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Section 2

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Experiment 8: Nanoparticle Aggregation – Ferrofluids

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Comparative Analysis of Magnetic Response in Ferrofluids: Magnetite (Fe_3O_4) vs. Cobalt Ferrite ($CoFe_2O_4$), vs. Cobalt Oxide (CoO) Nanoparticles

1. Introduction

A ferrofluid is a stable suspension of ferromagnetic (a material characterized by their strong attraction to magnets and ability to become magnets) nanoparticles in a carrier fluid, effectively combining the fluid properties of a liquid with the magnetic properties of a solid. Ferrofluids were discovered by NASA scientists in the 1960s, a result of an investigation to move liquids in space. Magnetic fields can control their location without the presence of gravity. Since then, ferrofluids have been employed in many fields, including chemical, electrical, nuclear, and biomedical technologies.[1] Ferrofluid is typically made up of magnetite (Fe_3O_4) suspended in water, stabilized with a surfactant such as tetramethylammonium hydroxide ($N(CH_3)_4^+ OH^-$) which prevents aggregation. This maintains a stable colloidal suspension, which means that the nanoparticles are evenly spread out within the water solvent.[1, 2]

Iron is a naturally ferromagnetic element. Its oxide, magnetite (Fe_3O_4), is a black, magnetic iron oxide material. It is known for its strong attraction to magnets, due to its

iron ions and crystal lattice structure. Thus, the magnetic phase of a ferrofluid is typically made up of magnetite nanoparticles.[2]

Cobalt is an element that shares many of the same properties as iron, notably its ferromagnetism and similar reactivity. While iron typically has a stronger magnetic response, there are instances where an alloy of cobalt and iron will exhibit a greater magnetic strength compared to just iron.[3] This knowledge prompted our investigation into whether cobalt-iron-oxide ($Co_xFe_{3-x}O_4$) ferrofluids could be synthesized, and if it could, its potential to achieve enhanced magnetic responsiveness compared to conventional iron oxide ferrofluids. Additionally, the idea of developing a purely cobalt-based ferrofluid was quite intriguing, as an exploration of an alternative ferromagnetic nanoparticle suitable for ferrofluids. While magnetite is well-studied, the potential for cobalt-based core alternatives for specific applications remains underexplored.

In this experiment, three different types of ferromagnetic nanoparticles-magnetite (Fe_3O_4), cobalt ferrite ($CoFe_2O_4$), and cobalt oxide (CoO) were synthesized to create three unique ferrofluids. We hypothesized that the magnetite ferrofluid would exhibit a similar or stronger magnetic response compared the cobalt ferrite ferrofluid, and a much stronger magnetic response compared to the cobalt oxide ferrofluid. While cobalt ferrite has its advantages – a high electromagnetic performance, high coercivity magetnocrysalline, and good sshowability, on top of sharing a similar spinel structure with magnetite[4] – magnetite's higher saturation magnetization and lower coercivity would make it respond quicker and more easily to everyday magnets, like the one we will be using for this experiment.[2] Cobalt oxide should also exhibit weaker magnetivity due to its antiferromagnetic nature and weak ferromagnetism at nanoscale.[5]

