JOURNAL OF CHEMICAL EDUCATION

Laboratory Experiment

pubs.acs.org/jchemeduc

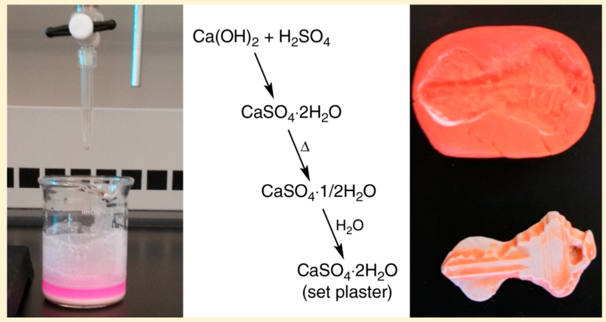
Synthesis, Dehydration, and Rehydration of Calcium Sulfate (Gypsum, Plaster of Paris)

Gergely Sirokman*

Department of Sciences, Wentworth Institute of Technology, Boston, Massachusetts 02115, United States

Ⓢ *Supporting Information*

ABSTRACT: Engaging students who are not chemistry majors is a significant challenge. Plaster of Paris is a substance well-known to undergraduate students from early life arts-and-crafts endeavors. Within, a lab is described that connects the synthesis and use of plaster of Paris with a number of chemical principles including stoichiometry, acid−base titration, hydration, and reaction yield.



KEYWORDS: *High School/Introductory Chemistry, First-Year Undergraduate, Laboratory Instruction, Physical Chemistry, Hands-On Learning/Manipulatives, Inquiry-Based/Discovery Learning, Acids/Bases, Materials Science, Nonmajor Courses*

The problem of teaching chemistry to nonmajors is a long-discussed issue.[1] Taught without context, chemistry can be perceived as abstract and having little to do with the real world.[2] A significant portion of the student body taking introductory chemistry at Wentworth Institute of Technology is made up of construction management and civil engineering and technology students. To engage these students, laboratories relating the relevance of chemical properties to materials used in these disciplines were developed. One such material is plaster of Paris, which is a well-studied compound important to the construction industry.[3] Industrially, plaster in the form calcium sulfate dihydrate is mined, dehydrated, and then rehydrated at the point of use. The mechanisms by which calcium sulfate dihydrate forms and plaster sets have been studied in depth.[4] Plaster is also a compound many people have experience with as part of arts-and-crafts projects from their youth.

■ BACKGROUND

Plaster of Paris is the hydrate of calcium sulfate, $CaSO_4$. When gypsum, $CaSO_4{\cdot}2H_2O$, is carefully heated to 120 °C, some of the water is driven off to give calcium sulfate hemihydrate, $CaSO_4{\cdot}{}^1/_2H_2O$.[1] Calcium sulfate hemihydrate reacts with water relatively rapidly to regenerate calcium sulfate dihydrate. This process turns powdery hemihydrate into the solid mass of dihydrate that we commonly think of as plaster, $CaSO_4{\cdot}2H_2O$. Care must be taken, however, as heating the hemihydrate over 180 °C will result in driving all of the water off of the compound, leaving anhydrous calcium sulfate, $CaSO_4$.[5] This material rehydrates very slowly, giving it little value as construction material.

■ EXPERIMENTAL OVERVIEW

In this lab, $CaSO_4{\cdot}2H_2O$ is first generated via a titration of calcium hydroxide with sulfuric acid. Afterward, it is dehydrated to the hemihydrate form, which can be rehydrated in a mold. Throughout this process, students are introduced to numerous topics from the lecture, thus emphasizing the importance of chemistry in other fields. A direct connection can be made by pointing out that reactions similar to the one in this experiment have been studied for industrial purposes.[6]

This experiment was designed to be run in two laboratory sessions, each two hours in length. This time constraint and the desire for well-controlled heating of the gypsum necessitated a break between the two halves of the lab. The exercise was performed by students in groups of two or three. The students are predominantly engineering technology or construction management students who take the Chemistry for the Built Environment course. The main goals of the laboratory exercise for students are

- To perform a titration to synthesize plaster.
- To observe and understand the effect of hydration and dehydration on the physical properties of plaster.
- To apply stoichiometric calculations in a practical situation.
- To recognize connections between chemistry and their own field of study.

