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Application of magnetic separation for oil spill remediation and recovery in Kuwait sea water

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ABSTRACT

Oil spill is one of the major environmental problems that cause serious damage for both environment and economy. The conventional clean-up methods were found not effective in terms of time, money, and efforts. The oil spreads rapidly in water and this spreading must be controlled quickly before affecting the marine environment. Magnetite nanoparticles have attracted great attention in environmental and in oil spills remediation. In the present study, application of the recent magnetic separation process for remediation and recovery of oil using magnetite (Fe_3O_4). The magnetite particles were mixed rapidly with the oil. Then a permanent magnet was applied to cleanly collect the oil from seawater. The efficiency of oil removal was tested for various concentration, magnetite particles size (35, 5 μm and 15 nm), and oil types. Superior results showed upon increasing the magnetite concentration and reducing the particle size to nano size. The results showed a lower efficiency as the oil get lighter. In this regard, it can be concluded that the magnetic separation for oil spill remediation and removal is a great application to be utilized without causing any kind of pollution rather than the conventional method.

Keywords: Magnetite; Particle size; Spill; Magnetic field; Remediation; Recovery

1. Introduction

Oil spills are accidental discharges of liquid petroleum products into surrounding water bodies, mostly seas. The Kuwait oil fire in 1991 was one of the biggest oil spills in human history, the volume spilled hovered around 11,000,000 US barrels (1,300,000 m^3) [1,2].

Oil production from its production sources to the desire location, has a lot of risks due to its accidental spill [3]. One of the main causes of oil spill is during transportation. Crude oil and refined fuel spills from tanker ship accidents have damaged vulnerable ecosystems in Alaska, the Gulf of Mexico, the Galapagos Islands, France, the Sundarbans, Ogoniland, and many other places [4]. The quantity of oil spilled during accidents has ranged from a few hundred tons to several hundred thousand tons (e.g., deepwater horizon oil spill, Atlantic empress, Amoco Cadiz). Smaller spills have already proven to have a great impact on ecosystems, such as the Exxon Valdez oil spill because of the remoteness of the site or the difficulty of an emergency environmental response [5]. Oil transportation will continue to increase worldwide as people needs it. Many communities are at risk of oil spill devastation [3].

Marine oil spill cleanup and control is the most debatable issue, due to the difficulty of clean up. Thus, it becomes

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increasingly important to employ various cleanup methods for tacking the menace they could pose to the marine ecosystem [6]. Oil spilled conventional techniques used for clean-up were biodegradation, mechanical method, chemical dispersants, and *in-situ* burning [7]. Biodegradation leads to by-products that has proven the risk posed to aquatic organisms [8]. Mechanical devices, such as skimmers and booms, can separate the oil slick from water. The efficiency of this technique is affected by lot of factors such as the technical features of skimmers and booms, the weather conditions, and oil properties [9]. Toxicity of chemical dispersants used for the cleanup and containment of crude oil became a major concern after the 2010 [10]. Petroleum toxicity of an oil slick raises with chemical dispersion [11]. Biodegradation leads to by-products that has proven the risk posed to aquatic organisms [8]. *In situ*-burning of the oil may produce large amounts of black smoke that can affect humans, wildlife, and the environment [12,13]. Removal of spilled oil on the coast is mostly done manually, which requires the involvement of a large number of labours, residual oil remnants still contain various toxic chemicals. Therefore, the health of the clean-up workers is of major concern [14]. Oil spill cleaning is considered a very expensive processes in the disaster management. Cleaning oil spill in the USA estimated cost is around 16 per gallon [15]. The disadvantages of conventional techniques for oil spill are secondary pollution, high cost, low efficiency, long process time for clean-up [9].

