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Article

The development of FeO(OH) nanoparticles to extract synthetic motor oil from freshwater

Esther Zhang[1], Terese Grateful[1]
[1] Council Rock High School North, Newtown, Pennsylvania

SUMMARY

Hydrocarbons, toxic to human and ecosystem health, are a leading contributor to the widespread pollution of freshwater systems. Lower volumes of hydrocarbon pollutants are often neglected by large-scale clean up techniques. To help address this problem, we designed modified iron(III) oxide-hydroxide (FeO(OH)) nanoparticles to extract synthetic motor oil, a model hydrocarbon pollutant, from freshwater. We hypothesized that there would be a positive, linear relationship between the mass of FeO(OH) nanoparticles dispersed and the mass of motor oil extracted. First, we developed the FeO(OH) nanoparticles through the thermal decomposition of FeO(OH) using oleic acid and eicosane, long hydrocarbon modifications that attach to the surface of the FeO(OH) nanoparticles. These modifications allow for the hydrophobic interactions that provide the primary functionality of the FeO(OH) nanoparticles: attracting and extracting the motor oil. We then dispersed increasing amounts of these FeO(OH) nanoparticles into a polluted sample with a low oil-to-water ratio, and later extracted the superparamagnetic FeO(OH) nanoparticles along with oil using a neodymium magnet. We found a significant positive, linear relationship between the FeO(OH) nanoparticle mass used and the motor oil mass extracted with a ratio of 3.57 g of oil removed per gram of nanoparticles. The water mass extracted remained consistently low with water making up, on average, only 3.2% of the total mass of water and oil extracted by the FeO(OH) nanoparticles. These findings provide a favorable solution to combat widespread hydrocarbon residue persistently polluting global water sources.

INTRODUCTION

Hydrocarbons are a leading contributor to water pollution, which limits the availability of freshwater. Water pollution is a major public health issue worldwide, affecting over one billion individuals each year (1). Hydrocarbon pollutants are insoluble in water, persistent, and highly toxic, even at low concentrations (2). Usual hydrocarbon removal procedures, such as skimming, are designed to clean up larger spills under ideal surface conditions (3). Consequently, these methods lack precision in avoiding water or marine life removal, and falter in efficacy when winds and waves naturally disrupt the water surface, fragmenting the oil into smaller dispersions throughout the water (3). Hydrocarbon pollution, particularly oil spills, occurs more frequently in freshwater and tends to be more destructive to environmental and human health as freshwater is used for drinking water as well as shelter and food for various organisms (4). The repercussions of an oil spill in standing water are also more severe than flowing water as pollutants simply gather rather than wash down with the current (4). Therefore, a demand for an effective and thorough method for the extraction of hydrocarbon pollutants in freshwater persists.

Nanotechnology is the branch of science and engineering devoted to studies of matter under 100 nanometers in dimension (5). As a material approaches the nanoscale, the surface area to volume ratio becomes drastically large compared to bulk material, which enables nanoparticles to possess emergent properties that the bulk material does not possess since surface atoms now dictate the physical and chemical properties of the material (5). In particular, nanoparticles of iron(III) oxide-hydroxide (FeO(OH)) possess superparamagnetic properties, meaning they should only aggregate when an external magnetic field is applied (6). These superparamagnetic properties facilitate unconstrained dispersal and a more effective collective extraction, as FeO(OH) nanoparticles cluster for removal rather than needing to be magnetized individually (6).

The main feature of FeO(OH) nanoparticles, aside from their inherent magnetic properties, is their capacity for modifications or coatings that provide the primary functionality of the particle. Specifically, FeO(OH) nanoparticles can be synthesized and modified to be hydrophobic through methodology derived from the thermal decomposition of FeO(OH) along with the hydrocarbons oleic acid and eicosane (7). The heating process of thermal decomposition is then followed by repeated precipitation and dispersion with the solvents acetone and hexane to procure the actual FeO(OH) nanoparticles (7). The hydrocarbon modifications on the nanoparticle, combined with the inherent magnetic qualities of FeO(OH), facilitate the extraction of the hydrocarbon pollutant through hydrophobic interactions (8).

