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RESEARCH ARTICLE

Synthesis of Iron Nanoparticles in Aqueous and Nonaqueous Solutions and Their Use in Simulated Waste Remediation: An Experiment for First-Year College Students

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Iron nanoparticles are used to remove pollutants from soil and water in the environment and from industrial waste streams. A laboratory experiment described here is based on this application of nanotechnology. The procedure is appropriate for first-year undergraduate students with some background in general chemistry. Students synthesize iron nanoparticles via borohydride reduction of iron(III) chloride. Following this, students perform a series of reactions in which iron nanoparticles or bulk iron powders react with an aqueous dye. Periodic measurements of the absorbance in the visible spectrum show the decline in dye concentration. Students can analyze their results several different ways. The nanoparticles consume orders of magnitude more dye per gram of iron than the bulk powders due to their greater surface area. On the other hand, economic considerations make the bulk iron a more appealing choice. This dichotomy can lead to an interesting discussion among students about the apparent value of nanotechnology for environmental remediation. A modified version of the experiment is described in which the chemical reaction between a dye and iron particles occurs within a nonaqueous solvent. This procedure is more similar to the actual method used for removing organic pollutants from soil and ground water.

Keywords: Iron Nanoparticles, Redox Reactions, Pollution Remediation, Laboratory, First-Year Undergraduate Students.

1. INTRODUCTION

The effect of nanoparticles' greater surface area on reaction rates is one of the simplest nanotechnology concepts for students to learn. For a given mass of spherical particles, the total surface area increases with the square of particle radius. Chemical reactions taking place on a material's surface are more efficient using many smaller particles because they provide many more reaction sites compared to using fewer but larger particles (Hornyak et al., 2009). This effect is separate from any changes in band gap structure caused by different surface morphologies (e.g., defect sites) or quantum size effects (Alivisatos, 1996).

Tests conducted at contaminated sites show that iron nanoparticles can remove pollutants from soil, ground water and waste streams in a manner that is more effective than using bulk iron. In this case, the iron particles' small size not only increases the surface area per gram of iron but also allows the iron nanoparticles to flow through the soil more easily to reach any underground source of pollution. Engineers at NASA's Kennedy Space Center conducted pilot tests in which iron nanoparticles suspended in cooking oil were injected to decontaminate underground pools of organic solvents. The organic compounds mixed with the oil, increasing their interaction with iron nanoparticles (O'Hara, et al., 2006; Quinn, et al., 2005). Iron nanoparticles can remove inorganic pollutants also (Scott, et al., 2011). A helpful review of this topic is available (Zhang, 2003). Recent studies focus on improving the nanoparticles' effectiveness by using a combination of metals (Cho and Choi, 2010).

Advantages of using iron nanoparticles include its moderate reduction potential ($E^o(Fe^{2+}/Fe) = -0.447$ V),

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capability of reacting with a wide variety of compounds, high surface to volume ratio and formation of nontoxic iron hydroxides in water. Suspensions can be injected into the ground to deliver nanoparticles to subterranean points of contamination. Alternately, streams of contaminated water can flow through porous media, known as a permeable reactive barrier (PRB), impregnated with iron nanoparticles (Zhang, 2003).

The potential cost of remediating polluted sites is almost as staggering as the environmental damage that has occurred. In 2009 alone, The U.S. Environmental Protection Agency expended \$3 billion USD to remediate 75 of the most polluted sites. Costs are expected to rise as more sites are added to the list and cleanup continues at other sites (GAO-10-380, 2010). This does not include costs of remediating sites around the world. Environmental engineers and scientists continue to investigate alternative methods of decontamination which are more effective and less costly.

This article describes a laboratory experiment which includes the synthesis of iron nanoparticles and their reaction with a dye. While this dye could be viewed as a nontoxic simulated pollutant, water contamination is often due to colorants used in textile manufacturing (O'Neill et al., 1999). Students also measure the rate of dye removal using iron powders of different average diameters. Their results show the influence of particle size on reaction rate.

The learning objectives of this experiment are to:

- Synthesize iron nanoparticles.
- React nanosized and bulk iron particles with a dye and record the results.
- Perform corrections to determine the absorbance of the dye and graph this data.
- Analyze the results in terms of the cost, time and other factors.