The development of possibilities for oil cleanup from water bodies has been a research focus on diverse areas of knowledge. Recently, new development technique was investigated for oil spill treatment using nano-based systems [16]. Nanotechnology has been a distinguished area of research due to the unique characteristics of developed materials and the promising results for high capacity of removal of contaminants from water [17]. Much attention has been paid recently to the high oil-absorption materials which have hydrophobic and oleophilic properties as oil absorbents [18]. The use of iron oxide magnetic nanoparticles as nano adsorbents has led way to a new class of magnetic separation strategies for water treatment [19–21]. Magnetic iron oxide nanoparticles material showed an efficient, reliable, inexpensive, and environmentally friendly technique in hydrocarbons recovery from water [9,22]. Magnetic nanoparticles of iron oxide have a strong ability of magnetism, has high surface area to volume ratio, and high adsorption [13,22]. When iron oxide exposed to magnetic field, the particles combined together, makes it easy, and fast for oil separation from aqueous solution [23,24]. Iron nanomaterials (e.g., magnetite) have found wide applications in environmental remediation, and in medical applications [25,26]. Magnetic separation treatment can minimize the impact of oil spills on marine ecosystem, marine life, plants, and animals live in that area, in comparison with other conventional methods that take longer time in containing and collecting the oil.

In this study, we focus on highlighting the application of the most recent development technique for remediation and separation of oil using magnetic iron oxide (Fe_3O_4) from seawater. The study will include the effect of different concentration, particle size (35, 5 µm, and 15 nm) and oil types on oil spill removal using a fast and environmentally friendly technique through experimental investigation. Percentage reduction efficiency was calculated for all experimental tests.

2. Experimental section

2.1. Materials

Magnetite *A*: iron oxide (Fe_3O_4) with high purity, 99%, 35 µm; magnetite *B*: iron oxide (Fe_3O_4) with high purity, 99%, 5 µm); and magnetite *C*: iron oxide (Fe_3O_4) nanoparticles (high purity, +99.5%, 15 nm). All magnetite's used were powder based. Magnetite's were purchased from US research nanomaterials inv. Methylene chloride and sodium sulfate were purchased from Aldrich Chemical Co. LLC, (Millipore Sigma, 6000 N Teutonia Ave, Milwaukee, WI, 53209-3645 United States). Pulling force rare earth magnet (super strong, Chemical Co., 750 LBS) Neodymium fishing magnets with countersunk hole diameter 3.54 inch was purchased from HGMAG, N52. Neodymium cylinder magnetic rod (10 mm × 60 mm) was purchased from DIYMAG. Crude and diesel oil samples were taken from Kuwait oil field. Seawater was taken from Kuwait Gulf Sea.

2.2. Test on crude and diesel oil using different magnetite particle size (A: 35 µm, B: 5 µm, and C: 15 nm)

Fifty milliliters of sea water was placed in 100 mL beaker, 2 g of crude, or diesel oil was added at the top of sea water. 0.333, 0.4, 0.5, 0.8, and 1.0 g of magnetite (*A*, *B*, and *C*) was sprayed at top of the oil (1:6, 1:5, 1:4, 1:2.5, and 1:2, magnetite to oil ratio). The preparation of the feed suspension of oil and particles involves weighing of particles with a precision balance. Particles were applied to rapid mix for 5 min with the oil. Magnetic rod was then inserted into the beaker, the time taking for a magnet to attract the magnetite, and the oil drops was 2 min. Then, the magnetic rod was place in a 25 mL glass tube containing 10 mL methylene chloride. Methylene chloride was used due to it high evaporation rate (27.5). In literature *n*-heptane was used for similar process [27]. Other method dissolve magnetite particles in ethanol by ultrasonic washing, then regenerate it through filtering, washing and drying at 40°C for 6 h [19]. In this study, the experiment was carried out under fume hood to avoid inhalation of methylene chloride. Methylene chloride dissolve all the oil that is attracted to the magnet. The magnetite was then recovered from the magnetic rod (regenerated particles to absorb oils for many times) and place back into the same beaker to attract the remaining oil and magnetite. Then, placed again into the glass test tube containing methylene chloride. To ensure accuracy, three trials were repeated, till most of oil removed. Sodium sulfate was added to the test tube to remove any water taken during the removal. A beaker weigh was taken using analytical balance. The oil recovered was then filtered to remove any sodium sulfate to the beaker. The tube was rinsed three times with methylene chloride to ensure all oil was collected. The filtrate was then left for the 24 h for the evaporation of methylene chloride. The beaker was measured after the evaporation of methylene chloride and the difference was then calculated. The experiments were conducted at room temperature of approximately 22°C and were repeated for three times. Schematic diagram of the recycle route of oil and magnetite experiment is shown in Fig. 1.

