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The Development of Microscale Laboratory: Titration

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Abstract: The study attempts to develop microscale laboratory techniques for titration experiments. The microburet module was applied to replace standard burets conventionally used in chemical laboratories, resulting in reduced chemical and waste disposal costs. The subject of microscale titration was introduced into "Chemistry for Engineers", one of King Mongkut's University of Technology North Bangkok's undergraduates required subjects .

The designed Microburet unit contains a 0.3 cm-diameter, 20 cm-long buret attached to a 5.0 cm^3 syringe to control solution dropping amount. Two experiments were applied 1) acid-base titration and 2) hardness water experiment. Results were compared with those attained from standard burets. Less than twenty five drops of the solution were titrated during each session. The outcomes were compared with those from the conventional experiments. The microburet module was applied by 20 college engineering students. The laboratory results were consistent with those from the standard laboratory. Therefore, the developed module can efficiently replace the buret module and, thus, is implementable in authentic classrooms. The results revealed that the module could facilitate learners' scientific skills, increase their positive attitude towards science and student satisfaction assessment revealed their approval of the microburet module. The particular dimensions of convenience, rapidity, ability to decrease problems of chemical use and disposing of chemical waste were rated at the highest level of contentment among learners.

Keywords: Microscale Laboratory**,** Titration**,** Microburet **,** Scientific skills.

Introduction

This research aims to replace standard burets with the microburet module and implement the latter in the "Chemistry for Engineers" course attended by engineering undergraduates of King Mongkut's University of Technology North Bangkok. The students are given opportunities to conduct experiments and make assessment as part of constructivism learning. It is highly hoped that by the end of the course, the students will possess a set of scientific skills including observation, using numbers and experimenting.

Titration is volumetric analysis, a type of quantitative chemical analysis, in which a solution of known concentration or a standard solution is used to determine the concentration of an unknown solution. Knowing the volume of the standard solution and the unknown solution engaged in a reaction allows the determination of the concentration of the unknown.

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Nowadays large amounts of chemicals are used in chemical experiments, namely titration, resulting in increasing chemical costs and a large volume of waste that needs to be disposed. Therefore, microscale laboratory experiments have been developed to reduce chemicals used in experiments and, subsequently, replace commonly-recognized standard experiments.

Very tiny amounts of chemicals are used in microscale techniques. In a regular laboratory experiment, 5 -100 grams of chemicals, a baret of 25- 500 cubic centimeter capacity and normal-sized equipment are used. On the other hand, microscale experiments require much smaller amounts of chemicals: 0.05 - 1.0 grams, and burets containing less than 25 cubic centimeters of chemicals. Thus, some materials employed in microscale experiments are different from standard experiments. Some new tools and equipment are particularly developed to match the volume of chemicals used in such experiments. Some equipment widely used in laboratories is adjusted for microscale experiments. Some materials found in daily life are also used in the experiments. For example, in a research on redox reaction by Salinee Acharry (2008, 2009), beverage cans and food cans were used, and plastic bags for food storage with holes were used to hold chemicals. Another example is that Yuttapong Uttnan and his research team (2002) used sheet film boxes as containers of different types of solutions in an experiment on galvanic cells. Results from these two experiments were the same as those from standard experiments.

Microscale techniques have been continuously developed. At present, microscale techniques are taught at both high school and university levels. A laboratory book by Zvi Szafran and his team (1992) revealed that microscale laboratory that uses small amounts of chemicals in experiments can be applied in many chemistry courses such as spectroscopy, inorganic chemistry and organic chemistry. The reasons for which microscale techniques have been constantly developed are increased safety, reduced chemical and equipment costs, reduced waste generation and shortened experiment times that allow students to perform a lot of experiments in a classroom.

Wood (1990) suggested that, rather than demonstration and video or computer viewing, miniscale laboratory is a good option for teachers because it makes chemistry more interesting and inspiring for learners. In addition, hands-on labs offer many scientific skills such as observation, experimentation, data collection and data conclusion.

According to Wood's research, benefits of microscale experiments are:

1. Reduced experiment costs and waste generation

2. Reduced experiment times that allow students to perform a lot of experiments in a classroom

3. Reduced reliance on ventilation systems, protective masks and other safety tools. Thus, apart from a laboratory, experiments can take place elsewhere including a normal classroom.

