Portable syringe-vial kit of gas-generating reactions for easy demonstration of chemical reaction rate

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Abstract. In this article, a low-cost, portable syringe-vial experimental activity of gas-generating reactions is reported for easy illustration of the concept of chemical reaction rate in a regular classroom context. The equipment setup consists of a Luer-lock plastic syringe, a vial and its screw-cap connected to a syringe by a 3-way Luer-lock stopcock for the quantification of the gaseous product generated from the reaction. To operate the kit, an acid solution is first poured into the vial, then a plastic container of sodium bicarbonate is carefully floated on the solution. All three components of the kit are gently connected together. Finally, the vial is shaken to start the reaction and then the volume of the gas product is collected in the syringe versus time. This activity allows students to easily investigate the rate of reaction and also concentrations affecting it by plotting a relation between volume of gas product with time.

1. Introduction
Rate of chemical reaction or chemical kinetics in particular is one of the most difficult topics in chemistry since it involves many factors that can influence the reaction rate and requires mathematical calculation [1]. Many students across the world encounter these difficulties as reported in previous studies [2-6]. The definition of the rate of chemical reaction is given as ‘the change in the concentration of a reactant or a product with time’ [7-8]. Several effective activities have been developed to enhance students’ experience and understanding of this concept. These include observation of the gas volume such as that of CO$_2$ generated from a reaction between an acid and calcium carbonate in eggshells [9], and color change by spectrophotometer or by naked eye of a clock reaction [10] or blue bottle reactions [11-12]. The concept of reaction rate has also been explored through observation of the oxidation rate of nails [13], iron(II)-induced oxidation of iodide by chromium(VI) [14], inquiry experiment incorporated with analogy activity [5], small-scale reaction between hydrochloric acid and sodium thiosulfate to produce colloidal sulfur [15], total order of the iron–oxygen reaction [16], kinetics of zinc with sulfuric acid [17], and yeast fermentation kinetics [18], etc. Many of these activities, however, have a disadvantage in that they are mostly performed on a time-consuming traditional scale, with difficult-to-set-up equipment making them unsuited for classes especially at a high school level.

Gas-generating reactions are frequently used to investigate the rate since they are easy, inexpensive, and green [19]. One of the most popular reactions is the generation of the CO$_2$ gas from sodium bicarbonate and an acid as shown in equation (i).

$$\text{NaHCO}_3(s) + \text{H}^+(aq) \rightarrow \text{CO}_2(g) + \text{Na}^+(aq) + \text{H}_2\text{O}(l)$$ (1)
For equation (1), one mole of NaHCO$_3$ will theoretically produce one mole of CO$_2$, 22.4 L at STP. The rate of this reaction is shown in equation (2). However, the most convenient and reliable way to determine the rate of this reaction is to investigate the volume of the produced gas with time.

$$\text{rate} = \frac{\Delta [\text{NaHCO}_3]}{\Delta t} = \frac{\Delta [\text{H}^+]}{\Delta t} = \frac{\Delta [\text{CO}_2]}{\Delta t} = \frac{\Delta [\text{Na}^+]}{\Delta t}$$ (2)

The volume of CO$_2$ under STP ($V_{\text{STP}}$) and under an experimental condition ($V_{\text{Exp}}$) can be interconverted using the combined gas law shown in equation (3).

$$V_{\text{STP}} = V_{\text{Exp}} \left( \frac{273.15 \text{ K}}{T} \right) \left( \frac{P}{760 \text{ mmHg}} \right)$$ (3)

Many methods can be used to measure the volume of an evolved gas such as water displacement and electronic sensing [18]. Water displacement in a glass-burette is often used as a standard traditional method for measuring the volume of a generated gas. Herein, we report an alternative, portable and time-saving method for measuring the volume of a gas. Available low-cost materials including a syringe and a glass vial and its screw-cap connected with a stopcock are used to quantitatively measure the volume of the gas. This small-scale and greener kit yields reproducible, quantitative data that can be used to explore the principles of reaction rate in chemistry classroom settings from high school to college.

2. Method

2.1. Development of the Syringe-Vial Kit

The volume of the CO$_2$ gas from conventional scale reactions (2.00 mmol of NaHCO$_3$ and 4.00 mmol of acid) measured by water displacement (WD) in a burette method (Figure 1a) was first investigated to obtain the optimum concentration of the acid. The normal scale was then downsized four times (0.50 mmol of NaHCO$_3$ and 1.00 mmol of acid) to be used in the small scale. The theoretical yields of CO$_2$ calculated from limited reagent, NaHCO$_3$, in both scale reactions. The volume of the CO$_2$ gas from the small-scale reaction was measured by both the WD (Figure 1b) and the syringe-vial (SV) methods (Figure 1c) to verify whether the latter can be effectively used as the alternative method.

