Revista de Chimie
https://revistadechimie.ro
https://doi.org/10.37358/Rev. Chim.1949

The Use of Magnetite Iron Oxide Nanoparticles in Water-Oil Separation

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Abstract: *Oil contaminations are a problem that Iran, as a country having oil reserves, faces it. These contaminations mostly occur in aquatic ecosystems and cause the death of many aquatic organisms, their entrance in the food chain, and health problems in human societies. Many solutions have been proposed for this problem, and using nanoparticles has been introduced as a new approach. In this project, iron oxide nanoparticles have been synthesized in order to be used in separating petroleum from water. These nanoparticles are biocompatible and could be directed and recovered by magnetism. Based on our experiments, nanoparticles were successful in separating petroleum from water. Furthermore, the recovered petroleum was analyzed by Gas Chromatography and showed no significant change. According to the results of the current investigation, the separated petroleum could be recovered and returned to the economic cycle of the country and inhibit the loss of national wealth.*

Keywords: *Oil Pollution, Aquatic Ecosystem, Iron Oxide Nanoparticles, Oil Recovery*

1.Introduction

Oil pollution is one of the unavoidable problems facing oil-rich countries. Iran is no exception. One of the most threatened ecosystems is the aquatic ecosystem. According to the head of the Caspian Sea Research Institute, 122,350 tons of oil pollution enters the Caspian Sea annually, which is imposed on the Caspian Sea through tankers, oil inlets, and outlets, exploration and extraction wells that do not use advanced technology [1]. The Persian Gulf and surrounding areas are no longer safe from these contaminants. According to Dr. Ismail Kahrom, an environmentalist, the Persian Gulf is 47 times more polluted than open waters due to a variety of oil and industrial activities [2,3]. Ballast water inflow is one of the main sources of pollution in the Persian Gulf. And we've always heard bad news from around the world, such as the April 2010 coastal drilling explosion in the Gulf of Mexico that led to the formation of the worst oil spill in American history [4]. Pollution in the Gulf of Mexico has killed more than 6,000 birds and 600 lbs. Many aquatic animals and organisms are at risk for these contaminants and can enter the food chain based on Figure 1 [5-7].

Figure 1. Living organisms exposed to oil pollution

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These contaminants not only cause environmental problems but also pose a great threat to human societies. Petroleum leaks with the release of petroleum vapors cause the inhalation of toxic substances by individuals, which leads to burns, skin disorders, and higher levels of cancer [8, 9]. The oil contains many volatile and toxic chemicals, some of which can be inhaled by pregnant women, which can lead to premature birth, low birth weight or miscarriage, and babies exposed to ethylbenzene, a toxic substance in crude oil, are at higher risk for congenital heart disease. Corexit, as one of the toxic and dangerous compounds that are released into the sea due to oil spills, can cause serious lung problems [10, 11].

Today, many measures have been taken to eliminate oil contamination in water, including the use of natural and synthetic adsorbents, mechanical methods, catalytic refractory methods, the use of infrared bacteria, and the use of nanoparticles as active catalysts [12].

Nanoparticles have a diameter between 1 and 100 nanometers, which means they are in a field between the quantum effects of atoms and molecules and the properties of masses. Nanoparticles show different and interesting properties by increasing the ratio of surface to volume [13, 14].

One of the most important groups of nanoparticles is magnetic nanoparticles, which can be controlled and separated by magnetic force due to their magnetic properties. They have many unique magnetic properties. Magnetic nanoparticles have attracted the attention of many researchers from a variety of disciplines. The most important applications of magnetic nanoparticles are in data storage, catalysis, and environmental applications [15].

Magnetic iron oxide nanoparticles have a high surface-to-volume ratio and high surface energy. Therefore, they tend to integrate to reduce surface energy. In the last decade, more research has been done on several types of iron oxide in magnetic nanoparticles, mostly magnetite, because they are so biocompatible [16]. Many methods are used to synthesize this type of nanoparticles, which include thermal decomposition, microemulsion, hydrothermal synthesis, photoreceptor synthesis, reverse micelles, and convection [17]. In this study, as a novel strategy, it was tried to find a new way to separate oil from aquatic environments has become an important and practical need. In this regard, first, magnetic nanoparticles of iron oxide with the practical purpose of separating oil from water were synthesized, and the process of separating oil from the water was investigated. According to the results of this study, separated oil can be recovered, and it can be returned to the country's economic cycle and prevent the waste of national capital [18].

