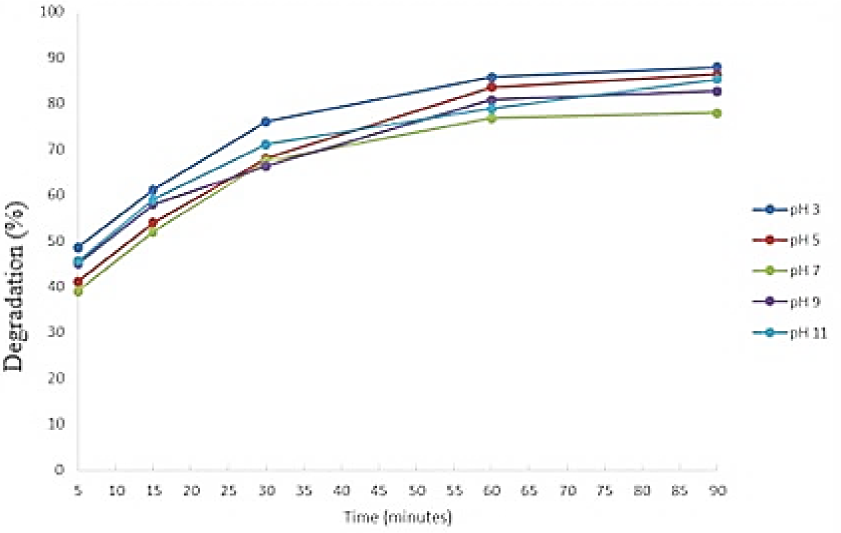

Fig. 5. Effect of pH on degradation efficiency (catalyst dose 0.05 g/L, Erythromycin concentration 0.02 g/L).

According to the above reactions, it can be stated that in acidic conditions, HO_2 is converted to hydroxyl radical. On the other hand, it can be said that in acidic conditions, the aggregation rate of photocatalyst particles is greatly reduced, which leads to an increase in the useful surface area of the catalyst. In addition, in acidic conditions, the surface charge of Erythromycin molecules is positively charged, which allows for greater reaction with hydroxyl ions. The results obtained are consistent with those of (Qi et al., 2018).

3.2.2. The effect of photocatalyst amount

Fig. 6 present the effects of catalyst amount on the Erythromycin degradation process. In order to obtain the optimum point for the photocatalyst amount, the amounts of 0.01, 0.025, 0.05, 0.075, and 0.1 g of the synthesized catalyst were used to degradation 0.02 g/L of Erythromycin at pH 3 in the photocatalyst process to determine the highest degradation efficiency and, consequently, the optimal amount of photocatalyst. In the context of antibiotic removal, Rivera-Utrilla et al., 2013 found that carbon-based nanomaterials could adsorb up to 25% of certain antibiotics before photocatalytic activation. It is necessary to note that the experiments related to photolysis alone, i.e., without the use of a catalyst, were investigated and showed a maximum degradation of 4.99% in 90 minutes.

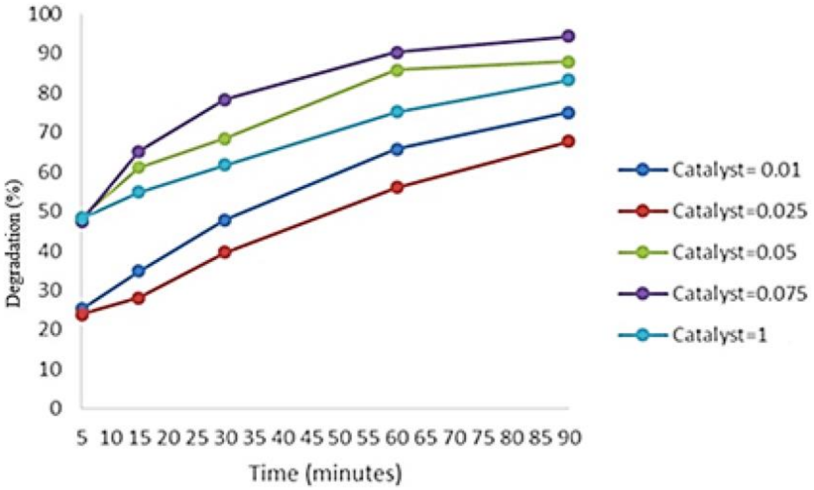

Fig. 6. Effect of catalyst dosage (g/L) on degradation efficiency (pH 3, Erythromycin concentration 0.02 g/L).

