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Development and Use of a Small-scale Experimental Kit for Observation of Recrystallization of Ammonium Chloride in Science Lessons and Workshops

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Abstract

We developed a small-scale experimental kit that can be used by young scientists to individually conduct experiments on the recrystallization of NH_4Cl. We used this experimental kit as a teaching material in a science class, a science workshop, and a research project. On the basis of responses from students and teachers who participated in these learning activities, we evaluated its usability as an aid to help in the learning process. The analysis results revealed that this experimental kit was easy and safe to operate, and it allowed learners to clearly observe the phenomenon of recrystallization and sense the heat of reaction. The use of this teaching material in the science workshop resulted in an increased interest in the phenomenon of recrystallization. Moreover, high school students were able to enhance their scientific thinking and expression through the research conducted with the teaching material. This teaching material can be useful for various learning activities.

1. Introduction

Recently, illustrative micro-scale experimental kits have been used as teaching materials so that students can individually perform experiments while generating less chemical waste [1]. Handy experimental kits comprised of inexpensive and widely used materials can be readily used by learners for conducting individual experiments in various educational settings. For teaching students about the properties of solutions, teachers often introduce several aqueous solutions. In addition to NaCl and urea, ammonium chloride and its aqueous solutions have been used to demonstrate recrystallization. Typically, these recrystallization experiments are performed in a test tube or beaker by the teacher; students are rarely allowed to perform this experiment individually. Furthermore, because solubilizing NH_4Cl is a moderately endothermic process, NH_4Cl has not been the primary choice while teaching solubility and the heat of reaction. Similar to NaCl, NH_4Cl is widely used in everyday life; therefore, NH_4Cl, a material familiar to students, can be a useful teaching material.

Even when a teaching material is used to demonstrate a well-known phenomenon, its educational effect on learners can increase when it is available for various educational situations. Even newly appointed teachers can assemble various solutions simply by changing solutes in a test tube. Moreover, small-scale experiments allow students to perform individual experiments effectively. Collectively, these facts improve the usability of the kit for both students and teachers. With these motivations, we developed a small-scale experimental kit that allows individual students to easily perform the recrystallization of NH_4Cl in various learning activities. In fact, we used this teaching material in a science class, a science workshop, and a research project [2]. In this paper, we report the results of these activities and discuss the usefulness of the teaching material in the context of chemical education.

2. Development of Teaching Material

2.1. Properties of NH_4Cl and its Aqueous Solution

Recrystallization is conveyed as a change in solubility with temperature. The change in solubility of NH_4Cl with temperature is not as remarkable as that of KNO_3 but similar to that of $CuSO_4$ [3]. Thus, a supersaturated solution of NH_4Cl has a wide temperature range where recrystallization proceeds gradually. This allows us to observe the process of crystal growth when the supersaturated NH_4Cl solution is slowly cooled by air. Furthermore, NH_4Cl crystal is more readily obtained on cooling than NaCl crystal. Because NH_4Cl shows dendritic crystalline forms, the interest of students in the process of crystal growth is kindled further. $AgNO_3$ and $CuCl_2$ are widely used to display the formation of the "chemical garden," i.e., dendritic crystal growth; however, the safe disposal of these chemicals involves complicated protocols. These factors indicate that an aqueous solution of NH_4Cl is a suitable reagent for observing recrystallization.

A characteristic of NH_4Cl is its negative heat of dissolution. The standard enthalpy of dissolution for NH_4Cl is 14.8 kJ/mol [3]. This value is marginally lower than those of KNO_3 (34.9 kJ/mol) and NH_4NO_3 (25.7 kJ/mol) and similar to that of urea (15.4 kJ/mol), which is used as a coolant gel. Even when a small amount of NH_4Cl is dissolved in a little water in a test tube, the decrease in the temperature of the liquid can be sensed by the skin of the palms. Thus, NH_4Cl can substitute for KNO_3 or NH_4NO_3, chemicals that have been used frequently to demonstrate endothermic reactions.