![Figure 1](image)

Figure 1. Gas volume determination by (a) normal-scale WD method, (b) small-scale WD method, and (c) small-scale SV method.

2.2. Construction of the Syringe-Vial Kit

The construction of the syringe-vial kit was adapted from two classroom demonstrations, rapid reaction between H$_2$ gas and Pd metal in plastic syringe system [20] and the syringe-to-syringe transfer procedure in microscale gas chemistry [19]. Equipment and materials needed for the fabrication of the kit are listed in Figure 2a. For the kit fabrication, a hole was made into the screw-cap of the 10-mL vial using an
electric drill for the insertion of the tubing port of a 3-way (or 2-way) Luer-lock stopcock. All purpose glue was applied on the top of the screw-cap to secure the connection tightly and Parafilm tape was applied at connectors to prevent gas leakage. In this setup, a syringe was connected to the 3-way stopcock glued to the screw-cap, while the vial was connected with its screw-cap on the opposite side (Figure 2b). Soapy water test was performed to ensure that there was no gas leakage at the joined pieces.

![Figure 2](image.png)

**Figure 2.** Equipment and materials (left) and assembly of the vial-syringe kit (right).

### 2.3. Chemicals and Hazards

The chemicals used in this experiment include sodium bicarbonate (NaHCO₃) and aqueous hydrochloric (HCl) solution. These corrosive acid solutions can cause skin, eye and respiratory system irritation. Sodium carbonate (Na₂CO₃) was also used as a primary standard of acid solutions by titration.

### 2.4. Experimental Procedure for Conventional Scale Reaction using WD Method

The volume of the CO₂ gas generated from the conventional scale reaction can be investigated by using the regular WD in a burette method as shown in the following procedure: 1) pipetting 20.00 mL of HCl solution and transferring it into an Erlenmeyer flask followed by assembling all equipment as shown in Figure 1a, 2) transferring a container of carbonate compound into the flask and closing the flask with a stopper, 3) adjusting the volume of water in the burette and then shaking the flask until the solid reactant is completely mixed with the acid solution, while starting the timer, and 4) recording gas volume in the burette with time until the reaction completion. Plotting a graph to show the relationship between gas volume and time to be used for the investigation of the rate.

### 2.5. Experimental Procedure for Small-Scale Reaction using WD Method

The water displacement in a burette method was then applied for the investigation of the volume of the CO₂ gas generated from the small-scale reaction. A small vial and its screw cap were used instead of an Erlenmeyer flask and its stopper. The vial screw cap was modified by connecting it with a syringe 3-way connector and rubber tube. The experimental procedure for the investigation of the volume of the CO₂ gas was similar to the conventional method as shown in Figure 1b.

### 2.6. Experimental Procedure for Small-Scale Reaction using SV Method

The procedure for the investigation of the volume of the CO₂ gas generated from the small-scale reaction by using the SV method (Figure 3, see also Figure 1c) is as follows: 1) pipetting 5.00 mL of HCl solution and transferring it into a glass vial, 2) transferring a container of carbonate compound into the vial by using forceps, 3) connecting all parts together as shown in Figure 1c, and 4) shaking the syringe-vial system until the solid reactant is completely mixed with the acid solution, while starting the timer.
Recording gas volume in the syringe with time until the reaction completion. The demonstration VDO is also available at https://youtu.be/TWRjEEMQgfU.

![Syringe Illustrations](image)

**Figure 3.** SV kit Illustrations for (a) preparation of samples, (b) loading sample container into the vial, (c) connecting all parts and shaking, and (d) measuring volume change with time.

3. **Result and Discussion**

The results of this study were divided into three parts: 1) volume of the CO$_2$ gas from conventional and small-scale reactions, 2) average rate of CO$_2$ generation from conventional and small-scale reactions, and 3) students’ achievement upon learning about chemical reaction rate by using the syringe-vial kit.

3.1. **Volume of the CO$_2$ Gas from Conventional and Small-Scale Reactions**

The results from the CO$_2$ gas volume investigation obtained from both conventional and small scales are shown in Table 1. Please note that the volume of gas reported in this study was converted to STP condition to make it possible for any comparison among different experimental conditions. There were 3 sub-reactions in this experiment in which the limiting reagents for reaction A and C were HCl and NaHCO$_3$, respectively.