2.3. Test on vegetable using magnetite different particle size (A: 35 μm, B: 5 μm, and C: 15 nm)

The same experiment was repeated by using 0.333 and 0.5 g of all magnetite's (*A*: 35 μm, *B*: 5 μm, and *C*: 15 nm) with 2 g of vegetable oil.

2.4. Oil removal process

Fig. 2 illustrates the oil removal process using magnetite for larger scale. Twenty-five grams of magnetite (5 μm) was sprayed at the top of the oil (50 g) and mixed rapidly in 500 mL seawater for 5 min. The magnet was applied outside the beaker. Time measured for oil and magnetite attraction via magnetic field was 2 min. The magnet attracted all oil and the magnetite. The oil and the magnetite were then recovered and separated.

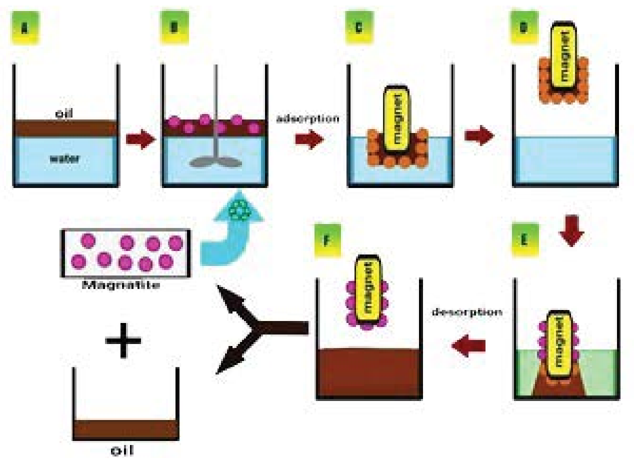

Fig. 1. Illustration of recycle route of oil and magnetite. (A) Oil in seawater, (B) stirring the magnetite with oil in seawater, (C) magnetic attraction of oil + magnetite, (D) removal of the magnet containing oil and magnetite, (E) immersing the magnet containing oil and magnetite in methylene chloride, and (F) removal of magnet containing magnetite from methylene chloride.

2.5. Evaluation of the magnetite particles as oil collector

The quality of the filtration experiments is specified by the removal efficiency. The removal efficiency was calculated for crude, diesel, and vegetable oil. The calculation of the total separation efficiency is done by using Eq. (1). Removal efficiency was performed by subtracting the mass of the oil removed from the original oil added [28]. Then dividing by mass of the original oil and multiplied by 100 (Eq. (1)).

$$\text{Removal efficiency percentage} = \frac{\text{mass of the original oil}(\text{g}) - \text{mass of the oil recovered}(\text{g})}{\text{mass of the original oil}(\text{g})} \times 100 \quad (1)$$

2.6. Transmission electron microscopy

Typical transmission electron microscopy (TEM) micrographs of the magnetic 5 μm and 15 nm iron oxide (Fe_3O_4) particles are shown in Fig. 3.

3. Results and discussion

Magnetite is main iron ore; and is one of the naturally occurring iron oxides. The chemical formula of iron oxide (magnetite) is Fe_3O_4. It can be attracted by a magnet and become a permanent magnet. The present work for remediation and collecting the oil from seawater was performed with the magnetite by different concentration (ratio of magnetite to oil 1:6, 1:5, 1:4, 1:2.5, and 1:2), particles size (35, 5 μm, and 15 nm), and oil types (crude, cooking, and diesel oil). All the tests were performed in 2 g of the oil. Rapid mix of oil and magnetite was kept for 5 min. Time measured for oil and magnetite attraction of via magnetic field was 2 min. Oil and magnetite were then collected and recovered.

3.1. Physical properties of crude, diesel, and vegetable oil

Table 1 illustrates the physical properties of crude, diesel, and vegetable oil used in the study.