2.2. Experimental Methods

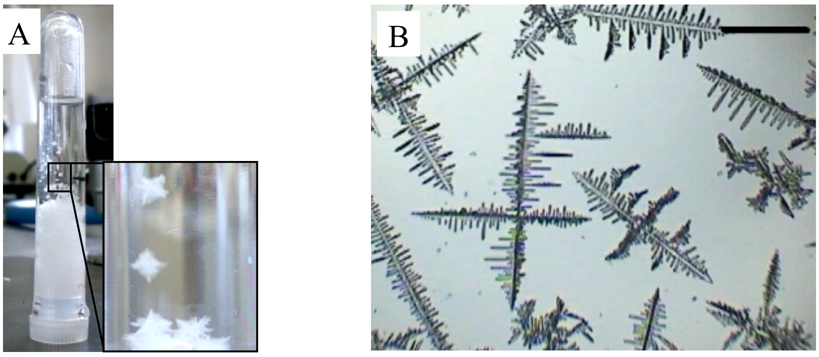

Figure 1. Macroscopic (A) and microscopic (B) images of NH_4Cl crystals obtained using the experimental kit. Scale bar in panel B represent a length of 0.5 mm.

A typical experimental protocol for the recrystallization of NH_4Cl has been introduced in an experimental guide book [4] and other associated teaching materials for setting up a demonstration are commercially available. Recently, Terashima *et al.* [5] reported that a clear dendritic crystal growth can be observed at room temperature by preparing a 30–35 wt% aqueous solution of NH_4Cl; this corresponds to a supersaturated concentration range of 5%–20%. In this study, we prepared an aqueous solution containing 33 wt% NH_4Cl.

We used a small plastic test tube (AS-ONE, SS-14, 5 mL, 14.5 mm-caliber, 83.1 mm long, polystyrene) as a container for the solutions. Commercially procured NH_4Cl (1.5 g; Yoneyama Yakuhin Kogyo Co. Ltd., purity >98.5%) and distilled water (3 mL) were mixed in a test tube. A cap was attached to the test tube and then the test tube was shaken to ensure that some amount of NH_4Cl was dissolved in water, which is accompanied by an absorption of heat. Subsequently, the mixture was heated for 3 minutes in a water bath at 85 °C. After the NH_4Cl was completely dissolved, the solution was cooled in ice water until it was safe to touch the tube with the hands. The test tube was then allowed to stand on its cap, i.e., upside down, at room temperature, as shown in Figure 1(A).

After a few minutes, small white flakes of NH_4Cl (up to 1 mm) began to form. The gradual recrystallization was observed for about 10 minutes with the naked eye, as shown in Figure 1(A). Next, we placed a drop of the solution on a glass slide and covered the drop with a cover glass. We observed the recrystallization at 40x magnification through an optical microscope. The growth of dendritic crystals, as shown in Figure 1(B), started immediately and was completed within 2-3 minutes. The process of recrystallization could be repeated by reheating and recooling the NH_4Cl solution.

3. Practical Reports

3.1. Science Class in High School

To evaluate the usability of these experimental kits, in 2010, we designed a learning

Table 1. A teaching plan for a science class with individual experiments on recrystallization of NH_4Cl.

Learning Activity	Related Learning Contents	Time (min)
Review of Last Lesson	Structure of Material	5
Guidance & Preparation for Experiment Preparation of Aqueous Solution Observation of Endothermic Dissolution Heating & Cooling Solution Observation of Recrystallization Microscopic Observation of Crystal Growth Cleaning Experimental Kits Summarizing Results	Change of Material Dissolution Heat of Reaction Recrystallization	35
Knowing Practical Applications of NH_4Cl	Use of Material	10

activity for high school students called "Integrated Science A." This science class, introducing the concepts of recrystallization, was scheduled after "Materials and Human Life," a class in which students learned about 1) chemical bonds between atoms or ions, 2) changes in particle states through physical and chemical reactions in relation to a change in energy, and 3) the physical properties of materials relevant to human life and their practical applications. Our objective was to help students, at the individual level, understand the structure of ionic compounds, changes in states and energy through dissolution and recrystallization, and the practical application of these concepts. Table 1 shows the outline of this class.