First-year students in an Introduction to Nanotechnology Lab course (Winkelmann, 2009; Winkelmann et al., 2008) were successful in meeting all these objectives. Since this experiment allows students to observe the effect of surface area on a reaction rate and the application of nanoparticles for waste removal, it is most appropriately implemented within a nanotechnology laboratory course. It may also be used successfully in a second semester general chemistry course since students will have some knowledge of kinetics from their lecture class. It would serve as an excellent introduction to applications of nanotechnology. However, the experiment described here is not designed to necessarily replace more common kinetics experiments which allow students to determine the reaction's rate law or activation energy.

The idea for the experiment was inspired by a published description of a general chemistry inquiry experiment using bulk iron for waste water removal (Balko and Tratnyek, 2001). The authors of the present article extend the study of the reaction between iron and dye in several ways:

(1) introducing the synthesis and use of iron nanoparticles,

(2) comparison of reaction rates on a per gram and cost basis, and

(3) providing a procedure for oil-based reactions between the iron and dye pollutant.

2. EXPERIMENTAL DETAILS

In order for students to fully appreciate the phenomena that they observe during this experiment, they must understand several general chemistry lecture concepts and some new topics and competently use a visible spectrometer. Oxidation-reduction reactions, precipitation reactions (formation of iron nanoparticles) and reaction rates form the basis for understanding the iron nanoparticle synthesis and their reaction with the dye. Both environmental remediation methods and surface reactions will be new topics for most students. While they will undoubtedly know about environmental pollution in general, the instructor may want to provide specific information about the uses of iron nanoparticles for pollution remediation (e.g., information found in Refs. O'Hara et al., 2006; Quinn et al., 2005; Scott et al., 2011; Zhang, 2003). Students' successful completion of the experiment depends on their ability to operate a spectrophotometer and convert absorbance values to concentrations. They should have experience operating the instrument and using Beer's Law. For students who do not know how to measure absorbance, the instructor should demonstrate the use of the instrument and provide some background theory. An instructor might convey all this information to students in a straightforward pre-lab lecture (i.e., a non-inquiry style of teaching), as the authors used with their students. Alternatively, the instructor could use inquiry pedagogy. For instance, students could perform this experiment without a pre-lab explanation of the relationship between particle size and reaction rate then determine for themselves the underlying cause of the trend in reaction rates.

Instructors should give thought to a potential misconception which may arise among students: the practice of adjusting experimental data. In this experiment, iron hydroxide products obscure the absorbance of the dye during the reaction. The procedure describes how students will measure the change in the background absorbance due to suspended iron hydroxide particles and subtract that value from the absorbance at λ_{max} of the dye. This adjustment or correction is entirely legitimate and necessary in the context of the experiment but students may fail to see the difference between it and simply changing their numbers so that their experimental result is more accurate. This is an opportunity for instructors to explain the circumstances under which researchers may manipulate data and when that is not allowed.

3. PROCEDURE

Standard laboratory equipment is used in this experiment. A visible light spectrophotometer is necessary for quantifying reaction rates. Spreadsheet and graphing software, such as Microsoft Excel, is recommended for either the instructor to use during the laboratory session or for students to use as they write their laboratory reports. Corn oil was purchased from a grocery store. All other chemicals were purchased from Fisher Chemical unless noted otherwise and required no additional purification. Deionized water was used to prepare all aqueous solutions. The authors use Vernier's LabQuest data collection units with Ocean Optics visible light spectrophotometers but other systems may work just as well.

It is advised that students calibrate the spectrophotometer and have all other materials and solutions ready before performing the synthesis. Students should reread the procedure for synthesizing nanoparticles and performing the reaction with indigo carmine (IC) dye. The iron nanoparticles degrade within 15 minutes so students must work efficiently.

Students prepare iron nanoparticles via the following reaction:

$$4Fe^{3+}(aq) + 3BH_4^-(aq) + 9H_2O(l) \rightarrow 4Fe(s) + 3H_2BO_3^-(aq) + 12H^+(aq) + 6H_2(g)$$

In this redox reaction, iron(III) cations are reduced to elemental iron and the borohydride anions (BH_4^-) are oxidized. Excess borohydride is used to accelerate the synthesis reaction and limit the growth of iron particles. The original published procedure (Zhang, 2003) described the formation of 70 nm diameter particles as measured by TEM but the current authors did not verify this independently. Borohydride also reduces water to form hydrogen gas, which students observe as bubbles that form during the reaction.

