Volume: 3 Issues: 23 [December, 2018] pp.10-16]
International Journal of Education, Psychology and Counseling
eISSN: 0128-164X
Journal website: www.ijepc.com

SMALL-SCALE SYNTHESIS OF LEAD IODIDE FOR CHEMISTRY TEACHING

Naruwan Pattarapongdilok[1]
Atthachai Siriwatthanasakdina[2]
Sakulrat Kamwongpool[2]
Benyarat Parephan[2]
Juntimakorn Kamon[2]
Jirapast Sichaem[3]

[1]Chemistry Program, Department of Science, Faculty of Science and Technology, Bansomdejchaopraya Rajabhat University, Bangkok 10600, Thailand (olinpoo@gmail.com)
[2]Science Program, Faculty of Education, Bansomdejchaopraya Rajabhat University, Bangkok 10600, Thailand
[3]Faculty of Science and Technology, Thammasat University Lampang Center, Lampang 52190, Thailand

***Accepted date**: 01-09-2018*
***Published date**: 15-12-2018*

To cite this document: Pattarapongdilok, N., Siriwatthanasakdina, A., Kamwongpool, S., Parephan, B., Kamon, J., & Sichaem, J. (2018). Small-scale Synthesis of Lead Iodide for Chemistry Teaching. *International Journal of Education, Psychology and Counseling*, 3 (23), 10-16.

Abstract: *Lead iodide synthesis is a popular chemistry experiment that is used to introduce ionic bonding and be interesting for young students with beautiful golden-yellow crystals. This research aims to develop the method of lead iodide synthesis in small-scale and survey students' feedbacks in their skills and experimental processes, understanding, and attitudes. We found that the forming of beautifully golden-yellow lead iodide crystals, golden rain, was prepared by mixing 5.00 ml of 0.005 M lead nitrate and 5.00 ml of 0.010 M potassium iodide that was heated for five minutes, cooled down and added with 1.0 M hydrochloric acid and five drops of 0.2 M lead nitrate. This synthesis was easily done by using a small-scale of chemicals produced rapidly hexagonal and triangular crystals for a minute, which were monitored by a scanning electron microscope (SEM). From students' feedbacks, the benefits of this method help students to improve their skills and experimental processes, attitudes, and understanding of chemistry content, respectively.*

Keywords: *Lead Iodide, Synthesis, Experiment, Chemistry Teaching*

Introduction

Nowadays, various techniques are used to teach in chemistry class for the increment of learners' understanding. Teaching techniques help to enhance learners' imagination and skills. One of good teaching techniques is the demonstration that affects students' interest in the content. The interesting chemical demonstration is the reaction producing lead iodide (PbI_2) which crystals of PbI_2 are beautiful golden yellow. Especially, The PbI_2 that is synthesized by reaction of lead nitrate (Pb $(NO_3)_2$) solution with potassium iodide (KI) solution is a simple and suitable approach for using practical chemistry teaching. This experiment introduces ionic bonding and is so interesting for young students with golden-yellow crystals (Günter & Alpat, 2017; Fleming, 2016; de Arellano & Towns, 2014; Taber, 2012; Baek & Jeong, 2006; Kind, 2004). Previously, lead iodide has been interested since 1970s as use of a high-efficiency detector in room temperature. It is applied in ecology, medicine, astrophysics, non-destructive defectoscopy, and X-ray and γ-ray spectroscopy (Matuchova, 2009; McGregor, 1997; Shoji, 1996; Shah, 1994; Zhang, 1992; Lund, 1989; Dugan, 1968). Crystals of PbI_2 consist of a repeating unit of a hexagonally closed-packed layer of lead ions sandwiched between two layers of iodide ions (Ahuja, 2002; Sengupta, 1999; Makino, 1998; Watanabe, 1994; Dorner, 1976). It can be grown from solutions, melts, vapors, and gels (Chand & Trigunayat, 1977) One of famous synthesis method, Fleming (2016) demonstrated the synthesis of PbI_2, golden rain, which was used 3 grams of KI and $Pb(NO_3)_2$ in 200 cm^3 deionized water and a few drops of 1.0 M HCl. When chemicals were mixed, it was heated and waited for crystal forming. Although this method easily observed the beautiful crystals of PbI_2, it uses large-scale chemicals and long-time for preparing. From this methodology, we interest to develop the synthesis of PbI_2 that can reduce these problems and complicatedness for demonstrating in chemistry class. Surely, as beautifully appearing crystals of PbI_2, it can induce the students' interest in chemistry content.