In this experiment, students learn about acid−base neutralization and titration via the synthesis of gypsum. The importance of stoichiometry is underscored several times. Stoichiometric calculations are needed to calculate the amount

Published: March 7, 2014

of sulfuric acid required to neutralize the calcium hydroxide. Success with these calculations can lead to improved experimental results, and errors can become immediately evident if the neutralization is not complete. Stoichiometry is also required to perform calculations involving the dehydration and rehydration of calcium sulfate. Yields of reaction are also covered, with examples of how yield can be dependent on both the underlying chemistry and on technique. Students also learn about waters of hydration and the chemistry behind the preparation of plaster. Formation of solid plaster from ground powder is an excellent showcase for the role of water in binding together certain types of bulk solid. Solubility and precipitation can be addressed as part of the synthesis process. Heat of reaction can be discussed, and it may be worthwhile to note for students that the heat generated by large amounts of plaster in contact with the body can lead to significant injury.[7]

■ EXPERIMENTAL DETAILS

Calcium hydroxide (2 g) was measured into a small beaker and suspended in 15 mL of water. Several drops of phenolphthalein (1% in ethanol) were added to the suspension, coloring it pink. A buret was filled with 1.0 M sulfuric acid. The calcium hydroxide was titrated with sulfuric acid until the solution remained white for 5 min while being agitated. The suspension was then poured into preweighed test tubes and centrifuged for 10 min. The supernatant solution was decanted, and the test tubes containing the white solid were placed in an oven.

The test tubes were heated at 60 °C for 12 h, then at 120 °C for 36 h, then cooled and allowed to sit, covered, until the next lab session. The test tubes were weighed to find the yield for the reaction. The dehydrated plaster was removed from the test tube and weighed to find a recovered yield, with the typical yield being approximately 3 g.

Artists' clay was used to make molds for casting the plaster by pressing a small object into the clay. The recovered plaster was crushed into a fine powder with a scoopula and mixed with 3.0 mL of water to make a slurry. The plaster was then poured into the mold and allowed to set.

■ ALTERNATIVE PROCEDURE

The entire lab can also be performed in a single three or four hour lab session with the following alterations. This procedure has been tested by the instructor but not used in a lab with students.

After synthesis, the calcium sulfate dihydrate was collected using suction filtration. The product was washed with several small portions of water to remove any trace sulfuric acid. The product was transferred to a crucible with a cover, which was heated for 15 min with a BernzOmatic propane torch. The evolution of steam is observed during this process. The product was allowed to cool and then crushed into a fine white powder. The powder is then readily rehydrated into plaster as described above. A 10 min period of heating generated a plaster that set very slowly, taking overnight to solidify.

■ HAZARDS

Safety goggles and gloves should be worn throughout this lab. Calcium hydroxide and sulfuric acid are both caustic. To prevent inhalation, handling powdered calcium hydroxide and powdered plaster of Paris under a fume hood, or with a dust mask, is advisable. Calcium hydroxide is best dissolved in water by adding the calcium hydroxide to water in small portions. Sulfuric acid is best handled by dissolving the acid by adding it to water, though students should receive a prepared 1.0 M solution to limit the hazards of dealing with a concentrated solution of acid. Calcium hydroxide or sulfuric acid exposure to the skin or eyes should be treated with immediate flushing of the area with copious amounts of water. Plaster of Paris is potentially very damaging for plumbing; students must be advised very strongly against sending any waste products down the laboratory drains. If using the alternative procedure, crucibles must be checked for chips and cracks before use to prevent them from breaking during the heating process.

■ RESULTS AND DISCUSSION

The titration of calcium hydroxide was a tedious process because of its slow dissolution. Thus, as the titration proceeds, a seemingly white solution would revert back to pink after being allowed to sit for a minute or two. Emphasizing the importance of allowing the reaction to sit for 5 min after achieving a white solution is critical to student success. In practice, the titrations performed by students were usually short of 1 equiv of sulfuric acid. This incomplete reaction often became apparent after centrifuging the products, as the solution reverts to a pinkish color. Depending on time constraints, an addition of more sulfuric acid may be called for. Nonetheless, slightly pink powder proved to behave well for the second part of the experiment.

The dehydration process had to be executed carefully. Initial attempts were carried out with the oven running at 120 °C from the start. This temperature, however, resulted in trace water boiling within the test tubes and foaming calcium sulfate that overflowed from the test tubes. On occasion, the foam was ejected with force, covering the inside of the oven. Preheating the sample at 60 °C drove off the remaining free water and subsequent heating at 120 °C led to the formation of the hemihydrate of calcium sulfate.

