Synthesis of Aqueous Ferrofluid

Procedure modified from *J. Chem. Educ.*, **76**, 943-948 (1999) by Jonathan Breitzer and George Lisensky. http://mrsec.wisc.edu/edetc/nanolab/ffexp/index.html
Additional information and procedures added by David A. Katz, Department of Chemistry, Pima Community College

Ferrofluids are colloidal suspensions of magnetic nanoparticles. Ferrofluids respond to an external magnetic field enabling the solution's location to be controlled through the application of a magnetic field. Fe_2O_3 magnetite nanoparticles can be produced by mixing Fe(II) and Fe(III) salts together in a basic solution. The particles must remain small and separated from one another in order to remain suspended in the liquid medium. Surfactants are used to prevent the nanoparticles from approaching one another too closely. Once prepared, ferrofluids have the captivating property of exhibiting “spikes” when placed in the proximity of a strong magnet.

Materials Needed

2.0 M $FeCl_2$ in 2 M HCl. (Each student needs 1 mL. Dissolve 19.9 g $FeCl_2 \cdot 4H_2O$ in 50 mL 2 M HCl.) NOTE: This solution is air sensitive and oxidizes to $FeCl_3$. The solution is best prepared the day it is used.

1.0 M $FeCl_3$ in 2 M HCl (Each student needs 4 mL. Dissolve 54.1 g $FeCl_3 \cdot 6H_2O$ in 200 mL 2 M HCl.)

The iron solutions need to be completely dissolved, well sealed and free of any particles. Color is important! Fe(II) is air sensitive. 1M $FeCl_3$ in 2 M HCl (shown at left) 2 M $FeCl_2$ in 2 M HCl (shown at right)

1.0 M NH_3 in water. (Each student needs 50 mL. Dilute 200 mL of concentrated ammonium hydroxide with water to 3.0 L.) Ammonia solutions lose concentration by evaporation of the ammonia. For best results, ammonia solutions should be prepared no more than 24 hours before use.

25% tetramethylammonium hydroxide in water (commercially available from Aldrich, Fisher, etc.). A strong, fishy, amine odor indicates hydrolysis products which may interfere with the synthesis reaction.

Beaker, 100 mL
Plastic weighing boats
Dropper
Glass stirring rod
Disposable gloves
Magnetic stir bar
Magnetic stirrer
Strong magnets such as cow magnets.

Safety Precautions

Wear approved eye protection at all times in the laboratory.

Ferrofluids are difficult to wash off and will permanently stain almost any fabric. Wear protective gloves when working with ferrofluid. Wear a lab coat or an apron to protect your clothes.

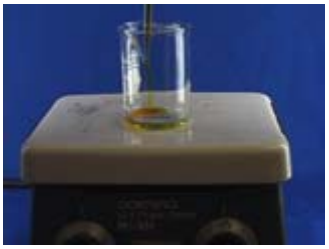
Ammonia vapors are irritating to the eyes and respiratory system. Work with ammonia solutions under a fume hood. In the event of eye contact, rinse well with water in an eye wash. For inhalation, move immediately to fresh air.

Tetramethylammonium hydroxide vapors are irritating to the eyes and respiratory system. It is readily absorbed through the skin. Work with tetramethylammonium hydroxide under a fume hood and wear protective gloves. In the event of eye contact, rinse well with water in an eye wash. For skin contact, wash the affected area with water. For inhalation, move immediately to fresh air. Get qualified medical attention.

Disposal

Dispose of all materials in the proper waste containers.

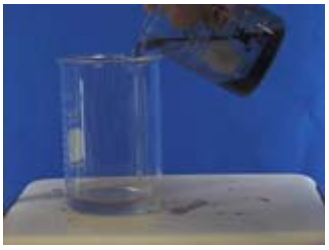
Procedure



Add 4.0 mL of 1M $FeCl_3$ and 1.0 mL of 2M $FeCl_2$ solution to a 100 mL beaker. Add a magnetic stirring bar and begin stirring.

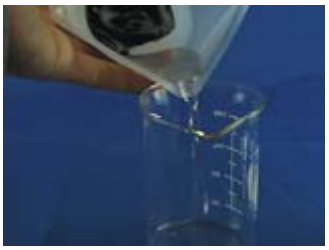
With continued stirring, use a dropper to add 50 mL of 1.0 M aqueous NH_3 solution slowly over a period of 5 minutes. After an initial brown precipitate, a black precipitate will form (magnetite). (Slow addition of the ammonia solution can also be accomplished using a buret or separatory funnel.)

Turn off the stirrer and immediately use a strong magnet to work the stir bar up the walls of the flask. Remove the stir bar with tongs or a gloved hand before it touches the magnet.



Let the magnetite settle, then decant (pour off) and discard the clear liquid without losing a substantial amount of solid. You can speed the settling process by putting a magnet under the container.

Transfer the solid to a weighing boat with the aid of a few squirts from a wash bottle.



Use a strong magnet to attract the ferrofluid to the bottom of the weighing boat. Pour off and discard as much clear liquid as possible. Rinse again with water from a wash bottle and discard the rinse as before. Repeat the rinsing a third time.

Add 1-2 mL of 25% tetramethylammonium hydroxide. Gently stir with a glass rod for at least a minute to suspend the solid in the liquid. Use a strong magnet to attract the ferrofluid to the bottom of the weighing boat. Pour off and discard the dark liquid. Move the strong magnet around and again pour off any liquid. If the ferrofluid does not spike, continue to move the strong magnet around, pouring off any liquid.

What happens when you move a magnet under the ferrofluid?

Questions

You will need to use the Internet to answer these questions. Reference the sources for your answers.

1. Ferrofluids are colloidal suspensions of nanoparticles of magnetite. What is the approximate size of the nanopartilces in a ferrofluid?

2. Describe the behavior of the ferrofluid when a magnet is placed near it.

3. What are some uses of ferrofluids? (Find at least two uses of ferrofluid.)