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4. Many experiments are considered unsafe for learners but when microscale techniques are used, those experiments become more effective and safer such as an experiment of hydrogen plus oxygen reaction.

Hands-on experience is one of the best ways to teach chemistry because it gets learners involved in a classroom and allows them to verify a theory's results themselves. Moreover, experiments contribute to increased scientific skills in learners. The American Association for the Advancement of Science (AAAS) stated a scientific process role of promoting 13 skills divided into two main categories: 1) Basic science process skill comprising of eight skills: 1.Observation; 2. Measurement; 3. Using numbers; 4. Classification; 5. Space/space relationship and space/time relationship; 6. Organizing data and communication; 7. Inferring; and 8. Prediction; and 2) Integrated science process skill comprising of five skills: 1. Formulating hypothesis; 2. Defining operationally; 3. Identifying and controlling variables; 4. Experimenting; and 5. Interpreting data and conclusion. Scientific skills that the author would like to promote in learners are observation, using numbers and experimenting.

Significant meanings of each skill are as follows.

1. Observation is the ability to objectively exercise a sense or a combination of senses on an object or an event.

2. Using numbers is the ability to make calculations or dealing with figures representing volume of a substance based on observation, measurement and experimenting or other sources. The calculated figures have to be represented in the same unit so that they give the desired meaning in a clear manner.

3. Experimenting is a process of finding solutions or testing an established hypothesis by performing an experiment including three steps:

   3.1 Designing an experiment – planning an experiment before getting started so as to determine experiment methods involving identifying and controlling factors and materials used in the experiment.

   3.2 Conducting an experiment – starting an actual experiment

   3.3 Recording experiment results – recording information derived from an experiment which is probably the outcome of observation, measurement, etc.

Methodology

Section 1: Developing microscale laboratory techniques for titration experiments by creating a microburet module to replace standard experiments.

A designed microburet unit contains a 0.5 cm-diameter, 10 cm-long buret attached to a 1 cm^3 syringe to control solution dropping amount. A recycled plastic bag with eight small

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holes is used to hold a chemical during a titrating process as seen in Figure 1. Figure 2 shows a standard buret commonly used in laboratories.

Figure 1. Microburet module

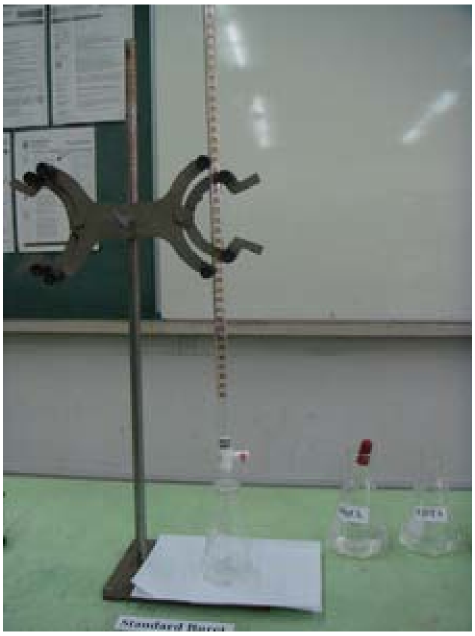

Figure 2. Standard buret

Section 2: Performing two titration experiments

1. Acid-base titration experiment – A base, NaOH at a 0.05 mol/dm^3 concentration, used as a standard solution, is titrated with three acid samples: sulfuric acid; hydrochloric acid; and acetic acid, with known volumes but unknown acid concentrations in order to find the volume of the standard solution, and consequently, find the acid concentrations of the three acids.

2. Water hardness experiment – Two hard water samples are titrated with EDTA to quantify calcium ion, Ca^{2+}.

Microburets were used in both experiments, as seen Figure 3. A titration experiment with the use of standard burets was compared and shown in Figure 4. Experiment results were analyzed and calculated.

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Figure 3. In microburet module titration, only 5-25 drops of chemicals were used.