**Table 1.** Rate of CO$_2$ gas generation from reaction between NaHCO$_3$ and HCl (all volumes were converted to STP condition)

| Methods | Conventional scale WD | Small-scale WD | Small-scale SV |
|---------|-----------------------|----------------|---------------|
|         | Rxn A | Rxn B | Rxn C | Rxn A | Rxn B | Rxn C | Rxn A | Rxn B | Rxn C |
| Mole (mmol) of NaHCO$_3$/HCl | 2.0/1.0 | 2.0/2.0 | 2.0/4.0 | 0.5/0.25 | 0.5/0.5 | 0.5/1.0 | 0.5/0.25 | 0.5/0.5 | 0.5/1.0 |
| Limiting | HCl | - | NaHCO$_3$ | HCl | - | NaHCO$_3$ | HCl | - | NaHCO$_3$ |
| Theoretical volume of CO$_2$ (mL) | 22.40 | 44.80 | 44.80 | 5.60 | 11.20 | 11.20 | 5.60 | 11.20 | 11.20 |
| Final time (s) | 900 | 1080 | 720 | 220 | 280 | 180 | 220 | 280 | 180 |
| Volume of CO$_2$ at 20s (mL) | 4.94 | 8.55 | 12.95 | 2.81 | 4.28 | 7.13 | 2.90 | 4.73 | 7.27 |
| Final volume of CO$_2$ (mL) | 20.52 | 38.70 | 41.22 | 4.97 | 9.52 | 10.09 | 4.77 | 9.45 | 9.93 |
| %Yield$^*$ | 91.61 | 86.38 | 92.01 | 88.75 | 85.00 | 90.09 | 85.18 | 84.38 | 88.66 |
| Rate at 20s, $r_{20}$ ($\times 10^{-2}$ mL/s) | 24.70 | 42.75 | 64.75 | 14.05 | 21.40 | 35.65 | 14.50 | 23.65 | 36.35 |
| Average rate, $r_{av}$ ($\times 10^{-2}$ mL/s) | 2.28 | 3.58 | 5.73 | 2.26 | 3.40 | 5.61 | 2.17 | 3.40 | 5.52 |
The theoretical yields of reactions A and B-C at STP condition were 22.4 and 44.8 mL for the conventional scale and 5.6 and 11.2 mL for the small scale. The percent yields of the CO$_2$ gas of the conventional WD, small-scale WD, and small-scale SV experiments were in the range of 86.38 – 92.01, 85.00 – 90.09 and 84.38 – 88.86, respectively. Their percent yields of all reactions were in a similar order. However, the yields of the CO$_2$ gas from the small-scale WD method were slightly lower than those of the conventional scale WD method (~ 1 - 3%). The experimental yield from both WD methods were lower than expected due to different equilibrium solubility in water for CO$_2$ [21], 0.034 mol/L at 25 °C and 101.3 kPa [22]. When considering only the small-scale reactions, it showed that the yields of CO$_2$ gas generation from the SV method were slightly lower than those of the WD method, ranging from 0.63 – 3.57%, but still following the same trend. The obtained lower yield from the SV method was due to the fact that in some cases the plunger did not move until a certain internal pressure was achieved [23]. Every syringe was different with regards to its movability; therefore, the range of observed gas volumes from the SV method was smaller than those of the WD method [23].

It was observed that reaction A produced the lowest CO$_2$ gas volume since the amount of HCl was limited and less than in other reactions. The reason behind the lowest percent yield in reaction B might be because either the amount of HCl or NaHCO$_3$ was exactly limited to lower than calculated. By comparison, reactions A to C can respectively demonstrate how the concentration of HCl affect the volume of the CO$_2$ product [2,5].

3.2. Rate of CO$_2$ Gas Generation from Conventional and Small-Scale Reactions

Average rates (r$_{av}$) of these reactions were also calculated from the total volume of CO$_2$ and reaction time required to complete the reaction. The average rates for the conventional scale WD method, small-scale WD method, and small-scale SV method were in a similar order (see Table 1). It was found that increasing concentration of HCl increased the rate of reaction [5,8]. It can be seen that when the concentration of HCl was doubled (A to B and B to C), the average rate (r$_{av}$) of the reaction was almost doubled (~ 1.51 – 1.73 times).