Herein, the aim of this work was to synthesize PbI_2 in a small-scale for students to study chemistry, which is cost and time saving and improves laboratory safety. In addition, we surveyed students' feedbacks in their skills and experimental processes, understanding, and attitudes.

Method

For synthesis of lead iodide, we used a 15-ml vial and 5.00 ml of 0.005 M Pb $(NO_3)_2$ and 5.00 ml of 0.010 M KI were mixed. The mixed solution was then heated in a water bath at 95 ℃ for five minutes (Figure 1), cooled down to room temperature naturally, and was added with five drops of 0.2 M $Pb(NO_3)_2$ with shaking for mixing. After mixed solution had rested for crystallizing at room temperature, the golden-yellow product, the crystals of PbI_2, was formed. For optimizing the condition for synthesis, mixed solution had varied rest times of mixed solution before dropping Pb $(NO_3)_2$. Optimized condition vial was cleaned many times with cool deionized water, filtered in vacuum, and dried at 40 ℃ overnight, and its shape was analyzed by a Scanning Electron Microscope (SEM). Previously, PbI_2 was compared with PbI_2 from a vial that was added with one drop of 1.0 M hydrochloric acid and five drops of 0.2 M $Pb(NO_3)_2$ in the final step. This method was used as an experimental set for twenty-one freshmen studying General Chemistry that would be taught on chemistry experimental lab at Bansomdejchaopraya Rajabhat University. Three students per group were used in this study. After having finished the experiment, students answered their questionnaires. The questionnaires contained three aspects: (1) skills and experimental processes, (2) students' understanding from experiment, and (3) students' attitudes.


(a) **(b)**

Figure 1: (a) Vial of Heated Solution and (b) Vial of Solution with Formed Lead Iodide Crystals

Results and Discussion

Lead Iodide Synthesis

From reaction between 5.00 ml of 0.005 M Pb $(NO_3)_2$ and 5.00 ml of 0.010 M KI, we did not find the PbI_2 forming but it was heated and cooled down at room temperature. The mixture was ready to form the PbI_2 crystals. When five drops of 0.2 M Pb $(NO_3)_2$ were added in the mixture, the reactants of reaction were increased which reaction equilibrium was disturbed. The reaction was moved forward following Eq.1 and PbI_2 crystals were occurred rapidly.

$$Pb(NO_3)_2(aq) + 2KI(aq) \rightleftharpoons PbI_2(s) + 2KNO_3(aq) \quad (1)$$

According to Table 1, the best rest time of this small-scale synthesis of lead iodide before adding five drops of 0.2 M $Pb(NO_3)_2$ was 20-30 minutes, which formed PbI_2 crystals of the largest size and quantity.

However, if carbon dioxide dissolves in solution, it will generate carbonate anion in solution. The crystals of lead carbonate ($Pb(CO_3)_2$) in the reaction following Eq.2. The reaction equilibrium was reversed to be lead cation and carbonate anion when added HCl as Eq.3 because $Pb(CO_3)_2$ can dissolve in acidic solution.

$$Pb^{2+}(aq) + CO_3^{2-}(aq) \rightleftharpoons Pb(CO_3)_2(s) \quad (2)$$

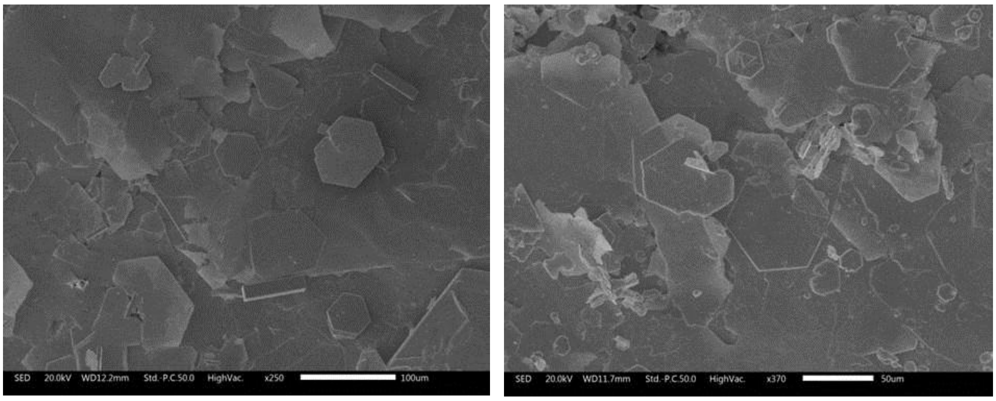
$$Pb(CO_3)_2(s) \overset{HCl}{\rightleftharpoons} Pb^{2+}(aq) + CO_3^{2-}(aq) \quad (3)$$