These hydrophobic interactions occur because hydrocarbons are hydrophobic substances composed of long, nonpolar chains of carbon and hydrogen (9). The longer the chain, the more intermolecular forces involved and the stronger the interactions. The nonpolar covalent bond between carbon and hydrogen means that they have no significant dipoles to interact with water, a polar molecule, resulting in their hydrophobic classification (9). Since water is strongly attracted to itself and other hydrophilic substances through hydrogen bonding, hydrophobic substances in water are collectively excluded and consequently gather together, strengthened by their London dispersion intermolecular

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forces (10). The subsequent "attraction" between the hydrophobic FeO(OH) nanoparticles and the hydrocarbon pollutant persists, even as an attractive magnetic force is applied, since water's attractions or exclusion forces are relatively stronger than other intermolecular forces (10). Essentially, the hydrocarbon modifications made to the FeO(OH) nanoparticles create hydrophobic particles that selectively attract and trap hydrocarbon pollutants.

In existing research, FeO(OH) nanoparticles have been surface-engineered for environmental remediation with surfactants and modifiers like β-cyclodextrin or formed into nanocomposites incorporating absorbent properties through accompanying materials such as chitosan, clay, and activated carbon (6). Although these methods yield high adsorption capacities, they are designed for organic pollutants and have high material costs (6). Therefore, in search of an accessible modification specific to synthetic motor oil pollution, we derived our FeO(OH) nanoparticle synthesis procedure from a pre-existing process that modifies FeO(OH) nanoparticles for magnetic particle imaging (MPI) (7). This procedure involves thermal decomposition, during which hydrocarbons attach to the FeO(OH) nanoparticle surface, followed by the attachment of lipids to form a micelle for the purpose of MPI (7). Our goal was to create a FeO(OH) nanoparticle whose surface was hydrophobic, so we only employed the thermal decomposition process in order to obtain FeO(OH) nanoparticles with the ability to extract synthetic motor oil through hydrophobic interactions.

A study of nanotechnology within environmental remediation has been done before with modifications of alkyl phosphonic acid on a pre-assembled iron oxide nanoparticle core (8). Such prior research investigated the ability of two different surface modifications to extract a range of pollutants, focusing on microplastics and other solid waste, and the results mostly compared extraction efficiency between the different pollutants and surface modifications (8). The study found stronger results when looking at the number of hydrocarbon precipitations, crystals, and spheres removed per gram of nanoparticle compared to the mass of pollutant removed per gram of nanoparticle (8). Their iron oxide nanoparticles were also generally more efficient for solid waste; however, out of the nanoparticles extracting liquid waste, the most effective used hydrophobic interactions (8). In extension, we considered the higher efficacy of modifications based around hydrophobic interactions when synthesizing our FeO(OH) nanoparticles and designed them particularly for synthetic motor oil water waste. We focused our study on the correlative relationship between the mass of FeO(OH) nanoparticles used and the subsequent pollutant mass extracted for a specific hydrocarbon.

We sought to address the demand for a method of extraction for lower levels of aquatic hydrocarbon pollution through the synthesis and modification of FeO(OH) nanoparticles to extract synthetic motor oil from freshwater. We hypothesized that if the mass of FeO(OH) nanoparticles distributed into a polluted sample increased by 1 g, the mass of pollutant extracted would also increase by 1 g in a 1:1 linear relationship; we expected the total surface area of the FeO(OH) nanoparticles to be directly proportional to the pollutant mass extracted. To test the efficacy of our synthesized FeO(OH) nanoparticles, we collected data on the oil mass extracted for increasing masses of FeO(OH) nanoparticles dispersed into an identical polluted sample. We also simultaneously measured the water mass extracted to determine the accuracy of the FeO(OH) nanoparticles in removing only the targeted pollutant. The environmental applications of nanotechnology drawn from our research results may be applied to address other types of pollutants in different mediums as well as hydrophobic aquatic pollutants.

RESULTS

To test the ability of our FeO(OH) nanoparticles to extract synthetic motor oil from a polluted sample where the concentration of pollutant is relatively low, we first synthesized FeO(OH) nanoparticles using thermal decomposition (7). We heated a mixture of FeO(OH), oleic acid, and eicosane that was then precipitated and redispersed with acetone, hexane, and oleic acid through centrifugation to create our superparamagnetic and hydrophobic FeO(OH) nanoparticles. Then we deposited varying amounts (0.03-0.07 g) of the synthesized FeO(OH) nanoparticles into a polluted sample in which the ratio of synthetic motor oil to water was relatively low (10% by volume).