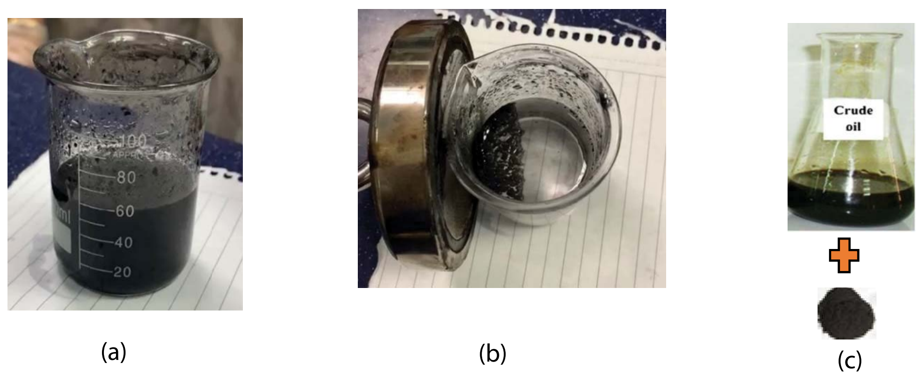

Fig. 2. Process of crude oil removal from sea water using magnetite (5 μm) by magnetic attraction; (a) mixture of crude oil + magnetite in seawater and (b) attraction of crude oil + magnetite with magnet; (c) recovery of crude oil and magnetite.

3.2. Effect of varying the magnetite concentration on oil removal

Fig. 4 shows the crude oil reduction percentage by varying the magnetite concentration for each magnetite *A* (35 µm), *B* (5 µm), and *C* (15 nm). In this step 0.333, 0.4, 0.5, 0.8, and 1.0 g for each of 35 µm magnetite (*A*1–*A*5), 5 µm magnetite (*B*1–*B*5), and (*C*1–*C*5) to 2 g crude oil.

The reduction percentage of crude oil recovered upon increasing the magnetite concentration was 62.87%, 63.19%, 65.6%, 66.38%, and 67.62%, respectively, upon using 35 µm magnetite (*A*1–*A*5). Whereas, the oil reduction percentage for 5 µm magnetite (*B*1–*B*5) was 83.95%, 85.58%, 86.97%, 87.05%, and 88.29%. However, the reduction percentage for 15 nm magnetite (*C*1–*C*5) was 93.62%, 95.87%, 96.31%, 97.08%, and 98.1%, respectively. The results indicated the higher reduction efficiency of crude upon increasing the concentration for all the magnetite's (*A*, *B*, and *C*). The results show promising data for the separation of the petroleum crude oil from aqueous solution in environmental pollution cleanup.

Similar trends were shown for diesel oil (Fig. 5). Percentage reduction efficiency using 35 µm magnetite (*A*1–*A*5) to diesel oil was 18.5%, 20.3%, 25.29%, 26.34%, and 26.59%, respectively. Whereas, the reduction percentage using 5 µm magnetite (*B*1–*B*5) was 33.79%, 37.05%, 40.96%, 41.96%, 41.96%, and 45.74%, respectively. However, the reduction percentage using 15 nm magnetite (*C*1–*C*5) was 37.83%, 46.3%, 47.59%, 48.03%, and 51.35%, respectively.

Fig. 6 shows that the percentage reduction efficiency for vegetable were 56.65% and 58.77% using 35 µm magnetite (*A*1, *A*3), 63.98%, 64.92% for 5 µm (*B*1, *B*3) and 72.64%, 79.07%, respectively using 5 nm magnetite (*C*1, *C*3).