2.Materials and methods

2.1. Materials and equipment

The certified oil was obtained from the Petroleum Industry Research Institute. Heptahydrate water sulfate (99%), hexahydrate iron chloride (99%), four molar Ammonia (25%), hydrochloric acid (37%), and normal hexane were purchased from Merck, Germany. Field Emission Scanning Electron Microscope (FE-SEM) S-4160 model by Hitachi with 30 KV power and 5 nm accuracy, and Transmission Electron Microscope (TEM3) CM30 model by Philips with 300 KV power, and chromatography gas device GC-15A is from Shimatzo Japan with FID detector and Rtx-5 MS column with a length of 30 meters [19].

2.2. Method

2.2.1. Method of Synthesizing Iron Oxide Nanoparticles

In this study, the ratio of 1 to 2 of heptahydrate iron sulfate to hexahydrate iron chloride in 1500 mL of distilled water was used and dissolved rapidly on a mixer with a 5 cm magnet. First, the *p*H (pH-meter, Seven Easy, Mettler Toledo) of the solution was measured (*p*H = 6.5), and ammonia was used to create alkaline conditions. Then, to prevent the reaction time from increasing, with the help of two boards that were stacked side by side, the increase of four molar ammonia was done slowly. The bolts were adjusted so that the droplets of both burettes were inserted in the center of rotation of the magnet, and also the droplets were dripped successively (Figure 2).

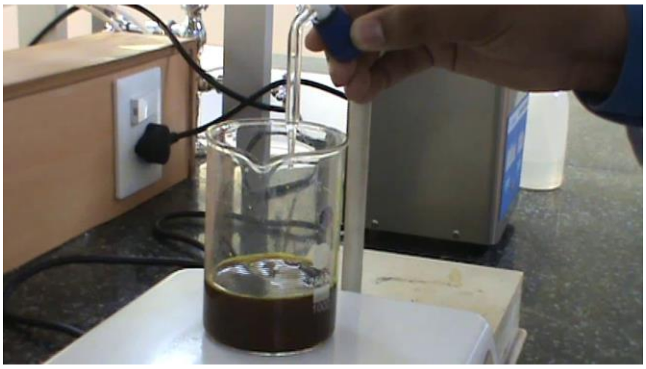

Figure 2. Synthesis of iron oxide nanoparticles

When the *p*H reached about 10, it was reduced by using hydrochloric acid to a pH of 3, and then the nanoparticles produced by the strong magnet were settled, and after several washes in distilled water, they were transferred to dry in a 50 °C oven [20].

2.2.2. Methods of examining oil separation from water in the presence of magnetite nanoparticles

In two Erlenmeyer flasks, a mixture of 250 mL of oil and water was mixed in equal proportions, and 1 mg of nanoparticles was suspended in water by an ultrasonic device and added to a mixture of oil and water in one of the samples. The samples were placed on a shaker for 24 h [21].

2.2.3. Preparation of samples and devices for gas chromatographic analysis

One of the best ways to separate petroleum compounds is to use organic solvents. Oil and water samples in both Erlenmeyer flask containing nanoparticles and Erlenmeyer control flask were separated using a separating funnel. Hegzan normal solvent was used to identify petroleum compounds in oil and water samples. To each of the Erlenmeyer flasks, 30 mL of normal hexane was added, and after mixing, a two-phase separator funnel was formed, and the hexane phase was separated. Chromatographic gas devices are used to quantify the organic compounds of oil. To measure the available compounds using a chromatographic gas device, the temperature of the injection site and the detector were set at 300 °C. The oven temperature started at 80 °C and increased by 10 °C/min until it reached 280 °C and was kept at this temperature for 10 minutes. The injection volume was one microliter each time [22]. It should be noted that the water-oil phase ratio is10-90% and the agitation speed is 800-1800 rpm.

3.Results and discussions

3.1. Analysis of the Size of Iron Oxide Nanoparticles

According to the method presented in the experimental section, nanoparticles were synthesized with a molar ratio of 1 to 2 of ferric and ferrous ions. According to the results, the highest mass of nanoparticles was synthesized with this molar ratio. The optimum temperature was 30 °C, and a suitable alkaline pH of about 10 was selected. To ensure the size of the particle size and morphological study of the synthesized nanoparticles, FE-SEM and TEM devices of Tehran University of Technology were used with the specifications provided in the experimental section, and the following results were obtained (Figure 3 and Figure 4).