We studied the oil and water yield by measuring extracted amounts directly from the neodymium magnet used to remove the FeO(OH) nanoparticle aggregates. We determined the total mass of the FeO(OH) nanoparticle aggregate after it was removed from the oil/water mixture, and again after the water evaporated from the aggregate over two days. We performed the experiment twice for each of the five masses of FeO(OH) nanoparticles dispersed. To determine the average mass of water removed relative to the total mass extracted, we divided the mass of water extracted by the mass of water and oil removed, and then averaged all 10 trials. We found that water removed during extraction made up, on average, 3.2% of the total mass of oil and water removed by the FeO(OH) nanoparticles **(Figure 1)**.

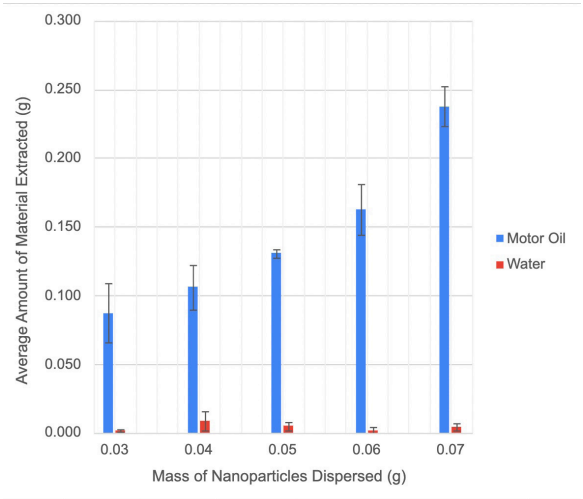

Figure 1: The amount of motor oil and water extracted for each mass of FeO(OH) nanoparticles used. The average amount of motor oil (blue) and water (red) extracted from the same polluted sample (n=2) graphed for each mass of FeO(OH) nanoparticles used. The magnet used to remove the aggregates was weighed immediately after removal and then weighed again after two days, during which the water evaporated. The average water percentage of the total extracted mass was 3.2%. The average of two trials is shown by the colored bar; error bars show range.

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As the mass of FeO(OH) nanoparticles released increased, the mass of motor oil extracted also increased, producing a positive correlation (r=0.92) between the FeO(OH) nanoparticle mass distributed and motor oil mass extracted and a trendline equation of 3.57x - 0.035 (R^2=0.844) **(Figure 2)**. As the mass of FeO(OH) nanoparticles dispersed increased, we observed that the FeO(OH) nanoparticles formed larger aggregates, enabling the aggregates to trap more oil. This resulted in a ratio of 3.57 g of oil removed per gram of nanoparticles, outperforming our previously hypothesized 1:1 relationship. The Pearson correlation coefficient (r=0.92) was statistically significant ($p<0.05$) for the positive correlation between FeO(OH) nanoparticle mass dispersed and oil mass extracted **(Figure 2)**.

DISCUSSION

We tested the ability of our synthesized FeO(OH) nanoparticles to extract synthetic motor oil pollution from freshwater. We hypothesized that a 1 g increase in the mass of FeO(OH) nanoparticles dispersed would correlate with a 1 g increase in the mass of motor oil extracted in a positive, linear relationship. We found that a higher mass of FeO(OH) nanoparticles dispersed resulted in an increase in hydrocarbon pollutant extracted, as shown by the statistically significant, positive correlation between the two variables in experimental extraction measurements. The Pearson correlation coefficient (r=0.92) established statistical significance in the relationship. The observed increase in the size of aggregations during their removal supported this finding and suggests that a larger surface area allows for greater contact with – and therefore removal of – the targeted pollutant. Larger hydrophobic aggregations may have also resulted in increased exclusion forces from water and stronger hydrophobic interactions (11). Additionally, each experimental group removed a higher mass in oil than FeO(OH) nanoparticles dispersed, as shown by the slope value of 3.57 g of oil removed per gram of nanoparticles **(Figure 2)**. This is likely because the superparamagnetic properties of iron oxide resulted in their extraction as a collective group, allowing for hydrocarbons to become trapped between the extracted particles as well as directly attached to the FeO(OH) nanoparticles (11).