Students add 5.0 mL of 0.20 M $NaBH_4$ to a small beaker and begin stirring the solution. They add an equal volume of 0.050 M iron(III) chloride dropwise over a period of several minutes with continued stirring. The rate of $FeCl_3$ addition should be ~1 drop per second. Large chunks form of iron and excessive foaming occurs when adding $FeCl_3$ too fast. If this occurs, it is best to begin again with a new solution of $NaBH_4$. It is normal to see small bubbles of hydrogen gas form during the synthesis. The solution turns gray during the synthesis but will become translucent after it is allowed to set, unstirred, for five minutes. The nanoparticle suspension begins to turn a yellow-brown color after an additional ten minutes. This color change indicates that the iron nanoparticles are reacting with oxygen in the air to form nonreactive iron hydroxides. Such a suspension will not react with the dye. Students should remake their nanoparticle solution if they do not use it in a timely manner.

In the next portion of the experiment, students perform a second reaction in which the iron nanoparticles are oxidized and indigo carmine dye is reduced. To transfer nanoparticles from the synthesis beaker to the cuvette, students should stir the nanoparticle suspension and draw a small volume into a pipette. Fifteen drops of this suspension (0.5 mL) are added to a cuvette containing 3.5 mL IC dye. (The cuvette should be as full as possible to minimize the amount of air in the system.) Using a stopwatch, students begin timing the reaction immediately upon the addition of the nanoparticles. After capping the cuvette, students should shake the nanoparticle suspension so that the nanoparticles mix with the dye. They monitor the absorbance of the dye solution using a visible spectrophotometer. Measurements are recorded every 30 seconds as the color of the IC dye. The contents of the cuvette are shaken between each absorbance measurement to ensure that all nanoparticles are exposed to the solution. The cuvette should remain capped through the reaction, which lasts for five minutes. Iron nanoparticles reduce the IC dye to form colorless products.

Absorbance measurements are complicated by the fact that the iron nanoparticles and the iron hydroxide products also absorb and scatter light from the spectrophotometer. These issues are corrected in the following ways.

First, students record absorbance values at λ_{max} (610 nm, $\varepsilon_{610} = 18200\ M^{-1}\ cm^{-1}$) and at another wavelength at which the IC dye does not absorb (e.g., 750 nm). This second absorbance value measures the scattering that takes place due to the formation of iron hydroxides as the iron nanoparticles reduce the dye. A correction factor of 1.5 is used to account for the higher absorbance of the iron hydroxide products at 610 nm compared to their absorbance at 750 nm. This corrected absorbance value is subtracted from the absorbance measured at 610 nm to yield the "adjusted absorbance" (i.e., adjusted absorbance@610 nm = measured absorbance at 610 nm − 1.5 × measured absorbance@750 nm). The adjusted absorbance represents the absorbance of just the indigo carmine dye without any interference from the iron hydroxide products. It is this value which students convert to dye concentration and graph as a function of time to determine the reaction's initial rate.

During the reaction, iron nanoparticles scatter light passing through the cuvette. This problem is solved by placing a magnet underneath the cuvette to pull the nanoparticles to the bottom, allowing the light beam of the spectrophotometer to pass through (Balko and Tratnyek, 2001). Attracting the nanoparticles to the bottom of the cuvette takes 5–10 seconds. Once they settle, the magnet can be pulled away and the cuvette placed into the spectrophotometer. Iron hydroxide does not interact with a magnetic field so they remain floating in the solution, necessitating the absorbance measurements at 750 nm as described above.

Students repeat the reaction between iron and indigo carmine dye using commercially available powders. Prior to the lab session, the instructor can separate the powders into different sizes using a particle sifter. Commercial powders usually contain a range of particle sizes up to a certain diameter. Students in the Introduction to Nanotechnology Lab had access to particles with average diameters of 1.5 mm, 0.15 mm and 0.07 mm. The procedure for performing the reaction with indigo carmine dye using commercial iron powders is the same as with nanoparticles except with one change. Since the bulk powders are dry and not a suspension, the students should add 15 drops of DI water to their cuvette, followed by the iron powder. This ensures that the initial concentration of dye in the cuvette is the same for all reactions. Experiments using 0.25 g of powders yielded satisfactory results.