2. Experimental Procedure

The procedure for the production of each ferrofluid started with the synthesis of the ferromagnetic nanoparticles through precipitation, followed by stabilization from adding the surfactant. For the magnetite ferrofluid, 1.0 mL of 2.0 $FeCl_2$ and 4.0 mL of 1.0 M $FeCl_3$ was combined in a 100 mL beaker, creating a 2:1 molar ratio, and stirred using a magnetic stir bar on a Hot Plate/Stirrer. As the solution stirred, 50 mL of 0.7 M NH_4OH was added dropwise, forming a black precipitate, magnetite. After all the NH_4OH was added, the beaker was removed from the stirring plate and the stir bar removed from the beaker. The beaker was left alone to allow the black precipitate to settle for a few minutes, effectively seperating the magnetite from the excess liquid. After the magnetite settled, the excess liquid was removed with a pipette, and the purified solid magnetite was transferred to a plastic weighing boat. A magnet was placed under the beaker to concentrate the nanoparticles towards the bottom, minimizing the amount of magnetite being removed during excess liquid extraction. The excess liquid was mostly transparent, with a slight tint of black. Deionized water was used to transfer any magnetite left stuck in the beaker. A magnet was then used to pull the magnetic particles to the bottom of the weigh boat, while the rest of the liquid was removed with a pipette. The weigh boat was then placed in an ice bath for around 4 minutes. After 4 minutes of cooling, the weigh boat was removed from the ice bath and 2.0 mL of 25% Tetramethylammonium Hydroxide was added to the boat, with gentle stirring with a glass rod. Once mixed, the magnet was placed at the bottom of the weigh boat to separate the excess liquid from the now-stabilized ferrofluid. At this point, the magnetite

ferrofluid showed a strong attraction to the magnet, which made it very easy to extract the excess liquid. The ferrofluid was transferred to a glass vial to await testing in the day after, and a sample of the excess liquid was collected in a cuvette.

For the cobalt ferrite ferrofluid, the same procedure as magnetite was followed, except the 1.0 mL of 2.0 $FeCl_2$ was replaced with 6.0 mL of 2.0 M $CoCl_2$, which we made ourselves by combining 0.437g $CoCl_2 \cdot 6H_2O$ with 0.773 mL distilled water in every 1 mL of solution, to form $CoFe_2O_4$. The intended amount of 2.0 M $CoCl_2$ to be added to the $FeCl_2$ was 1.0 mL, but 6 mL was added on accident. However, this did not influence the properties of the cobalt ferrite, as the molar ratio was maintained. For the cobalt oxide ferrofluid, the 1.0 mL of 2.0 $FeCl_2$ and 4.0 mL of 1.0 M $FeCl_3$ was replaced with 5.0 mL of 2.0 M $CoCl_2$. Several key differences in the properties of these two nanoparticles were observed throughout the synthesis of the ferrofluids. The color of the cobalt ferrite precipitate was greenish brown, and the excess liquid was still transparent with a magenta tint, while the cobalt ferrite precipitate was a bright teal, and the excess liquid was transparent with a lighter, pinkish tint. Cobalt ferrite also demonstrated a unique interaction when reacted with the surfactant tetramethylammonium hydroxide, which turned the solution from teal to a dark blue. However, both ferrofluids demonstrated little to no magnetic response, resulting in an inefficient separation of the colloidal nanoparticles from the excess liquid during the purification phase. This yielded a low-viscosity suspension. Likewise, the ferrofluids were then transferred to glass vials and stored away for testing in the day after, and a sample of the excess liquid was collected in a cuvette.

In the day after, the vials were taken out, and a layer of excess liquid was observed in the cobalt ferrite and cobalt oxide ferrofluids – overnight, a large percentage of the excess liquid still mixed with the ferrofluid had been separated. This excess liquid was pipetted out, leaving behind a much slurrier version of cobalt ferrite and cobalt oxide ferrofluid compared to the day before. Additionally, the cobalt oxide ferrofluid had changed color again, from dark blue to a bright pink. A magnet was used to compare the strength of magnetic response by holding it against the side of each vial. The cobalt oxide and cobalt ferrite ferrofluid were found to have gained weak ferromagnetic properties, while the magnetite ferrofluid was the most attracted to the magnet.
The cuvettes containing samples of the excess liquid were utilized to measure the maximum absorbance of each, using a UV Spec device.