As there is adding of HCl, the occurred crystals of PbI_2 were pure. When the PbI_2 crystals were analyzed by SEM, they formed as hexagonal crystals (shown in Figure 2(a)) and had tetragonal crystals with impurity. When the vial was added with one drop of 1.0 M hydrochloric acid, PbI_2 crystals were hexagonal and triangular (shown in Figure 2(b)) which it could generate several shapes (Liu, 2015; Bhavsar, 2002). Therefore, the method of adding one drop of 1.0 M hydrochloric acid was better than non-adding with lower impurity, but other results were same. This condition used an experimental set (Figure 3) for chemistry teaching in which PbI_2 crystals were golden yellow crystals with a weight of approximately 2.2 to 2.5 milligrams as shown in Figure 1(b). Because of very lightweight, finding the weight of crystals was calculated from the average weight of eight vials which have three trials.

Thus, the best method is the reaction between 5.00 ml of 0.005 M $Pb(NO_3)_2$ and 5.00 ml of 0.010 M KI adding 1.0 M hydrochloric acid.

Table 1: Times of Adding Five Drops Of 0.2 M Pb $(NO_3)_2$

Time	Crystals	Quantity
10 minutes	Very small crystals in yellow solution	Few
20 minutes	Medium crystals in colorless solution	Much
30 minutes	Small crystals in colorless solution	Much
40 minutes	Very small crystals in colorless solution	Medium
50 minutes	Very small crystals in colorless solution	Few
1-19 hours	Very small crystals in colorless solution	Fewer than 50 minutes
20-23 hours	Very small crystals in colorless solution	Very few
1-15 days	Very small crystals in colorless solution	Very few



(a) **(b)**

Figure 2: SEM Images of PbI_2 Crystals From: (a) Non-Added HCl and (b) Added HCl

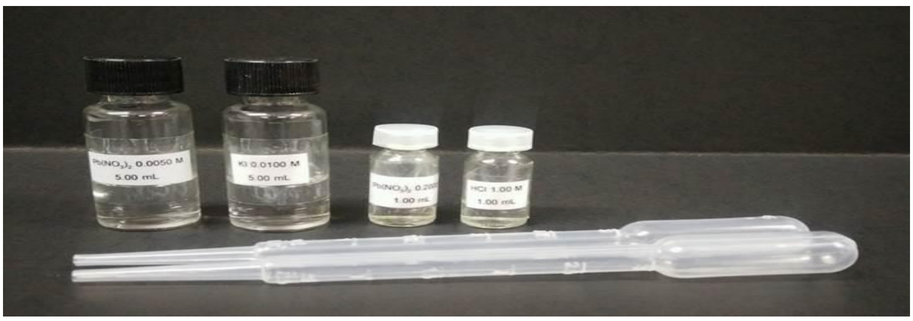


Figure 3: Experimental Set of Lead Iodide Synthesis

Study of Students' Feedbacks

The students' attitude survey form was a rating scale with 5 levels including: 1 is strongly disagree, 2 is disagree, 3 is neither agree nor disagree, 4 is agree, and 5 is strongly agree. After this experiment was used in the chemistry teaching, students' feedbacks in their skills and experimental processes, attitudes, and understanding obtained average values as 4.19, 4.07, and 3.93, respectively (see Figure 4), as follows:

Skills and experimental processes consist of four aspects: (1) easy, convenient, and fast activity as 4.29, (2) safe activity as 4.29, (3) easily observed activity as 4.29, and (4) activity supporting scientific process skills as 3.90.

Students' attitudes towards learning chemistry when they conduct chemistry experiment in laboratory are as follows: (1) benefit of experiment as a teaching tool as 4.19, (2) increasing environmental awareness about green chemistry as 4.14, (3) improving scientific attitudes as 4.05, and (4) increasing attention in chemistry content as 3.90. Six scientific students' attitudes are as follows: (1) rationality as 4.14, (2) curiosity as 4.10, (3) honesty as 4.05, (4) perseverance as 4.00, (5) suspended–judgment as 4.00, and (6) open-mindedness as 3.90.