After completing each reaction, students can enter their data into a class spreadsheet which graphs their data as dye concentration versus time. To save class time, the instructor could set up the spreadsheet to automatically perform many of the calculations as students enter their raw data. The instructor tabulates the initial rates of the class's reactions and discusses the results with the students. Presenting the data in this manner alleviates the need for students to know how to use a spreadsheet software program.

The authors encountered no unusual safety issues when performing this laboratory experiment. Students should wear goggles and lab coats or aprons at all times. A small amount of hydrogen gas forms in solution during the synthesis of iron nanoparticles but no sparks or open flames are used in this experiment. Students should dispose of waste solutions according to school policies. They should wash glassware and cuvettes which contained any iron compounds as soon as possible so that iron hydroxides do not stain. Discolored glassware requires additional cleaning in a base bath. Stained plastic cuvettes should be thrown away since they cannot be cleaned. Prompt rinsing of plastic cuvettes prevents most iron hydroxide stains from forming. Students should check the condition of the cuvette after each experiment with iron. A stained cuvette can result in less accurate absorbance measurements.

To expedite the completion of this experiment, the lab instructor should prepare all solutions beforehand. The sodium borohydride will react slowly with water so that solution must be prepared within an hour prior to use to ensure best results. Iron(III) chloride and indigo carmine dye solutions are more stable but they will degrade within a month. Authors found that the reactivity of the indigo carmine dye with iron particles changes over time even if the dye's absorbance remains constant during storage. Solutions can be stored at room temperature before use.

Plastic cuvettes are permissible for spectrophotometer measurements although the iron hydroxide products can irreversibly stain the plastic. Plastic cuvette caps or paraffin film work equally well for covering the cuvette when shaking the contents. Do not allow students cover the cuvette with their fingertips and shake since this exposes their skin to chemicals and contaminates the dye-nanoparticle suspension.

The following procedure uses corn oil instead of water as the solvent for the reaction between iron nanoparticles and 2,6-dichloroindophenol (DISS) as the organic dye. Students in a teaching laboratory have not completed this procedure although some undergraduate authors did successfully prepare the dye and perform the reaction with iron nanoparticles as part of their research for this project. It is included here in the interest of providing readers with a more realistic variation of the procedure which is consistent with a large scale method used to remove organic pollutants from contaminated soil. Other dyes and organic solvents may work equally well.

A solution of 2,6-dichloroindophenol in corn oil is prepared according to the literature (Kelly, 1993). Rapid mixing creates a water-in-oil emulsion which should be avoided. Complete dissolution of the DISS dye in the corn oil can take several hours so the instructor should prepare this solution prior to the laboratory session. The DISS dye in corn oil is stable for at least 24 hours. Students prepare the iron nanoparticles as described in the previous procedure with one modification. Instead of adding a portion of the aqueous suspension of nanoparticles to the dye, students should retain all the nanoparticles in the beaker and pour out all the aqueous solution. This is accomplished by holding a strong magnet under the beaker used to prepare the nanoparticles and decanting the solution. The magnet holds iron nanoparticles and the stir bar in place. The corn oil DISS solution can then be poured over the iron nanoparticles and stirred. Iron nanoparticles are stable for a few hours in oil before signs of oxidation appear. Due to the viscosity of the oil, a magnet cannot pull the nanoparticles to the bottom of the cuvette between absorbance measurements in a reasonable amount of time. As with the experiment performed in an aqueous dye solution, absorbance of the dye ($\lambda_{max} = 517$ nm) is calculated by subtracting the absorbance due to the suspended particles at 750 nm multiplied by a factor of 1.1. (If another organic solvent is used, the instructor can determine the corresponding factor by preparing a suspension of the nanoparticles and measuring its absorbance as the nanoparticles stir in the solvent without any dye.)

4. RESULTS AND DISCUSSION

Typical data generated by students performing the aqueous dye reaction is shown below in Figure 1. As expected, the initial rate of dye consumption increased as the particle size decreased with the largest particles (1.5 mm, ▲) yielding the slowest reaction and the nanoparticles (♦) reacting the fastest. Although the data clearly shows that the nanoparticles consumed much less dye overall than the