3.1.1. Image of Scanning Electron Microscope

To examine the nanoparticles synthesized by the FE-SEM device, after placing the sample in the chamber, the atmosphere inside the microscope column is vacuumed using existing pumps. When the required vacuum is obtained, an electron beam is generated and narrowed by electromagnetic lenses and focused on the sample. In fact, an electron beam is placed on the sample to obtain information from different parts of it. As a result of the collision of the electron beam with the sample, the appropriate signals are generated, which are received by the detectors and eventually converted into the desired image or other information. In conventional electron microscopy, the tungsten filament is used as the source of the electron beam, which has little power to focus the electron beam on the target, which reduces the resolution of the image. The FE-SEM electron microscope uses a single crystal to produce the electron beam, which has a very high focusing power over the target area and still maintains image quality up to very large magnitudes of about 750,000 times. Another advantage of this device is the ability to check the level of magnetic materials, which is not possible in ordinary electron microscopes due to the deflection of the electron beam. Due to the advantages mentioned in this study, the FE-SEM device was used, and as shown in Figure 3, the synthesized nanoparticles have a suitable size in the range of 20 to 50 nm and are spherical in shape.

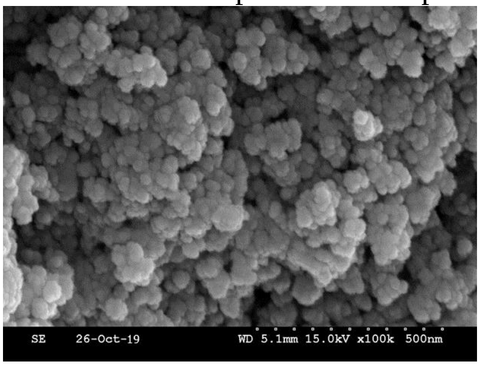

Figure 3. SEM image of nanoparticles of magnetite iron oxide

3.1.2. Image of a transmission electron microscope

Transmission electron microscopy (TEM) is a special tool for determining the structure and morphology of materials that make microstructural studies of materials with high resolution and very large magnification possible. Crystal orientation and defects can be used. The synthesized nanoparticles are not dense and intertwined according to Figure 4, which is related to the transmitting electron microscope, and are uniform and have a suitable crystal structure.

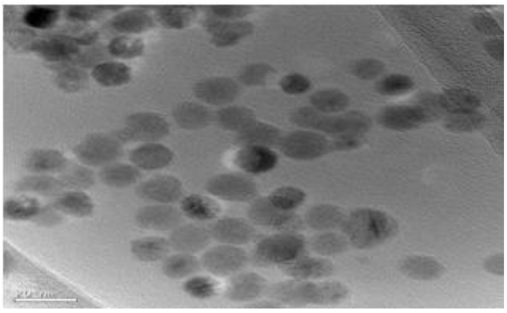

Figure 4. TEM image of nanoparticles of magnetite iron oxide

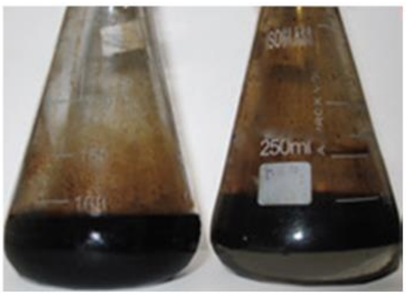

Figure 5. Erlenmeyer, on the right side, is the example of this study, showing the separation of oil from water by magnetite nanoparticles. The control Erlenmeyer on the left has no proper separation.

3.2. The Effect of Iron Oxide Nanoparticles on Oil Separation from Water

Investigating the use of nanoparticles in the separation of oil from water as one of the important environmental problems is very important. The study sample and the control sample showed significant results after one day and one night, as can be seen in Figure 5. The study sample shows good separation of oil from water. Nanoparticles have been very successful in separating water from oil, due to the presence of magnetite nanoparticles. The reason for this is the increase in hydrophobic forces in the oil structure, which causes the oil molecules to be more attracted to each other. Due to the biological compatibility of magnetite iron oxide nanoparticles and having a magnetic property, this property can be used to separate oil pollution from the price and even guide the oil stain by its magnetic property [23].

Figure 6 indicates the Fourier transforms infrared spectroscopy (FTIR) spectra of nanoparticles had the absorption bands similar to the absorption bands of magnetite nanoparticles which produced by the normal method.