This experiment made students understand four topics: (1) formation of ionic compound as 4.05, (2) chemical equilibrium and dissolution of solid in liquid as 3.95, (3) stoichiometry as 3.86, and (4) rate of reactions as 3.86.

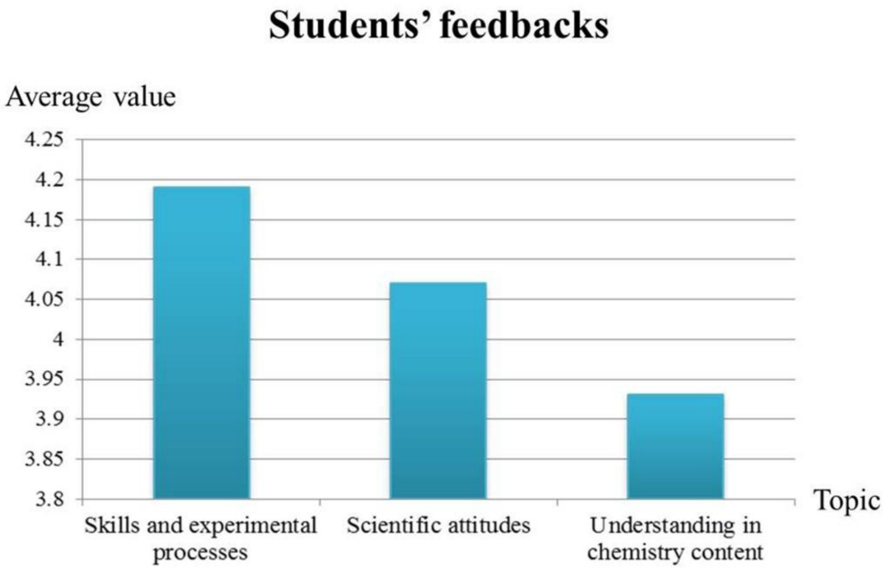


Figure 4: Students' Feedbacks Graph

Conclusion

This research was involved with a small-scale synthesis of lead iodide that was synthesized by mixing 5.00 ml of 0.005 M lead nitrate and 5.00 ml of 0.010 M potassium iodide in a 15-ml vial. A small-scale of chemicals was used for synthesis that was easily approximately. Getting lead iodide crystals as a result shown in chemistry class for a short time was easily observed as well. After having been heated in water bath at 95°C for five minutes and cooled down to room temperature, the mixed solution was added with one drop of 1.0 M hydrochloric acid and five drops of 0.2 M lead nitrate. Crystals of lead iodide with a weight of approximately 2.4 to 2.6 milligrams falling for a few minute were golden-yellow. The PbI_2 crystals were hexagonal and triangular ones analyzed by Scanning Electron Microscope (SEM). This method was used for twenty-one freshmen studying General Chemistry that would be taught on chemistry experiment. The experiment can help students to improve their skills and experimental processes, attitudes, and understanding in chemistry content.

Acknowledgments

We thank the students of faculty of science and technology for being cases study. Moreover, we appreciate the faculty of Education at Bansomdej-chaopraya Rajabhat University for supporting instruments and laboratory. Assoc. Prof. Poramest Boonsri, Mr. Gilbert E. Mowmessie, and Dr. Kasame Tritrakan for proving the manuscript.

References

Ahuja, R., Arwin, H., Ferreira da Silva, A., Persson, C., Osorio-Guillén, J. M., Souza de Almeida, J., Moyses Araujo, C., Veje, E., Veissid, N., An, C.Y., & Pepe, I. (2002). Electronic and optical properties of lead iodide. *Journal of Applied Physics*, *92*(12), 7219-7224.

Baek, S. H., & Jeong, S. J. (2006). A research of the Difference in Teaching Styles and Understanding of 9th Grade Students About Lead-iodide Precipitation Reaction Experiment. *Journal of the Korean Chemical Society*, *50*(5), 374-384.

Bhavsar, D. S., Saraf, K. B., & Seth, T. (2002). Studies on growth and microstructure of lead iodide single crystals. *Crystal Research and Technology: Journal of Experimental and Industrial Crystallography*, *37*(2-3), 225-230.