As can be seen in the Fig. 6, the highest degradation efficiency (94%) was observed 90 minutes after the start of irradiation and at 0.075 g/L of photocatalyst. The lowest degradation efficiency was also observed at 0.1 g/L (83%). Considering that increasing the amount of catalyst in the reaction medium leads to an increase in empty sites for pollutant adsorption and also increases the production of hydroxyl radicals, it is observed as shown in the figure that with an increase in the amount of pollutant, the degradation efficiency increases, but the continued increase leads to high turbidity of the medium and, as a result, a decrease in radiation penetration, which in turn leads to a decrease in the pollutant destruction efficiency in the presence of a higher amount of catalyst. The results of the current study are similar to the results of (Chamanehpour et al., 2023).

3.2.3. The effect of pollutant concentration

To investigate the effects of pollutant concentration on the degradation efficiency of the photocatalyst process, different concentrations of Erythromycin (10, 20, 30, 40, and 50 mg/L) at pH 3 and 0.075 g/L of catalyst were used in the photocatalyst degradation process. As can be seen in Fig. 7, the degradation efficiency decreases with increasing pollutant concentration. Within 60 minutes of the start of irradiation, all the drug present in the solution with a concentration of 10 mg/L is degradation. The reason for this is that with an increase in the initial pollutant concentration, the amount of active sites on the photocatalyst surface decreases, so the production of hydroxyl radicals and subsequent pollutant degradation decreases. In addition, an increase in pollutant concentration leads to the production of intermediates, which itself is one of the factors that reduce the rate of pollutant degradation. Azizi et al. achieved similar results in the photocatalytic degradation process of clindamycin using titanium dioxide (Azizi et al., 2024).

3.3. Photocatalyst reusability

One of the important parameters in the use of nanoparticles for pollutant degradation is their reusability for the process. This is especially important in the study of catalyst economics. Since the synthesis of nanoparticles for specific applications may be time-consuming or expensive, it is certainly important to use a catalyst that maintains its capabilities repeatedly. Fig. 8 shows the reusability of the synthesized catalyst over 4 consecutive cycles. As the figure shows, using 0.075 g/L of catalyst in a solution with a concentration of 0.02 g/L of Erythromycin at pH 3, the overall efficiency of the process decreases from 94% to 71%. The results show that the zinc-doped magnetic carbon nanotubes maintain their ability to degradation Erythromycin well even after 4 cycles of use. The reason for the decrease in the ability to degradation pollutants from the 4th cycle onwards can be attributed to the occupation of active sites on the catalyst surface by intermediates. In 2019, Fred et al. observed a decrease in the ability of the catalyst to degradation pollutants after 4 cycles of using the catalyst in the photocatalytic degradation of the antibiotic ciprofloxacin by the photocatalyst $CuFe_2O_4$ @methylcellulose (Fard et al., 2023).