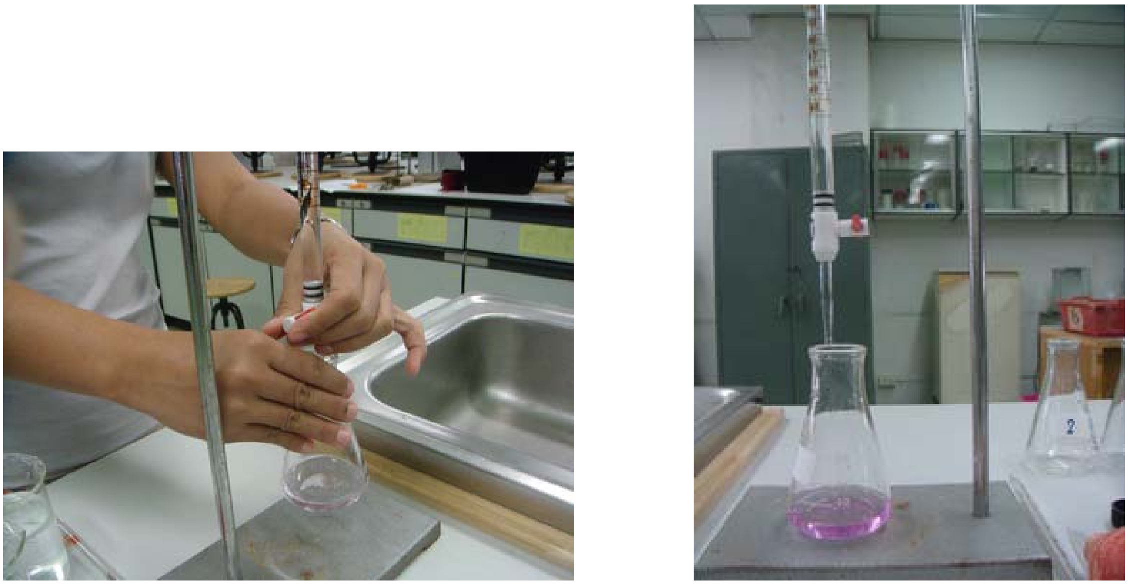

Figure 4. In standard buret titration, around 10-40 cm^3 of chemicals was used.

Section 3 Learning Activities

Twenty engineering undergraduates of King Mongkut's University of Technology North Bangkok were divided into ten groups (two persons/group). Each group conducted titration experiments using microburets with microscale techniques, and standard burets. The students studied experiment handouts from the teacher and conducted the experiments by themselves. They were told to collect data from the experiments, record, report and present the results, show their calculation methods, discuss the results and conclusions with their classmates. Meanwhile, the teacher observed the learners' scientific skills: observation, using numbers and experimenting.

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Figure 5. During the learning activities

Result

Section 1 Comparison of microscale and standard experiments

1.1 It was found that results from acid-base titration microscale laboratory experiments were consistent with those from the experiments using standard burets. Table 1 shows results from acid-base titration microscale laboratory experiments. Table 2 shows results from acid-base titration standard buret experiments.

Table 1. Results from acid-base titration microscale laboratory experiments

Samples (5 drops = 0.2 cm^3)	Number of drops : NaOH volume at 0.05 M concentration				Acid concentration from calculation mol/dm^3	Concentration from preparation mol/dm^3
	1st time drop : cm^3	2nd time drop : cm^3	3rd time drop : cm^3	Mean volume cm^3		
1. HCl	4 drops = 0.16 cm^3	6 drops = 0.24 cm^3	5 drops = 0.20 cm^3	0.20	0.05	0.05
2. H_2SO_4	24 drops = 0.96 cm^3	24 drops = 0.96 cm^3	26 drops = 1.04 cm^3	0.99	0.12	0.10
3. CH_3COOH	8 drops = 0.32 cm^3	8 drops = 0.32 cm^3	9 drops = 0.36 cm^3	0.33	0.08	0.10

Note: One drop of solution equals 1/25 cm^3.

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Table 2. Results from acid-base titration standard buret experiments

Samples 10 cm^3	NaOH volume at 0.05 M concentration (cm^3)				Acid concentration from calculation mol/dm^3	Concentration from preparation mol/dm^3
	1st time	2nd time	3rd time	Mean volume		
1. HCl	12	14	13.5	13.17	0.06	0.05
2. H_2SO_4	46	48	44.5	46.17	0. 12	0.1
3. CH_3COOH	16	14	14	14.67	0.07	0.1

Reactions of acid-base titration:

1. $HCl\ (aq) + NaOH\ (aq) \rightarrow NaCl\ (aq) + H_2O\ (l)$
2. $H_2SO_4\ (aq) + 2\ NaOH\ (aq) \rightarrow Na_2SO_4\ (aq) + 2\ H_2O\ (l)$
3. $CH_3COOH\ (aq) + NaOH\ (aq) \rightarrow CH_3COONa\ (aq) + H_2O\ (l)$

1.2 Two water samples were analyzed to determine water hardness. A titration method was used to quantify calcium ion, Ca^{2+}. Water hardness results from microscale laboratory experiments are shown in Table 3.