Owing to constraints in time and equipment, the teacher prepared a small parcel of NH_4Cl for each student prior to the class. At the beginning of the class, the teacher sequentially reviewed the concept of ionic compounds, introduced NH_4Cl as one example of an ionic compound, and mentioned the purpose of the study. The teacher distributed the individual experimental kits to students and demonstrated its practical use. Each student started to prepare aqueous NH_4Cl solution and experienced its endothermic dissolution by feeling a rapid decrease in the temperature of the solution. At this stage, the teacher explained to the students that the breakdown of solid NH_4Cl into NH_4^+ and Cl^- during dissolution involved a change in energy from lower to higher levels, which appeared as the endothermic reaction. While heating the solution to dissolve NH_4Cl, the teacher elucidated the mechanism of recrystallization in terms of the temperature dependence of solubility.

After the NH_4Cl was completely dissolved, each student observed the process of recrystallization with the naked eye and through the microscope as the solution cooled. The teacher explained that recrystallization occurred as a result of the rearrangement of dissolved ions and that this process was used for the separation and purification of mixtures. After all the students completed their observation of the recrystallization process, the teacher displayed typical products that contained NH_4Cl, such as food additives, chemical fertilizers, and manganese batteries.

Table 2. Responses from science teachers after using the experimental kit.

Usability Attention when using the kit Remedy	● The growth of impressive crystals, which appear very different from initial granular ones, are clear to observe. ● NH_4Cl is a nontoxic material, therefore I can use it safely. ● When I told students that NH_4Cl is used as a food additive, they were very comfortable handling it. ● There is a moderate risk of the test tube cap coming off during heating. ● Screw-type caps can be relatively safer for use. ● To observe crystal growth clearly, we had to cool the solution very slowly, a process that was time consuming.
Utilization examples	● As an example of exo and endothermic dissolution of chemicals such as NaOH, $CaCl_2$, and urea. ● As an example of salts such as NaCl, CH_3COONa, and Na_2CO_3. ● As an example of food additives such as in yeast foods. ● As a research theme for students' extracurricular inquiry activities.
Students' responses	● Students were strongly impressed by the beautiful dendritic crystals. ● Students asked me the reason behind the change in crystal forms, indicating their interest in the microscopic mechanism of recrystallization. ● Students asked me why the temperature decreased during dissolution and whether this happens with other substances, indicating their interest in the mechanism of endothermic reactions. ● A few students who assisted teachers in the science festival were able to clearly explain the mechanism of endothermic reaction and crystal growth to participants of the event.

At the end of the class, several students wanted to take the used kits home for further experimentation. The teacher gave them the kits, advising them to pay attention while using the kit. Since crystallization is such a fundamental phenomenon of everyday life that every student needs to understand, the teacher performs this experiment in science class every year.

Using a descriptive questionnaire, we asked the teachers who taught the science class and conducted the workshop to provide feedback on the usability of the kit and teaching material. The results are shown in Table 2.

3.2. Science Workshop for Children

This experiment on the recrystallization of NH_4Cl, with the associated teaching material, has also been introduced in a science workshop for children, "Science Festival in Tokushima," sponsored by Tokushima University, conducted every summer. The teacher and some interested high school students set up a booth for two days and evaluated the interest of young scientists in the experiment. About 500 participants (predominantly, elementary and kindergarten students) visit the booth every year. These preteen visitors performed all the steps of the experiment with assistance from the students at the booth. Some participants recorded the crystal growth by taking a photo through a microscope using a mobile camera. Figure 2 shows a scene at the workshop and a typical microscopic image of NH_4Cl crystal.