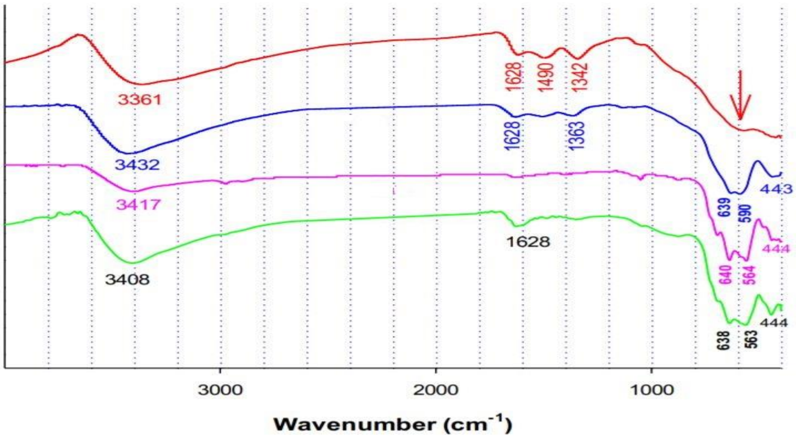

Figure 6. FTIR spectra of the nanoparticles

3.3. Investigation of Oil Separation from Water Using the Chromatographic Gas Device

According to the explanations provided in the experimental section, first in two Erlenmeyer flasks with a volume of 250 mL, 100 mL of oil was mixed with 100 mL of water and stirred. Then, 1 mg of nanoparticles was suspended in the water by an ultrasonic device and added to the oil and water mixture in one of the samples. The samples were placed on a shaker for 24 hours. The oil and water samples were then separated in both Erlenmeyer-containing nanoparticles and Erlenmeyer flask using a separating funnel. Hegzan normal solvent was used to identify petroleum compounds in oil and water samples. To each of the Erlenmeyer flasks, 30 mL of normal hexane was added, and after mixing, a two-phase separator funnel was formed, and the hexane phase was separated. To measure the available compounds using a chromatographic gas device, the temperature of the injection site and the detector were set at 300 °C. The oven temperature started at 80 °C and increased at a rate of 10 °C/min until it reached a temperature of 280 °C and was kept at this temperature for 10 minutes. The injection volume was one microliter each time. Chromatograms of the oil samples and remaining oil in separated water were then taken [24].

3.3.1. Investigation of the Effect of Magnetite Nanoparticles on Oil Samples by Gas Chromatography Method

With the help of normal hexane, samples were prepared for chromatographic gas analysis. Gas chromatography is one of the methods of chromatography that is used to study and separate organic materials without decomposing them. In gas chromatography, the gas phase is an inert phase (for example, helium, nitrogen, argon, and carbon dioxide). The stationary phase is a solid or thin layer of a non-volatile liquid that is deposited on the inner wall of the column or as a coating on the surface of glass or metal pellets. In gas chromatography, the separation of components of a mixture takes place in proportion to the amount of distribution of the components of the mixture between the mobile gas phase and the solid or liquid stationary phase. In this method, the gas carrying the mixture is moved inside the column, and gas-liquid (components of the mixture) is distributed between the two phases in equilibrium. Therefore, the moving phase moves the components of the sample to the outside of the column, and any molecule that has been absorbed by the tether sauce is removed sooner, and the part that has the ability to absorb more with the column is removed later. Therefore, the components of the mixture are separated from each other. Gas chromatography is used to separate and identify components and quantify their decomposition. The control oil sample and the studied oil sample were analyzed after separation by normal hexane with the chromatographic gas device with the Rtx-5 MS column, which is suitable for the separation of organic oil compounds. The addition of magnetite iron oxide nanoparticles did not make a significant difference in the resulting chromatogram. According to Figure 6, the samples tested and the control sample showed similar chromatograms, which can be concluded that the nanoparticles did not change the structure of the oil [25,26].

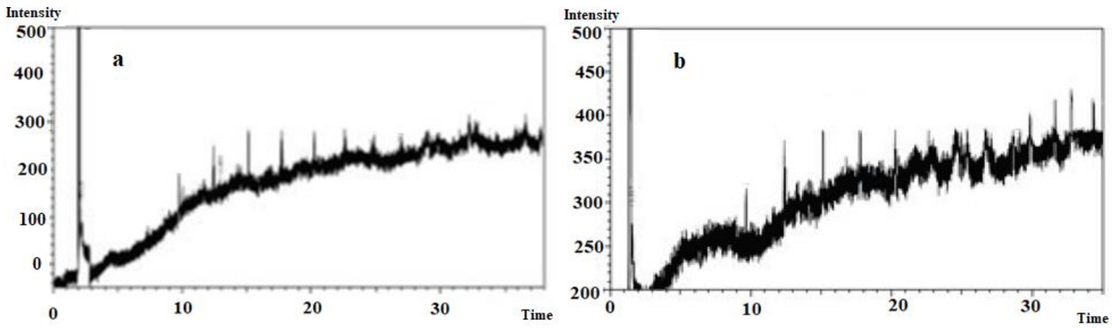

Figure 7. a) Chromatogram of oil in the control sample. b) Chromatogram of oil in the test sample