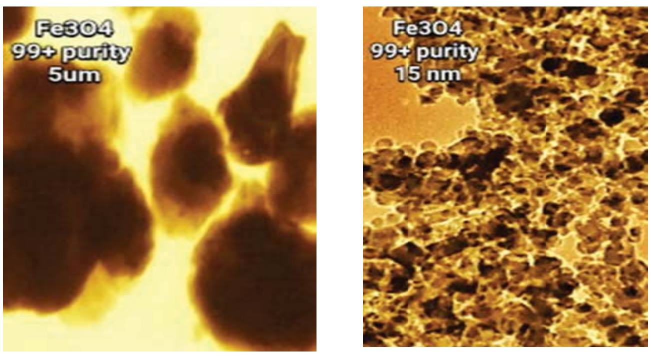

Fig. 3. Transmission electron microscopy (TEM) images for 5 µm and 15 nm Fe_3O_4.

The results for all oil types (crude, diesel, and vegetable oil) indicated that the percentage reduction efficiency increased upon increasing the magnetite concentration. The reason behind that as we are increasing the concentration of magnetite, the oil droplets will face more of the magnetite particles and become more magnetized. Hence, will be more attracted by the magnet and so increases the adsorption efficiency. In addition, to the hydrophobic nature of Fe_3O_4 that's makes it more combined to the oil particles.

3.3. Effect of varying magnetite particle size on crude, diesel, and vegetable oil removal

Fig. 4 indicates that the percentage reduction efficiency of crude oil from seawater increases as we reduce the particle size of the magnetite. The results show that nanoparticle magnetite *C* (15 nm) has a superior oil removal efficiency with percentage of 93.62%–98.1% compared to magnetite *B* (5 µm) with 83.95%–88.29% and to magnetite *A* (35 µm) with 62.87%–67.62%. The superior adsorption capacity of the nanoparticle magnetite *C* (15 nm) is due to the large exposed surface to volume ratio that makes the oil spread more on the surface than magnetite *A* and *B* with smaller particle size (smaller surface area). Hence, larger adsorption capacity, higher oil collecting and higher removal efficiency. Similar trends were shown in for vegetable and diesel oil (Figs. 5 and 6). The results also evaluated that by using a 0.333 g of magnetite C the oil recover by 93.62% compared to 62.87% for magnetite *A* and to 83.95% of magnetite *B* under the same condition.

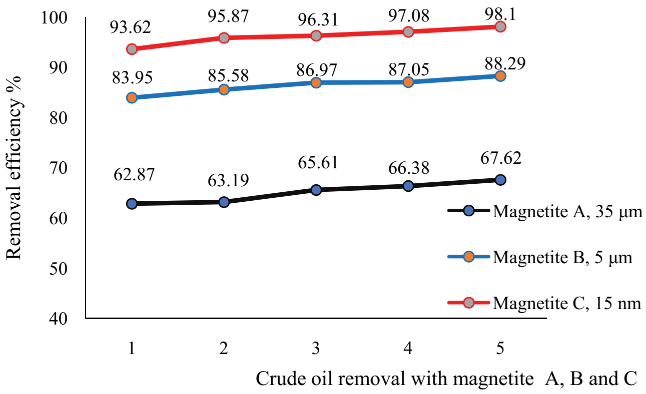

Fig. 4. Removal efficiency percentage of crude oil with different magnetite particle size (*A*1–*A*5, 35 µm; *B*1–*B*5, 5 µm; *C*1–*C*5, 15 nm).

Table 1
Physical properties for crude, diesel, and vegetable oil

Test	Crude oil	Vegetable oil	Diesel oil
Density at 15°C, g/mL	0.90–0.92	0.87–0.89	0.84–0.86
Density at 65°C, g/mL	0.87–0.89	–	–
API gravity at 15°C	22–25	28–30	33–37
Total sulfur, wt.%	2.5–2.9	–	1.2–1.5
Kinematic viscosity at 50°C, cSt	11.0–13.0	8–9	3–5

All results indicate that the oil percentage reduction efficiency increases as the particle size decreases. The effect of magnetization is stronger with smaller magnetite particle size. Nanoscale, superparamagnetic Fe_3O_4 nanoparticles exhibit high magnetic susceptibility, which provides a stronger and faster magnetic response. Their superparamagnetic properties together with other intrinsic properties such as low toxicity, high surface area-to-volume ratio, superhydrophobic, and simple separation methodology, making them ideal to remove oil from water surface. In addition, for environmental remediation, biomedical, and agricultural applications [13].

