Synthesis of Magnetite (or Lodestone)

Background

Magnetite is an oxide of iron that has been known for thousands of years. Magnetite (Fe_3O_4) is thought to have been named after Magnesia, in Asia Minor. The importance of magnetite in history is due not only to its use as a source of iron, but also to it's magnetic properties. Magnetite, also commonly known as a lodestone, is a natural magnet and was the source of the first compasses for navigation. Today, magnetite is used widely in magnetic recording media (such as VCR tapes), in forming magnetic fluids, and as catalysts.

It may seem strange that the purpose of this experiment is to synthesize a "rock". Why would anyone want to make a mineral from chemicals such as iron sulfate? Natural minerals contain various amounts of impurities and have other defects that affect their properties. By synthesizing minerals, the impurities (if any) and properties can be controlled. It is easier to synthesize the mineral with the properties you want than to search the world for a vein of the mineral with the right properties.

Try to determine the oxidation state of the iron ions in magnetite (look at the formula) Do not read further until you try) Confused? The oxidation states of the three irons must add up to +8 (because of the four -2 oxygen ions). The simple answer is that the oxidation state is +2.67, but that requires partial electrons. One of the Fe ions is +2 and the other two are +3. Iron in the +3 oxidation states has 5 valence electrons and therefore has at least 1 unpaired electron. Paramagnetic species (species with unpaired electrons) are strongly attracted to magnetic fields. When a solid contains many paramagetic centers (such as many Fe^{3+} ions) this attraction can become rather intense and give rise to the properties that we expect from a magnet. The magnetite that you make may not act as a magnet, but it should be strongly attracted to a magnet (strong enough to stick to it).

Procedure for the synthesis of magnetite

Into a 400 mL beaker, add 2.0 g of $FeSO_4•7H_2O$ and 150 mL of water. Into a 100 mL beaker, add 60 mg of KNO_3 (or 50 mg of $NaNO_3$) and 0.80 g of KOH (0.56 g of NaOH) along with 60 mL of water. Heat the two solutions to 75 ºC and mix the two solutions while stirring (use a stirring rod, not a magnetic stir bar). Use tongs to pick up the hot solution (see Figure 1). A green suspension will form and rapidly turn black. Heat the solution to 90 ºC for 10 min while stirring. Cool the black suspension to RT and acidify the solution with 3 M HCl. Filter the precipitate with a Büchner funnel. Wash the black solid twice with 50 mL of water while in the funnel. Pull the filter paper out of the funnel, place it on a watch glass, and dry it in an oven at 100 ºC or on a hotplate. Scrape the black powder off of the filter paper.

Caution: The potassium and sodium hydroxide pellets are bases. If they or their solutions come into contact with skin, itching and then burning will result. If your skin starts to itch, wash with water for 10 min. Consult the MSDS for further warnings.

(a) (b)

Figure 1. Heating of the two solutions (a) and (b) pouring the solutions together. Note the use of tongs, do not try to pick up the hot beaker with bare hands.

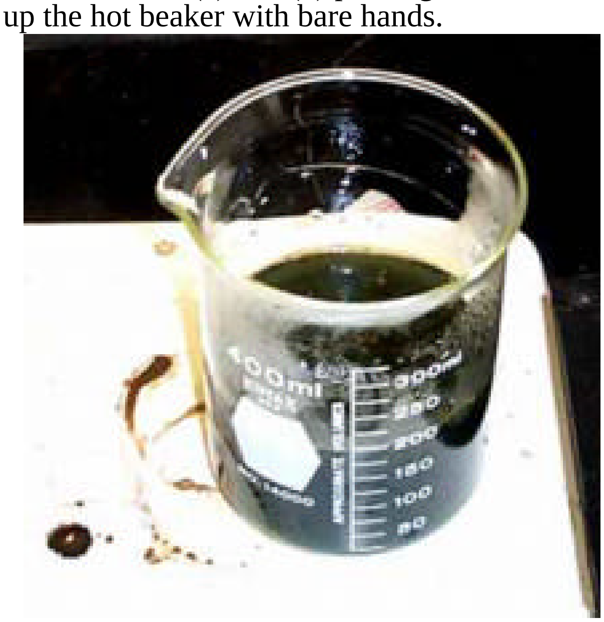

Figure 2. This green solution results from the combination of the two solutions above.

Figure 3. The filtration of the product

Figure 4. The final dried product (still on the filter paper).

Questions:

(1) How strongly was your magnetite attracted to a magnet? How strongly attracted to steel was it (did it stick to a steel object)?

(2) Look up information about magnets and magnetite. Are the magnetic properties of magnetite the same for all samples of magnetite? Why or why not?

(3) What other naturally occurring minerals act as permanent magnets?