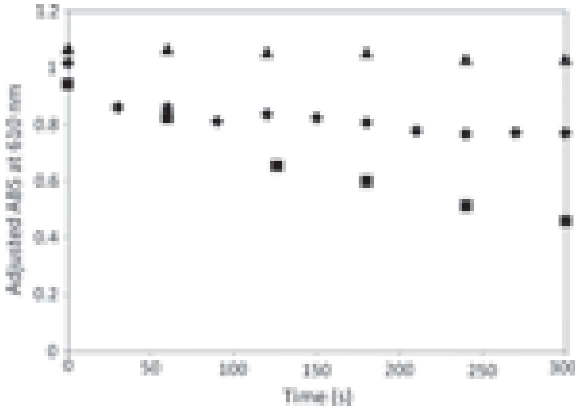

Fig. 1. Typical student results showing the adjusted absorbance of IC dye as a function of time during the reaction involving iron particles with different diameters: (♦) 70 nm (synthesized), (■) 0.07 mm and (▲) 1.5 mm.

0.07 mm particles (■), this is not an appropriate comparison. Students used 0.25 g of bulk particles but added only 7.0×10^{-4} g iron nanoparticles. (The synthesis procedure yields 0.014 g iron nanoparticles assuming complete conversion of the $FeCl_3$, which is limiting. Students use only 0.5 mL of the 10 mL suspension they prepare.) Therefore, the consumption of dye was much greater when considered on a per gram iron or per mole iron basis. The complete consumption of the small amount of iron nanoparticles accounts for the reaction rate slowing after 90 seconds.

Variation of absorbance measurements among the different lab groups increased for reaction times greater than 3 min. This caused comparisons of lab groups' overall reaction rates to be difficult. The variation in absorbance measurements performed in the middle and end of the reaction were observed when students used either nanoparticles or bulk particles. The cause of this variation could be students' inconsistent shaking of the cuvette, not recording accurate measurement times or other errors associated with not following the procedure carefully. The authors experienced consistent results over the entire period of the reaction when performing the experiment themselves. Due to the possibility of inconsistent results at long reaction times, instructors may choose to limit data analysis to students' initial reaction rates.

Initial reaction rates are based on the slope of dye concentration versus time data recorded at the beginning of the reaction. Initial rates for the different sized iron particles are summarized in Table I. Results agreed fairly well among all student groups with a coefficient of variation (standard deviation expressed as a percentage of the mean) of 14 to 19%. As noted previously, students use a much smaller mass of nanoparticles compared to the amount of bulk iron powder. Therefore, analyzing the rate in terms of the amount of dye consumed during the initial reaction period (90 s) per gram of iron added to the cuvette shows that the nanoparticles consume much more dye than the bulk powders.

Nanoparticles are more effective at removing the dye but this does not mean that bulk iron powders would be a poor choice of materials. The right column of Table I shows an estimated cost of chemicals ($NaBH_4$, $FeCl_3 \cdot 6H_2O$ and iron powders) per gram of iron consumed. Iron powders remove less dye but they are inexpensive, suggesting that the most economical choice would be 0.07 or 0.15 mm iron powders. This is an interesting starting point for a discussion with students about how and when it is appropriate to use nanomaterials as opposed to more well known bulk materials. If they consider the use of iron particles for removing pollutants (maybe something more toxic than indigo carmine dye) from a lake, what factors should they consider? On one hand, rapidly removing the pollutant could be the most important factor so they are willing to pay more to use nanoparticles. Students might realize that chemicals are just one portion of the overall budget for such a project. Synthesizing the nanoparticles would require more labor than using off-the-shelf iron powders. In this experiment, the iron powders were sifted to separate them into different particle sizes but that would not be necessary for the purpose of remediation. Materials and labor costs would change upon scale up in ways that are not obvious. While there is no definite "right" answer to the question of which size of particles are best, it is worth having the discussion with students so they realize the many variables that must be considered. It also highlights the idea that although nanotechnology is considered cutting-edge science, it does not mean it is the solution to every problem.

The results discussed above were collected by first-year science and engineering students enrolled in Florida Tech's

Table I. Table of average initial reaction rate data gathered by students in an introductory nanotechnology lab course and estimated consumable materials costs in U.S. Dollars.