3.4. Effect of different oil API (crude, diesel, and vegetable oil) on oil removal efficiency

Fig. 7 shows the removal efficiency using 15 nm magnetite. The results indicate that the magnetite nanoparticle *C*1–*C*5 with 15 nm has a superior removal efficiency for collecting heavier crude oil with a removal percentage of 93.62%–98.1% than the lighter diesel oil of 37.83% to 51.35%. The results reveal that magnetite with nano-particle size *C*1–*C*5 has a quite good efficiency in collecting diesel oil up to 51.35% although in the conventional method it's very difficult to be remove due to its low density that will make it disperse. Magnetite nanoparticle (15 nm) exhibit selective absorption to crude oil than diesel oil.

Fig. 8 shows similar trends with magnetite *A* and *B* for heavier crude oil and lighter diesel oil. The reduction percentage were 62.87% to 67.62% for magnetite *A* by increasing the magnetite to oil ratio. However, for lighter diesel oil were 18.5%–26.59%. Similarly, for magnetite *B*, the reduction percentage for heavier crude oil were 83.95%–88.29%. Whereas, for the lighter diesel oil were 33.79%–45.74%. The results reveal that magnetite was able to remove even the light oil by magnetic separation fast technique before it gets dispersed. However, traditional methods take longer time for collecting the oil. This will allow the oil to disperse and remain in water until the procedure is accomplished that will affect the marine environment. The results also indicated that the sorption capacity of magnetite depends on the density and viscosity of the oil and this is seen by the highest adsorption of the crude oil more than the lightest cooking and diesel oil.

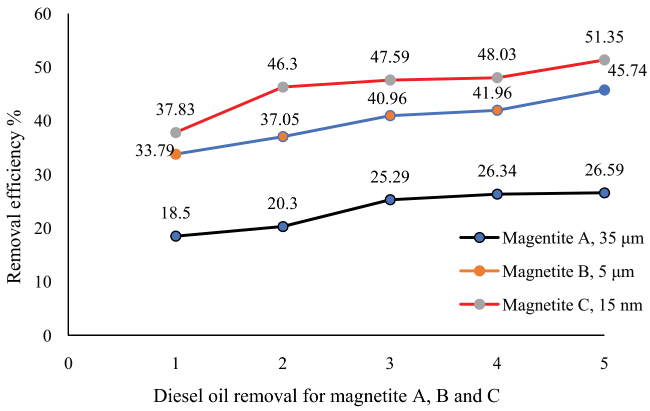

Fig. 5. Removal efficiency percentage of diesel with different magnetite particle size (*A*1–*A*5, 35 µm; *B*1–*B*5, 5 µm; *C*1–*C*5, 15 nm).

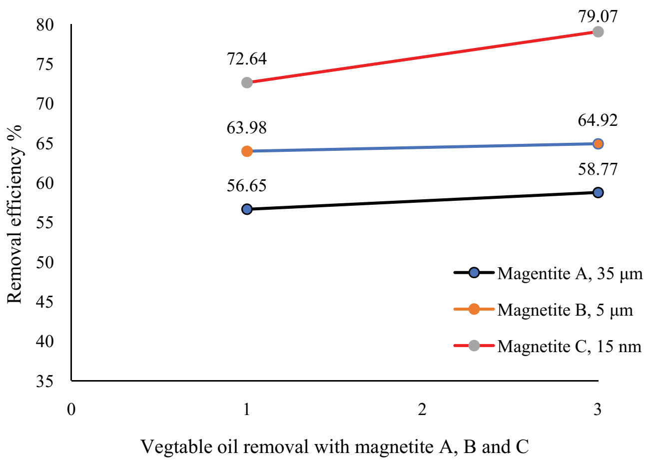

Fig. 6. Removal efficiency percentage of vegetable oil with different magnetite particle size (*A*1–*A*5, 35 µm; *B*1–*B*5, 5 µm; *C*1–*C*5, 15 nm).