3.3.2. Examine the Chromatograms of the Remaining Oil Samples in the Separated Water

After studying the separated oil, the aqueous sample was thoroughly mixed with normal hexane, and, with the help of a separator funnel, the hexane phase was separated for chromatographic gas analysis. Due to the fact that the residual oil in the aqueous phase in the control sample and the studied sample tends to be more soluble in hexane compared to water, due to its hydrophobic properties and a greater tendency to the organic environment, it enters the normal hexane solvent. By measuring the amount of residual oil with a chromatographic gas device, we can compare the amount of residual oil in both samples. Figure 8 shows the chromatogram of the oil remaining in the water in the two control samples and the test sample [27, 28].

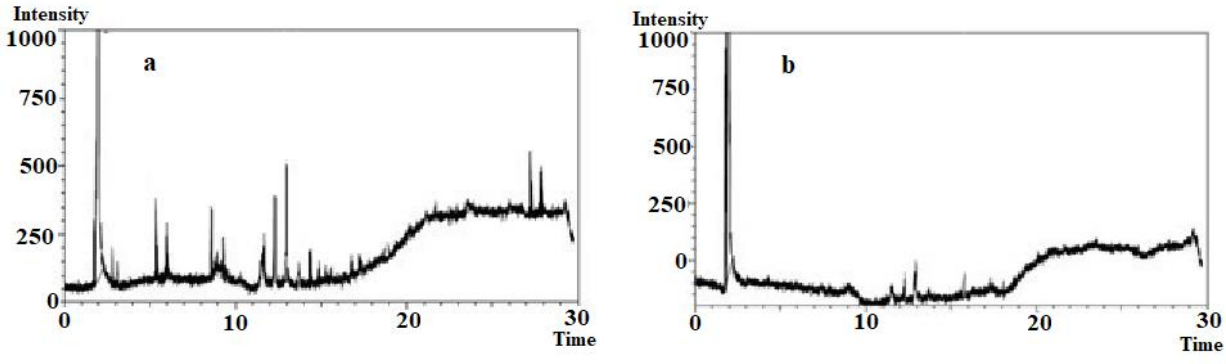

Figure 8. The chromatogram obtained from the remaining oil in the separated water. a) Chromatogram of the control sample, b) Chromatogram of the test sample

As can be seen in Figure 8, the oil compounds identified in the test water sample are very small compared to the control water sample. Results show that more than 90% of petroleum compounds are separated by magnetite nanoparticles. Magnetite nanoparticles have been quite successful in analyzing chromatographic gas and have been able to significantly separate petroleum compounds from the aquatic environment [29-40].

4.Conclusions

In this study, a new method for separating oil from aquatic environments has been considered as an important and practical need. Magnetite iron oxide nanoparticles used in this study caused oil to separate from water. In this project, iron oxide nanoparticles have been synthesized in order to be used in separating petroleum from water. These nanoparticles are biocompatible and could be directed and recovered by magnetism. Based on our experiments, nanoparticles were successful in separating petroleum from water. Furthermore, the recovered petroleum was analyzed by Gas Chromatography and showed no significant change. According to the results of the current investigation, the separated petroleum could be recovered and returned to the economic cycle of the country and inhibit the loss of national wealth.

According to the results of chromatographic gas analysis, two achievements are understandable. Due to the slight changes in the organic compounds identified in the chromatogram obtained from the control sample and the sample under study, the separated oil can be recovered and can be returned to the country's economic cycle as the national capital. On the other hand, by examining the chromatogram obtained from the water separated in the control sample and the studied sample, the separation of petroleum compounds in the presence of nanoparticles has been done up to more than 90% and promises the possibility of using magnetite nanoparticles in aqueous contaminants. It is hoped that the results of this study in the use of magnetite nanoparticles with biological safety will be used as a suitable method for oil recovery in this type of pollution.

Acknowledgement: The authors acknowledge the Foundation of the training plan of young innovative talents in Colleges and Universities," Study on the numerical simulation method of considering the internal fluidity of fault" (Grant: UNPYSCT-2018037/15011011801).

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Manuscript received: 16.04.2020