Average particle diameter	Average initial rate with standard deviation	Initial ratio of dye consumed to iron used	Materials cost per gram iron
70 nm	$2.3\ (\pm 0.4) \times 10^{-7}$ M s^{-1}	2.1×10^{-5} mol dye (g iron)$^{-1}$	\$6 (g iron)$^{-1}$
0.07 mm	$6.9\ (\pm 1.3) \times 10^{-8}$	7.2×10^{-7}	0.06
0.15 mm	$7.1\ (\pm 1.0) \times 10^{-8}$	7.2×10^{-7}	0.06
1.5 mm	$8.4\ (\pm 1.4) \times 10^{-9}$	1.1×10^{-8}	0.17

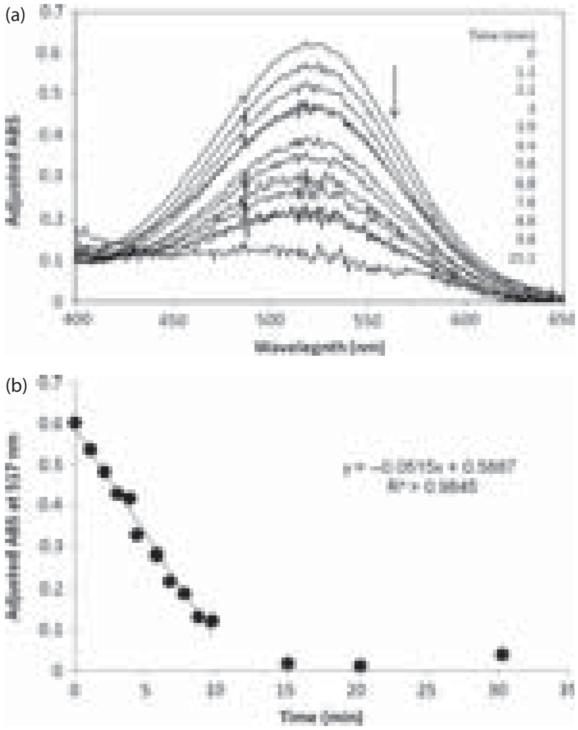

Fig. 2. Preliminary data showing (top) the decrease in absorbance as the DISS dye reacts with the iron nanoparticles and (bottom) the DISS dye absorbance as a function of time during the reaction between dye and iron nanoparticles.

Introduction to Nanotechnology Laboratory. This is a one-credit elective course. All students had completed General Chemistry I, a prerequisite for the class and most, but not all, were enrolled in General Chemistry II concurrently.

All students successfully synthesized the iron nanoparticles and performed reactions with the aqueous dye. Data among all student lab teams were consistent with each other and with the hypothesis that small particles react more rapidly with the dye. After completing the experiment, most students engaged in a discussion of the applications of iron nanoparticles versus bulk particles for environmental remediation. Their successful completion of the experiment and analysis of their data demonstrates that they met the learning objectives of this experiment and that the experiment is appropriate for first-year students with some background in general chemistry.

The authors have introduced this experiment in General Chemistry I lab and found that students require much more time to complete the procedure. The slower pace appears to be the result of General Chemistry I students' lack of laboratory experience and their unfamiliarity with kinetics. In order to accommodate these students, the authors have created a multi-week inquiry-based lab project in which students study the reaction between iron and indigo carmine dye. A description of that version of the experiment will be provided in a future article.

A similar reaction involving iron nanoparticles and DISS dye in an organic solvent (corn oil) was briefly investigated as part of this study. Figure 2 illustrates results of a reaction between DISS dye and iron nanoparticles performed in corn oil. The spectra in the top graph have been repositioned along the *y*-axis in order to remove the absorbance due to particle scattering (each spectrum is shifted downward so that the absorbance at 750 nm is equal to zero). In the lower portion of Figure 3, the absorbance of the dye solution at 517 nm has been corrected using the adjustment factor of 1.1 as described previously and plotted as a function of reaction time.

In this case, all the dye is consumed within 15 minutes. Reactions involving greater initial amounts of DISS dye can continue for over 30 minutes. The initial rate of dye remediation is linear though the authors have not performed a thorough analysis of the kinetic data for this system. These preliminary results indicate that iron nanoparticles can react in nonaqueous solvents and the reactions yield results that are appropriate for a general chemistry II laboratory experiment.

5. CONCLUSIONS

Results presented here demonstrate that first-year students can synthesize iron nanoparticles and use them to perform a reaction with an aqueous dye. Students can consider the implications of their data in terms of the reaction's rate or the cost of the materials (bulk vs. nanoparticles). A procedure and preliminary results indicate that a similar reaction between iron nanoparticles and an organic dye can take place in a nonaqueous solvent also.

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