The volumes of the CO$_2$ gas for these reactions were also investigated versus time which can then be used to determine the rate of the chemical reaction including their initial rates (Figure 4). The rate of a reaction goes through changes during any reaction. It reaches its maximum at the beginning, decreases during the reaction, and is equal to zero when the reaction is completed. As a result, the initial rate (r$_i$), the instantaneous rate at the start of the reaction (t = 0), provides meaningful kinetic information. The initial rate is equal to the slope of the tangent of the volume of carbon dioxide (product) versus time at time zero [24]. However, the instantaneous rate during the first 20 s (r$_{20s}$) was discussed instead of the initial rate (r$_i$) in this study. It can be seen that the initial rate (r$_i$) and the average rate (r$_{av}$) of the reaction were in the same trend. When the concentration of HCl was doubled (A to B and B to C), initial rate (r$_i$) of the reaction was almost doubled (~ 1.53 – 1.65 times).

Figure 4. Volume of CO$_2$ gas versus time from (a) Conventional scale by WD method, (b) Small scale by WD method, and (c) Small scale SV method.
All graphs (Figure 4.) illustrate the same trend that the concentration of HCl affects the rate of the reaction. The results show that increasing concentration or mole of the HCl solution (mole of NaHCO₃ is constant) increased the rate of the reaction [6-9]. Both methods provided the same trend of the rate (Figure 4b-c) corresponding with the rate of the conventional scale (see also Figure 4a). When considering only the small-scale reactions, the WD method gave slightly greater initial rate than that of the SV method. This may arise from stiffness of the syringe that resisted movement until the internal pressure was adequate [23]. Some petroleum gel can be applied to reduce this issue. Please note that the S.D. of the SV method was slightly greater than that of the WD method because the graduation interval of the syringe (0.2 mL) was rougher than that of the burette (0.1 mL).

### 3.3. Students’ Achievement of Chemical Reaction Rate upon Learning by using the Syringe-Vial Kit

The SV kit was used to demonstrate the concept of chemical reaction in the chemistry course for grade-11 students. With prior permission from the school principal, 30 students (one regular-sized classroom) at a public school in Thailand voluntarily participated in this study during semester 1 of 2019. By using the SV kit, they participated in three inquiry-based learning activities to determine the rate of the reaction between NaHCO₃ and HCl (60 min, including discussion), and to investigate the effects of acid concentration (60 min) and species on the rate (60 min). They performed the activities as a pair in a normal classroom context. They were able to repeat the experiment easily since the time required for investigating each round of the reaction was only about 5 min and there was no need to change the volume measuring equipment (top part); they only had to change the vial (lower part). To verify that the kit was an effective tool that aided their understanding of the reaction rate concepts, they took a 30-min pre-test prior to the activity, which was evaluated against a post-test. Both tests contained the same 10 multiple-choice questions. Their scores on the pre-lab and post-lab test are shown in Figure 5.

![Figure 5. Students’ scores on the pre- and post-lab test of chemical reaction rate.](image)

Obviously, students showed a significant improvement in their understanding of the rate of chemical reaction concept after undertaking the experiment, which aligned with the previous studies [2,5]. Their average post-lab test score (mean 8.20, S.D. 1.32) was statistically higher than the pre-lab test score (mean 4.02, S.D. 1.13) at the 95% significant level (p < 0.001, df = 29, t = 16.399). This finding suggests that the use of the kit helps students gain a better understanding of the rate of chemical reaction. In addition, students were asked to comment on the experiment and their responses were positive. For example, they revealed “I loved participating in this experiment since it was easy to perform and observe.” and “This was one of the safest chemistry experiments I’d ever performed so I did it without any worry.” This kind of experiments allows students to actively improve their corresponding understanding and achievement [25-26] as effective as a traditional experiment.

### 4. Conclusion

This study verified that the use of the syringe-vial kit as a classroom activity was suitable for the investigation of the rate of CO₂ gas-generating reactions and demonstration of the concept of reaction rates. This kit minimized certain serious limitations compared to the WD method because it was
repeatable, easy to set up, low-cost, safe to perform, and less time-consuming for operation (< 5 min). We have shown that the students were able to easily carry out and repeat the experiment within less than 10 min for each reaction investigated. In addition, it provided students with a chance for individual hands-on activity, which contributed to their improvement on the scores when comparing the post- with the pre-tests.

One of the limitations of using this SV kit was the inherent differences among the syringes. Some syringe plungers did not move until a certain internal pressure was reached which resulted lagging of the chemical reaction. In case that these differences are significant, the observed volume and time should be subtracted by the volume and lag time before use [24]. This kit can be easily applied to further investigate how acid species (i.e., weak CH₃COOH, strong monoprotic HCl, and strong diprotic H₂SO₄) and/or carbonate species (i.e., NaHCO₃, Na₂CO₃ and CaCO₃) affect the rate of CO₂ gas-generating reactions.

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