3. Results

Magnetic Response (1=Strongest, 3=Weakest)	Ferromagnetic Nanoparticle in Ferrofluid and Formula	Qualitative observations and description on magnetic response	Absorbance of Excess Liquid (AU)
1	Magnetite (Fe_3O_4)	Had a strong, clear response to the magnet when brought near (doesn't have to touch surface of vial to initiate response). Instantly attracted to both the stronger and weaker magnets. The strong magnet would "stick" to the wall	0.405

		of the vial. Could clearly see the “spikes”. It is the least fluid out of all the ferrofluids. Overall, its location is very easy to manipulate using a magnet.	
2	Cobalt Oxide (CoO)	Magnetic response was not very strong. When the magnet was brought to the wal of the vial, the ferrofluid was weakly but visibly attracted to the magnet, moving slightly. When moving the magnet up and down the side of the vial, little hill shaped portrusions of the pink ferrofluid show from the inside of the vial.	0.598
3	Cobalt Ferrite ($CoFe_2O_4$)	Magnetic response was barely noticeable, but still present. Was less noticable than the cobalt oxide. Small portrusions also show when magnet is moved up and down on the side, but are smaller in magnitude.	0.642

Table 1: Magnetic Response of Ferromagnetic Nanoparticles in Ferrofluid

4. Discussion and Conclusion

The results reveal a significant difference in the strength of magnetic response between the magnetite-based ferrofluid and the cobalt ferrite and cobalt oxide-based ferrofluids. Described as having a strong, immediate, response to the magnet, it clearly is the ferrofluid with the strongest magnetic attraction when compared to cobalt ferrite's "barely noticeable" and cobalt oxide's weakly attracted response to the magnet. This hierarchy in magnetic performance was further supported – though less conclusively – by UV-Vis absorbance measurements of the excess liquid samples. A lower absorbance number (meaning that there is a lower number of particles in the liquid blocking light from being absorbed) correlated to a stronger magnetic separation, as during the purification, a magnet was placed under the beaker, so the ferrofluid with the most ferromagnetic nanoparticle would have the lowest absorbance value. This is consistent with magnetite's superior performance, followed by cobalt oxide and then cobalt ferrite.

The experimental results partially supported the hypothesis: while the magnetite-based ferrofluid, as predicted, demonstrated the strongest magnetic response,, cobalt oxide and cobalt ferrite both placed unexpectedly, with cobalt oxide having a slightly stronger response. This contrasted with my hypothesis, where I stated that the cobalt ferrite was likely to exhibit similar or slightly weaker magnetic attraction as magnetite and cobalt oxide, which I anticipated would show little to no signs of magnetic response. Most notably, cobalt ferrite barely had any response to the magnet.

There are a few potential sources of error could have affected the results, but the effects would not be too substantial. For example, the NH_4OH was added quite quickly to the $CoCl_2$ and $FeCl_3$ solution, at around 2-3 drops per second, when the intended

rate was 1 drop per second (which was followed during the formation of magnetite and cobalt oxide). This rapid, inconsistent precipitation may have caused issues such as high pH zones, leading to the formation of clumpy aggregates, inconsistent particle size (some big, some tiny), and formation of other unwanted byproducts such as α-Fe_2O_3, which has weaker ferromagnetic properties, causing the ferrofluid to lose purity. These can lead to a less stable ferrofluid, and a lower saturation magnetization and higher coercivity, making the ferrofluid less responsive.

In this work, three different ferromagnetic nanoparticles were synthesized and their magnetic response when acting as a core in a ferrofluid was compared, while keeping the surfactant, carrier fluid, and synthesis method consistent. The results reveal that magnetite-based ferrofluid showcased the greatest magnetic response when a magnet is placed within close range of it, while the cobalt-containing cobalt ferrite and cobalt oxide ferrofluids demonstrated a much weaker magnetic response.

Cobalt-containing ferrofluids have their own advantages, such as having high electromagnetic performance, decent saturation magnetization, high coercivity, and good chemical and physical stability, which are utilized daily in many applications including in a range of commercial electronics.[4] However, the iron-based magnetite ferrofluid is still a substantially more effective choice for stronger magnetic attraction, proving its effectiveness in serving in roles such as in speakers, seals for devices, and drug delivery.[7]

5. References

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