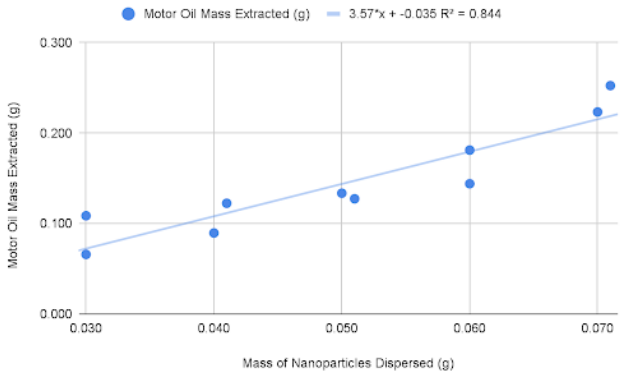

Figure 2: Mass of motor oil extracted for each mass of modified FeO(OH) nanoparticles dispersed. The amount of motor oil removed from the polluted sample by increasing amounts of FeO(OH) nanoparticles with a linear trendline (n=10). Modified FeO(OH) nanoparticles were dispersed into a polluted sample composed of 10% motor oil in tap water, and the mass of the pollutant extracted through the FeO(OH) nanoparticles was directly measured. The trendline equation of 3.57x - 0.035 (R^2=0.844) indicates a relationship between the two variables in a proportion greater than three to one. The Pearson correlation coefficient (r=0.92) was statistically significant ($p<0.05$).

The intention behind the hydrophobic modifications of the FeO(OH) nanoparticles was to target hydrocarbon pollutants and avoid extraneously extracting water. The extraneous extraction of water would not only lower the efficacy of the FeO(OH) nanoparticles in extracting the targeted pollutant but may also lead to the disruption of aquatic environments and marine life. Our experimental results supported the general lack of binding interaction between the particles and water in comparison to the particles and the oil. The small amounts of water that were extracted in each trial were not unexpected; it is logical that minute amounts of water may have been trapped inside or been in contact with the hydrophobic aggregations and were extracted along with the oil and FeO(OH) nanoparticles (12).

The variability between our two trials could have come from several sources, including the FeO(OH) nanoparticle extraction procedure, the sample size, and our data collection methods. The magnetic removal of the FeO(OH) nanoparticles required manual input, which potentially led to inconsistencies throughout the extraction process. This may have resulted in minor amounts of FeO(OH) nanoparticles and their collected pollutant left behind in the sample, but we were unable to eliminate this limitation due the magnet type available for use, and the size constraints of the beaker. Furthermore, the synthesis process yielded only a small mass of FeO(OH) nanoparticles each time, so the FeO(OH) nanoparticle masses and sample sizes used in this experiment were small. Future research may involve larger sample sizes and additional repetitions, which would also reduce the impact of outside variables and would likely produce stronger results. Additionally, when the extracted FeO(OH) nanoparticles, oil, and water aggregates were left out to dry over the span of 48 hours, we did not cover the aggregates to prevent extraneous collection of dust. This may have resulted in a higher overall mass when the aggregates were weighed after the 48 hours, meaning the calculated mass of water extracted was less than the actual amount; however, this error is likely minor considering the negligible weight of dust particles. Additionally, the aggregates were left to dry in the same place for all of the experimental groups, so any resulting error would have likely been constant between groups. Moreover, although we derived the procedure for FeO(OH) nanoparticle synthesis from previously established nanoparticle production methods, we did not have access to the resources necessary to characterize the particles that were produced, so we could not be certain of their exact properties.

The experiment was designed to focus on hydrocarbon pollution in a controlled freshwater environment, so the functionality of the FeO(OH) nanoparticles in uncontrolled or saltwater environments would require further testing. Although this experiment did not test such conditions, the synthesized FeO(OH) nanoparticles should work similarly within saltwater bodies; the hydrophobic interactions on which the extraction mechanism relies would remain intact and may even be strengthened by the presence of salts (13). On the other hand, the functionality of our FeO(OH) nanoparticles in

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flowing water systems is more unclear and requires further testing; the hydrophobic interactions that allow the FeO(OH) nanoparticles to target the pollutant may be facilitated or disrupted by water currents (14). The ability of these FeO(OH) nanoparticles to withstand different environmental conditions may be experimentally investigated and optimized within future experimental designs. The FeO(OH) nanoparticles were also designed to target all types of hydrophobic pollution but were only tested with hydrocarbons, specifically synthetic motor oil. Future research should test the efficacy of these FeO(OH) nanoparticles in extracting other hydrophobic pollutants such as microplastics through hydrophobic interactions (15).