Student yields ranged from 50% to 90%. Students with particularly low yields were instructed to consider the consequences of an incomplete titration of the calcium hydroxide on the product yield. Loss of product from imperfect transfer techniques was most common, though incomplete conversion to calcium sulfate, as evident from phenolphthalein-pink colored batches of plaster, was also an issue.

The powdered calcium sulfate hemihydrate was readily rehydrated, though the amount of water required had to be adjusted on a case-by-case basis. Plaster not powdered before addition of water tended to form a wet slurry that did not set well. Given the relatively small amount of plaster, too small a quantity of water would lead to a paste that would not pour. Excess water added to these mixtures improved flow but significantly lengthened setting time.

■ CONCLUSION

A laboratory exercise engaging nonchemistry majors was designed and executed successfully. Students responded positively to the connection of real-world materials to the theory they were studying in chemistry. The arts-and-crafts portion of the lab got the students particularly excited. The lab allowed for students to combine several theoretical principles with practical skills they learned over the semester in one exercise, including acid−base reactions, solubility, precipitation, and stoichiometry.

dx.doi.org/10.1021/ed400004b | J. Chem. Educ. 2014, 91, 557−559

ASSOCIATED CONTENT

Supporting Information

Instructor notes and student handouts. This material is available via the Internet at http://pubs.acs.org.

AUTHOR INFORMATION

Corresponding Author

*Corresponding Author E-mail: sirokmang@wit.edu.

Notes

The authors declare no competing financial interest.

ACKNOWLEDGMENTS

I thank Laurie Grove, Robert Moran, Armen Casparian, Jarad Kohl, and Paloma Valverde for their feedback and for having conducted the lab for students. I would also like to thank all of the students in CHEM 120 and CHEM 360 classes who have participated in the lab and given feedback.

REFERENCES

(1) (a) Vanderryn, J. The teaching of chemistry to nonmajors: A survey. *J. Chem. Educ.* **1958**, *35* (5), 256−259. (b) Wolke, R. L. Chemistry for the nonscience major: An experiment in relevance. *J. Chem. Educ.* **1970**, *47* (12), 788. (c) Finholt, A. E.; Miessler, G. L. Chemistry for antiscience students: A "great conversation" in chemistry. *J. Chem. Educ.* **1986**, *63* (4), 331−333. (d) Walczak, M. M.; Walczak, D. E. Do Student Attitudes toward Science Change during a General Education Chemistry Course? *J. Chem. Educ.* **2009**, *86* (8), 985−991.

(2) Barker, G. K. Why Do I Have to Study Chemistry? *J. Chem. Educ.* **2000**, *77* (10), 1300.

(3) Fesling, W. A.; Potter, A. D. Gypsum and gypsum products. *J. Chem. Educ.* **1930**, *7* (12), 2788−2807.

(4) (a) Saha, A.; Lee, J.; Panera, S. M.; Bräeu, M. F.; Kempter, A.; Tripathi, A.; Bose, A. New Insights into the Transformation of Calcium Sulfate Hemihydrate to Gypsum Using Time-Resolved Cryogenic Transmission Electron Mocroscopy. *Langmuir* **2012**, *28* (30), 11182−11187. (b) Wang, Y.-W.; Kim, Y.-Y.; Christenson, H. K.; Meldrum, F. C. A new precipitation pathway for calcium sulfate dehydrate (gypsum) via amorphous and hemihydrate intermediates. *Chem. Commun.* **2012**, *48*, 504−506.

(5) Ramachandran, V. S.; Paroli, R. M.; Beaudoin, J. J.; Delgado, A. H. *Handbook of Thermal Analysis of Construction Materials*; Noyes Publications: Norwich, NY, 2002; p 455.

(6) Bard, F.; Bilal, E. Semi-Batch Precipitation of Calcium Sulfate Dihydrate from Calcite and Sulfuric Acid. *Carpathian J. Earth Environ. Sci.* **2011**, *6* (1), 241−250.

(7) Wainwright, M. Girl loses finger in school art lesson. *The Guardian (online)*. http://www.theguardian.com/education/2009/oct/12/girl-loses-fingers-school-art (accessed Feb 2014).