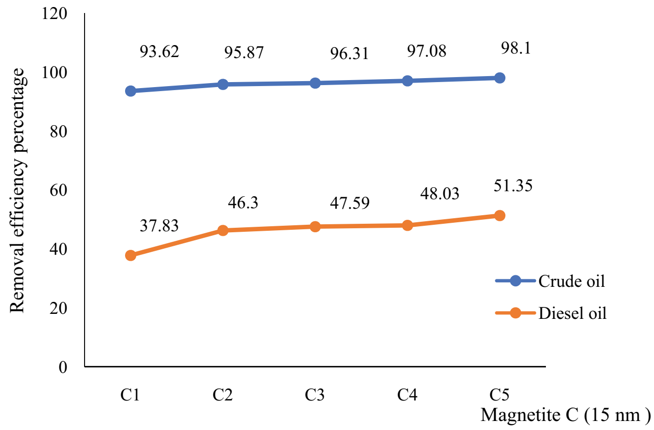

Fig. 7. Removal efficiency percentage of crude and diesel oil with 15 nm magnetite (*C*).

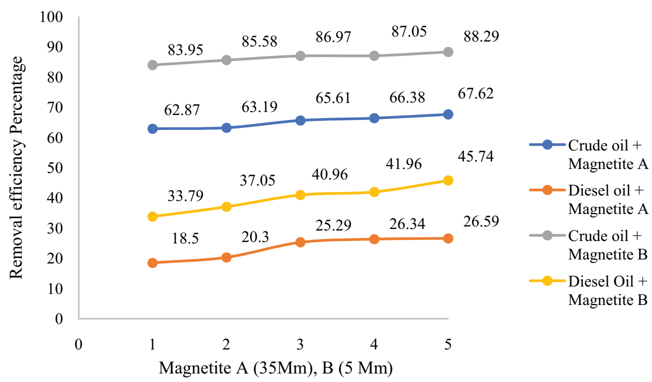

Fig. 8. Removal efficiency percentage of crude and diesel oil with magnetite *A* (35 µm) and magnetite *B* (5 µm).

4. Conclusion

This paper covered the application of a recent development technique for oil remediation and recovery from Kuwait seawater using external magnetic field, with a special emphasis on different magnetite particle size, different concentration and different oil types. In recent years, there are many research articles have been published in this field and significant development has been achieved. The work on oil remediation and recovery is important because oil spills affect marine life, human health, and the economy of the involved countries. In addition to that, in Kuwait over three billion people depend on marine and coastal biodiversity for their livelihoods. Due to that, the authors think that oil recovery is a very important topic to work with using alternative technique to overcome the disadvantages from the conventional method. The experimental results indicate the following:

The oils were selectively absorbed by magnetite particles, and the oil-absorbed nanoparticles was recovered under external magnetic field.

- All magnetite's used in this work were capable of collecting crude, diesel, and vegetable oil from water surface due to its hydrophobicity effect.
- The results demonstrated that the removal efficiency enhances with increasing the concentration of the magnetite.
- Fe_3O_4 nanoparticles *C* (15 nm) exhibited the best removal efficiency for oil–water separation than the magnetite *A* (35 μm) and *B* (5 μm). This reflects that as the magnetization increases with nanoparticle, the oil recovery % increases.
- The results indicated that magnetite particles are more effective in removing heaver crude oil than the lighter vegetable and diesel oil.
- The fast recovery of the oil and the reusability of the magnetite materials increase the environmental friendliness and could decrease the cost of the treatment method.
- Oil companies could use this innovative technique to help clean up oil spills in marine and to purify water that contains oil.
- The application of oil magnetic separation technique by magnetite is highly promising, simple, rapid, safe, and efficient. However, conventional treatment methods are slower, low efficiency, high cost, hazard to marine, and coastal ecosystems and require long process time to clean up.
- Magnetic particles could be easily separated and recycled from different oil solution by magnetic field because of their strong magnetic property, indicating that this material could be widely used in the removal of different oil leakage accidents.
- Further improvement of removal efficiency percentage could be made through functionalization of magnetite which can leads to increase the hydrophobicity of magnetite and enhance the oil adsorption from water or by the utilization of smaller particle magnetite size.

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