Nanoparticles synthesized in previous research for the purpose of extracting hydrocarbon pollutants were found to remove between 0.5 and 1 g of hydrocarbon per gram of nanoparticles used (8). Such research employed a different modification of iron oxide nanoparticles and focused on the extraction of solid, hydrophobic pollutants such as microplastics. Our research found a similar positive correlation between the mass of FeO(OH) nanoparticles dispersed and the mass of motor oil extracted, but with around 3.57 g of motor oil removed per gram of FeO(OH) nanoparticles used. Further studies could test if the FeO(OH) nanoparticles synthesized in this study remove other types of pollutants tested in prior research in this same ratio to determine the most optimal nanoparticle modifications. The versatility of FeO(OH) nanoparticles could allow for application of this method in different environments and for different targeted pollutants. This study introduced a new possible variation of FeO(OH) nanoparticles that could be applied in commercial markets for the targeted extraction of pollutants in aquatic environments ranging from natural waterways to household water sources.

MATERIALS AND METHODS

FeO(OH) nanoparticle synthesis

To synthesize and modify the FeO(OH) nanoparticles, 0.10 g of FeO(OH) (Fisher Scientific), 3.80 g of oleic acid (Carolina Biological Supply), and 0.60 g of eicosane (Fisher Scientific) were mixed in a 150-mL beaker. The mixture was gradually heated under magnetic stirring to 360°C. This temperature was maintained for 105–120 minutes until the color changed from reddish-brown to black and the liquid evaporated. The beaker was then removed from the heat and set to cool overnight at room temperature. The next day, 10 mL of n-hexane (Fisher Science Education) was added to the beaker, and the mixture was stirred for 10–15 minutes. To precipitate the FeO(OH) nanoparticles, 20 mL of acetone (Fisher Scientific) was added. The solution was distributed into 13x100 mm centrifuge tubes and centrifuged at room temperature at approximately 448 x g for 30 minutes. The supernatant was then removed using a disposable pipette. The pelleted particles were redispersed with 10 mL of hexane, 20 µL of oleic acid, and 20 mL of acetone, and the tubes were then centrifuged at 448 x g for another 30 minutes; these wash steps were repeated two more times. After centrifugation and removal of the liquid, the tubes were left open at room temperature for 24 hours to allow the FeO(OH) nanoparticles to fully dry. The FeO(OH) nanoparticles were then poured from the centrifuge tubes into a beaker and stored at room temperature when not in use. The mass of FeO(OH) nanoparticles synthesized in each batch ranged from 0.118–0.230 grams.

Extracting pollutant with FeO(OH) nanoparticle dispersal

To make the polluted sample, 2 mL of synthetic motor oil (Mobil 1, synthetic 5W-30) was added to 20 mL of tap water. The initial oil and water volumes were weighed. The FeO(OH) nanoparticles were then added to the polluted sample. The target masses of FeO(OH) nanoparticles dispersed were 0.03, 0.04, 0.05, 0.06, and 0.07 g. The polluted sample with the FeO(OH) nanoparticles was lightly swirled for 2–3 seconds and allowed to sit for 4–6 minutes. The FeO(OH) nanoparticles were then extracted using a neodymium magnet covered in a plastic sheet to allow separation of the particles from the magnets. The plastic was weighed prior to usage and immediately following the extraction process with the FeO(OH) nanoparticles, oil, and water included. The used plastic was then left out for 48 hours to allow any water extracted to evaporate and then weighed again to record the mass of just the plastic, FeO(OH) nanoparticles, and oil. The polluted sample following the experiment was separated using a separating funnel and properly discarded. This procedure was done twice for each of the five FeO(OH) nanoparticle masses dispersed.

Statistical analysis

To determine if the positive relationship between the mass of FeO(OH) nanoparticles used and motor oil extracted is significant, we plotted our data points into Google Sheets to produce a scatterplot. We then applied a linear line of best fit to the graph, which produced the trendline 3.57x - 0.035 with R^2=0.844. To find the Pearson correlation coefficient, r, we took the square root of R^2=0.844 to obtain r=0.92. Using a critical value table with df=8 (n-2), we found that the minimum correlation coefficient needed for $p<0.05$ is 0.632 (16). Since $r=0.92>0.632$, we determined that $p<0.05$, and the positive linear relationship between mass of FeO(OH) nanoparticles dispersed and motor oil extracted is statistically significant.

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