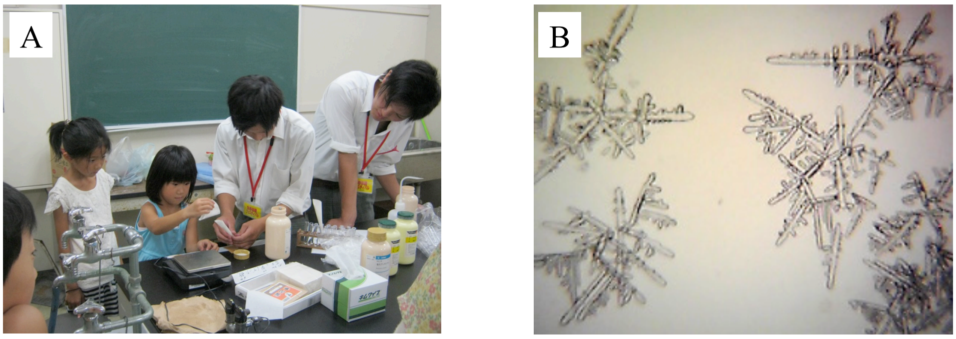

Figure 2. Recrystallization being performed by a young researcher at a Science Festival in Tokushima (A) and a microscopic image taken by a participant using a mobile camera (B).

Table 3. Results of the questionnaire for participants in Science Festival in Tokushima (n = 66).

Questions	Number	%
Have you ever done this experiment?		
Yes	6	9%
No	60	91%
Difficulty in this experiment		
Very easy	38	58%
Slightly easy	6	9%
Neither	19	29%
Slightly difficult	2	3%
Very difficult	1	1%
Satisfaction at this experiment		
Very satisfied	49	74%
Slightly satisfied	12	18%
Neither	1	1%
Slightly dissatisfied	4	6%
Very dissatisfied	0	0%
Do you want to try again?		
Yes	63	95%
No	3	5%

Classification	Number of descriptions
Impression upon recrystallization and crystal growth	24
Satisfaction at the first experience in observing NH_4Cl	16
Usability of the experimental kit	10
Satisfaction at assistance and explanation by student staffs	7
Others	6
No answer	22

Both children and their parents seemed to be strongly interested in the recrystallization process and observed “crystal flakes that fall down like white snow.” Most participants asked the students volunteering at the booth if the experiment can be done again at home. The participants were given the kits and advised to use them with care.

We sought feedback from participants by asking them to fill out a questionnaire about the experiment. After conducting the experiment, the participants were asked to describe their opinions by answering multiple choice questions about 1) experience, 2) difficulty, 3) satisfaction, and 4) intention of repeating the experiment. Table 3 shows the results from this survey.

We categorized the descriptive opinions of the participants, as shown in Table 4. There were 24 descriptors for evaluating the experience of the participant after conducting the experiment, including "I observed the wonderful shape of crystals for the first time" and "I felt a sense of wonder at the process of recrystallization, which is beautiful." This survey included 16 observation-based statements such as "I observed crystal growth through a microscope" and "I'm satisfied with the first experience of making crystals by myself." In addition, there were 10 opinion-based statements to evaluate the usability of the teaching material, such as "I can do the experiment easily and quickly with the handy kit" and "I'll do the experiment again at home because the kit is easily portable." Some participants were pleased with the level of assistance provided by staff during the execution of these activities. From these responses, it was evident that most participants considered this experiment to be easy to perform individually and were satisfied with the experience of clearly observing the growth of impressive crystals.

Furthermore, we gathered feedback on the experience of the nine volunteering high school students who assisted in the booth through a descriptive questionnaire seeking opinions on the knowledge and experience gained after the workshop. Consequently, all students except one were found to have gained an understanding of the endothermic dissolution of NH_4Cl for the first time. One student reflected, "I was able to understand the mechanism of the crystallization gradually by repeating this experiment, so I was able to answer the questions from participants properly." Another student pointed out that both the time and cooling temperature are critical factors that control the recrystallization process.

3.3. Extracurricular Inquiry Activity

Some students who attended the science class and the workshop expressed a strong desire to learn more about the process of dendritic crystal growth, which can be controlled by the temperature and the concentration of the solute. These students took part in an extracurricular inquiry activity entitled "Research on Changes in States of Matter" (AG102089) [6] in the Science Partnership Program (SPP) supported by the Japan Science and Technology Agency. The activities were performed through collaboration between