Chand, M., & Trigunayat, G. C. (1977). Effect of impurities on solid state structure transformations in gel-grown PbI2 crystals. *Journal of Crystal Growth*, *39*(2), 299-304.

Cruz-Ramírez de Arellano, D., & Towns, M. H. (2014). Students' understanding of alkyl halide reactions in undergraduate organic chemistry. *Chemistry Education Research and Practice*, *15*(4), 501-515.

Dorner, B., Ghosh, R. E., & Harbeke, G. (1976). Phonon dispersion in the layered compound PbI2. *physica status solidi (b)*, *73*(2), 655-659.

Dugan, A. E., & Henisch, H. K. (1968). Defect Energy-Level Structure of P bI 2 Single Crystals. *Physical Review*, *171*(3), 1047.

Fleming, D. Exhibition Chemistry: Golden Rain, Royal Society of Chemistry, https://eic.rsc.org/exhibition-chemistry/golden-rain/2000048.article. Accessed 20 September 2016.

Günter, T., & Alpat, S. K. (2017). The effects of problem-based learning (PBL) on the academic achievement of students studying ‘Electrochemistry’. *Chemistry Education Research and Practice*, *18*(1), 78-98.

Kind, V. (2004). Beyond appearances: Students’ misconceptions about basic chemical ideas. *School of Education, Durham University, UK.*

Liu, X., Ha, S. T., Zhang, Q., de la Mata, M., Magen, C., Arbiol, J., Sum, T. C., & Xiong, Q. (2015). Whispering gallery mode lasing from hexagonal shaped layered lead iodide crystals. *ACS nano*, *9*(1), 687-695.

Lund, J. C., Shah, K. S., Squillante, M. R., Moy, L. P., Sinclair, F., & Entine, G. (1989). Properties of lead iodide semiconductor radiation detectors. *Nuclear Instruments and Methods in Physics Research Section A: Accelerators, Spectrometers, Detectors and Associated Equipment*, *283*(2), 299-302.

Makino, T., Watanabe, M., Hayashi, T., & Ashida, M. (1998). Time-resolved luminescence of exciton polaritons in PbI 2. *Physical Review B*, *57*(7), 3714.

Matuchova, M., Zdansky, K., Zavadil, J., Danilewsky, A., Maixner, J., & Alexiev, D. (2009). Electrical, optical and structural properties of lead iodide. *Journal of Materials Science: Materials in Electronics*, *20*(3), 289-294.

McGregor, D. S., & Hermon, H. (1997). Room-temperature compound semiconductor radiation detectors. *Nuclear Instruments and Methods in Physics Research Section A: Accelerators, Spectrometers, Detectors and Associated Equipment*, *395*(1), 101-124.

Sengupta, A., Jiang, B., Mandal, K. C., & Zhang, J. Z. (1999). Ultrafast electronic relaxation dynamics in PbI2 semiconductor colloidal nanoparticles: A femtosecond transient absorption study. *The Journal of Physical Chemistry B*, *103*(16), 3128-3137.

Shah, K. S., Lund, J. C., Olschner, F., Bennett, P., Zhang, J., Moy, L. P., & Squillante, M. R. (1994). Electronic noise in lead iodide X-ray detectors. *Nuclear Instruments and Methods in Physics Research Section A: Accelerators, Spectrometers, Detectors and Associated Equipment*, *353*(1-3), 85-88.

Shoji, T., Sakamoto, K., Ohba, K., Suehiro, T., & Hiratate, Y. (1996, November). Characterization of the PbI/sub 2/crystal as a material for radiation detectors. In *Nuclear Science Symposium, 1996. Conference Record., 1996 IEEE* (Vol. 1, pp. 25-28). IEEE.

Taber, K. (2012).Teaching Secondary Chemistry, 2nd edn. (Hodder Education), 121.

Watanabe, M., & Hayashi, T. (1994). Polariton relaxation and bound exciton formation in PbI2 studied by excitation spectra. *Journal of the Physical Society of Japan*, *63*(2), 785-794.

Zhang, J., Shah, K. S., Olschner, F., Lund, J. C., Moy, L. P., Daley, K., Cirignano, L., & Squillante, M. R. (1992). An improvement in growing large, oriented lead iodide single crystals for detector applications. *Nuclear Instruments and Methods in Physics Research Section A: Accelerators, Spectrometers, Detectors and Associated Equipment*, *322*(3), 499-503.