Table 3. Water hardness results from microscale laboratory experiments

Samples (10 drops = 0.4 cm^3)	Number of drops : EDTA volume at 0.0026 M concentration				Hardness	
	1st time drops : cm^3	2nd time drops : cm^3	3rd time drops : cm^3	Mean volume – Adjusted volume (cm^3)	Calculation (ppm)	Preparation (ppm)
1st hard water sample	24 drops = 0.96 cm^3	24 drops = 0.96 cm^3	24 drops = 0.96 cm^3	0.96-0.60 = 0.36	234	220
2nd hard water sample	28 drops = 1.12 cm^3	30 drops = 1.20 cm^3	27 drops = 1.08 cm^3	1.13-0.60 = 0.53	344.5	350

Note: One drop of solution equals 1/25 cm^3.

Reaction of EDTA with Ca^{2+} :

$$EDTA\ (H_2Y^{2-})\ (aq) + Ca^{2+}\ (aq) \Leftrightarrow CaY^{2-}\ (aq) + 2\ H^+\ (aq)$$

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It was found from percent error calculations of water hardness analysis that:

1. Percent error of the first hard water sample

$$= \frac{(234 - 220) \times 100}{220} = 6.36\ \%$$

2. Percent error of the second hard water sample

$$= \frac{(350 - 344.5) \times 100}{350} = 1.57\ \%$$

It was clear that microscale laboratory experiments of water hardness yielded precise and realistic results.

Section 2 Results from classroom activities

It was found that microscale laboratory exercises were successful in that learners are eager to perform experiments, find information and answer questions, and feel inspired by their own experiments. Furthermore, having a true understanding of titration, students are more willing to participate in class, and more equipped with scientific skills. For example, 1) Observation skill – students observe the changing color of indicators as solutions react and reach the equivalent point, where a chemical reaction stop. 2) Number using skill – students make calculations to determine acid concentrations of acid samples and hardness of water samples. 3) Experimenting skill – students were able to perform titration experiments by themselves and receive accurate and precise results.

The teacher was able to run the class within the pre-planned time period since microscale experiments require small amounts of chemicals reducing the experimenting and clean-up time, and leaving more time for discussion between teacher and students.

Conclusion

Results of microscale laboratory titration experiments using a microburet module are as follows.

1. Microscale experiments boost understanding and learning as much as standard-scale experiments.
2. Scientific skills of observation, using numbers and experimenting are enhanced in students.
3. The experiments are uncomplicated. Small amounts of chemicals are used. Equipment is easy to use. Experiments can take place in a classroom, not only in a laboratory.
4. A class can be run within a pre-planned time period.
5. Using small amounts of chemicals helps reduce chemical and decontamination costs.

Acknowledgement

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Each titration must be continued through two equivalence points.Â 1 Adapted for microscale quantities by M. D. Gheorghiu. The experiment includes contributions from past instructors, course textbooks, and others affiliated with course 5.310. Titration-1.Â Bring your analysis of your titration data to the laboratory. If you are not satisfied with your data discuss your concerns with your TA before carrying out additional titrations. V. data analysis and discussion-required. Computer Microscale Acid-Base Titration 36 py A titration is a process used to determine the volume of a solution needed to react with a given amount of another substance. In this experiment, you will titrate hydrochloric acid solution, HCl, with a basic sodium hydroxide solution, NaOH. The concentration of the NaOH solution is given and you will determine the unknown concentration of the HCl. Hydrogen ions from the HCl react with hydroxide ions from the NaOH in a one-to-one ratio to produce water in the overall reaction: H+(aq) + Clâ€“(aq) + Na+(aq) +OHâ€“(aq) ï£§ ï£§â†’ H2O(l) + Na+(aq) + Clâ€“(aq) co W