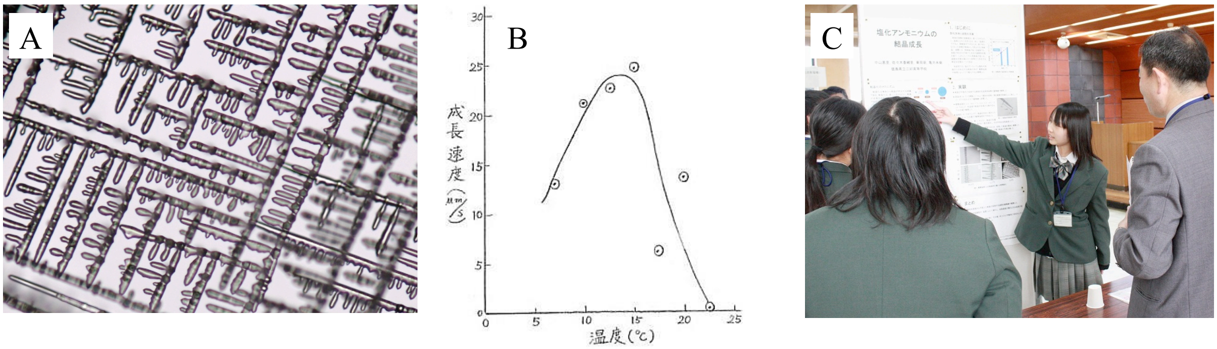

Figure 3. Results from the extracurricular inquiry activity (SPP). A. a microscopic image of dendritic crystals of NH_4Cl at 5 °C; B. temperature dependence of the velocity of crystal growth; C. a student presenting a poster at the 21st Symposium on Physics Education.

Tokushima Prefectural Miyoshi High School and Naruto University of Education. The students studied changes in crystal form and the velocity of crystal growth using an optical microscope equipped with a temperature control unit under the guidance of university professors. Figure 3 shows some results recorded from this activity. The students found that the growth of different crystal forms was dependent on the temperature of observation. Moreover, they found that the velocity of crystal growth at the rate of length per second (μm/s) is dependent on the temperature. These students presented the results at "The 21st Symposium on Physics Education" organized by the Japan Society of Applied Physics in 2011 [7]. There was a lively discussion of the study between the presenters and the audience. Furthermore, the students summarized their findings in a paper, submitted it to a high school science contest, and won a prize for their seminal research.

4. Evaluation and Discussion

Teaching materials that clearly display principles related to a phenomenon are useful to both learners and teachers. We have evaluated the usability of a small-scale experimental kit for the demonstration of crystallization by conducting successful experiments and through feedback from learners and teachers who participated in these studies.

Even when we use the same teaching material, its effect on the learning experience of the students can vary depending on the different educational settings. We have obtained feedback from participants in the science workshop to estimate the effect of this teaching material on elementary and kindergarten children. We have also evaluated the effect of this educational kit on insights gained by high school students who assisted in the science workshop or participants in the extracurricular inquiry activity.

4.1. Usability

Using this small-scale experimental kit, the teacher was able to teach high school students the principles related to constitution, a change in state, and applications of NH_4Cl through individual experimentation in one class (50 minute duration). Since the process of crystal growth depends on the extent of supersaturation, the teachers needed to measure the appropriate quantities of NH_4Cl (depending on the temperature) before initiating a class. Such a practice can save time. While the growth of crystals in our demonstrations was observed through a microscope with 40x magnification, crystal growth can also be clearly observed at lower magnifications. After use, the NH_4Cl solution can be disposed of in water because of its low toxicity. Since the test tube is made of polystyrene, it is unbreakable and safe for children to use. Furthermore, since this test tube can stand erect and independently on its cap, the crystallization process can be clearly observed even in the absence of test tube stands. However, note that when the solution is heated >90 °C, the cap on the test tube can sometimes be ejected suddenly because of pressure from steam. Therefore, the temperature of the solution should be monitored carefully by both the teacher and students.

The capacity of this test tube is nearly 5-fold lower than that of the standard glass test tubes (up to 27 mL); requiring lower amounts of reagents and solution and consequently generating less waste. The unit cost of the kit, including the test tube, chemicals, and the wrapping paper is so low that a teacher can prepare this experimental kit for an individual student for less than 30 JPY.

The feedback from the participants in the science workshop (Table 3) indicated that this teaching material is easy to operate and safe for use by children. While >90% of children performed the experiment for the first time, up to 67% considered the experiment to be easy (this includes the choices "very" and "slightly" easy), and up to 92% were satisfied (this includes the choice "very" and "slightly" satisfied) with the experiment.