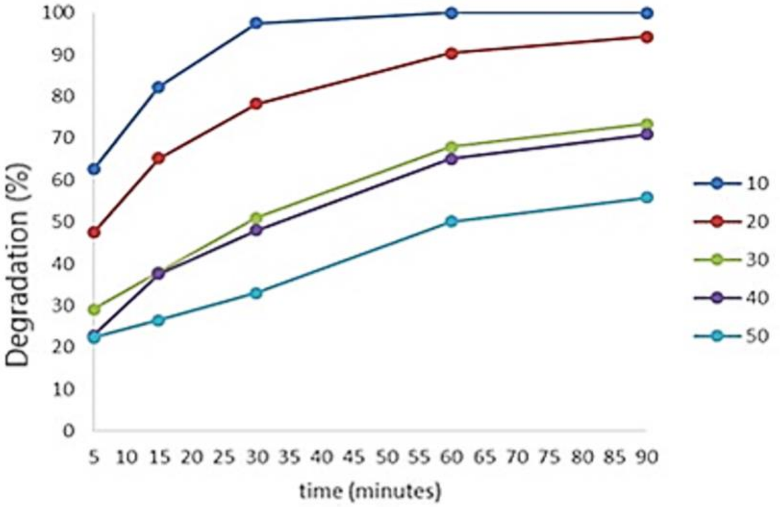

Fig. 7. Effect of pollutant concentration (mg/L) on degradation efficiency (pH 3, catalyst concentration 0.075 g/L).

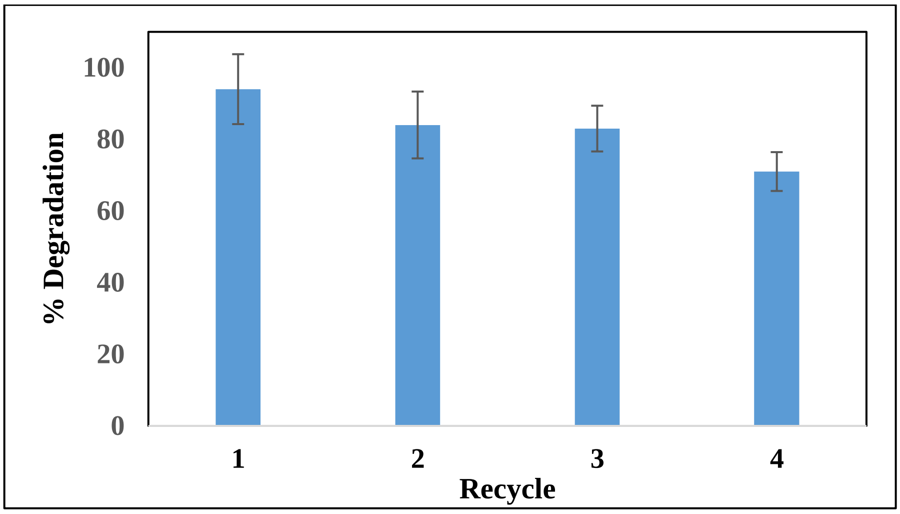

Fig. 8. Reusability of the photocatalyst.

4. Conclusion

In this study, magnetic carbon nanotubes doped with zinc were used for the degradation of erythromycin. First, the carbon nanotubes were magnetized by iron salts, then to increase its efficiency, they were doped using zinc salts. The synthesized nanotubes were investigated using specific nanoparticle identification analyses. For this purpose, SEM analyses combined with EDS were used. The results showed that the nanoparticles had a uniform morphology consisting of carbon, zinc, iron and oxygen. The presence of different functional groups was confirmed by FTIR results. The crystal structure of the synthesized carbon nanotubes was also determined by XRD. VSM results also showed the magnetic capability of the carbon nanotubes. Photocatalytic experiments for the degradation of erythromycin were conducted using a single-agent method at a time. A photoreactor equipped with a UV lamp was used to conduct these experiments. The optimal parameters obtained in the experimental process were pH 3 and a catalyst dose of 0.075 g/L with a concentration of 10 mg/L of the pollutant for 60 minutes, according to which this carbon nanotube was able to degradation 94% of erythromycin from the environment. One of the most important parameters from an economic point of view and for use on an industrial scale is the reusability of the synthesized photocatalysts. For this purpose, the ability of magnetic carbon nanotubes doped with zinc was tested in four consecutive cycles. The results showed that even after 4 cycles of using this nanotube, the degradation ability of more than 70% remained for the degradation of erythromycin. Therefore, it is suggested that the synthesized nanoparticle can be used to degrade antibiotics.

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