4.2. Educational Effect on Children

In the Japanese curriculum, the basic concept of recrystallization is introduced to fifth grade students under elementary science. Pupils often learn that the solubility of a solute depends on the temperature of the solution and that there is an upper limit for the amount of substance such as sodium chloride or boric acid that can be dissolved in water. A pedagogical experiment to evaluate the enhancement of interest in the recrystallization of NH_4Cl was conducted with fifth grade students [8]. We found that children who participated in the science event are strongly interested in recrystallization after observing/performing the experiment. By employing this experimental kit, even students under the fifth grade were able to perform the experiment on the recrystallization of NH_4Cl with support from the staff. The enrichment of real experience of natural phenomena in daily life and playing is emphasized for preschool education in Japan [9]. After the workshop, several children expressed the desire to repeat the experiment. This handy teaching material can be useful for children who observe recrystallization at home and enrich their experience of natural phenomena.

4.3. Educational Effect on High School Students

As listed in Table 2, the benefits for students participating in the extracurricular inquiry activity are significant. Although there is room for improvement in the safety and clarity of observation, this portable experimental kit is suitable for clearly demonstrating dissolution and recrystallization and for exemplifying practical applications of ionic compounds in daily life. The teacher introduced NH_4Cl as an example of endothermic dissolution, salts, and food additives. For high school science education, the emphasis was on understanding the relationship between science and human life, as indicated by the introduction of “Science and Everyday life” as a new course in the curriculum [10]. Moreover, “Basic Chemistry” has a section on chemistry in daily life. In the context of these developments, this experimental kit can be a useful heuristic tool to help students develop an understanding of the practical applications of chemical substances in daily life.

The questions typically asked by high school students after performing these experiments include “Why do the crystals change their form on recrystallization?” and “Why does the temperature decrease on dissolution?” These questions evinced their interest in the microscopic mechanism of crystal growth and endothermic dissolution of NH_4Cl in addition to their interest in macroscopic recrystallization. The teacher explained plainly to the former question that the crystals of NH_4Cl have equilibrium cubic and non-equilibrium dendrite forms, and the non-equilibrium crystals observed in the early stage of recrystallization are gradually transformed into equilibrium ones. The teacher responded to the latter question that the dissolution of NH_4Cl is a change in energy from lower to higher levels, and such reactions involve a decrease in temperature by heat absorption. This teaching material can also enhance the interest of students in recrystallization from the perspectives of energy and particles.

To enhance scientific aptitude and expression in students, activities that nurture an inquisitive nature in high school students are required. It has been reported that a micro-scale experiment, highly efficient in terms of time, allowed students to adequately discuss and express the experimental result. Such experiments are expected to be effective in enhancing scientific thinking in students. This small-scale experimental kit was also used by individual students in the science class. In the science workshop or SPP, the high school students were able to explain the mechanism of recrystallization to children and parents in simple terms or present and discuss the results of their research positively with the audience. These activities can prove to be good opportunities for the high school students to enhance not only their interest in dissolution and recrystallization, but also their scientific thinking and expression. We expect this experimental kit to be useful for enhancing learners’ scientific thinking by conducting experimental activities.

5. Conclusions

We developed a small-scale experimental kit for use by individual students to observe the recrystallization of NH_4Cl and its endothermic dissolution easily. Using the handy teaching material, we were able to establish a heuristic pedagogical approach to communicate the principles of recrystallization, and then we evaluated its usability by obtaining feedback from students and teachers. According to the analysis of the feedback, we concluded that this experimental kit made it easy for students to learn about recrystallization and the heat of dissolution through individual experimentation, and it could be used under various educational settings. Moreover, this experiment was an effective tool for high school students to enhance their interest in scientific thinking and expression. Our future efforts will be directed toward predicting the usability of this teaching material more specifically and quantitatively. The handy experimental kit is expected to be useful for other experiments simply by changing the substances. We will explore other concepts and educational practices to identify multiple uses of this teaching material.

Acknowledgments

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