Microscopic Observation of Solid-Liquid Reaction: A Novel Laboratory Approach to Teaching Rate of Reaction

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ABSTRACT

The importance of observation in science and science education has triggered this laboratory development study that investigated the value of an observation kit as a new approach to teaching rate of reaction in general chemistry class. The kit consists of a digital microscope, a "chemical reactor", and a tailor-made computer application and was used to video-record a solid-liquid reaction and to produce a series of two dimensional solid images that indicate the extent of reaction. The two dimensional image areas were calculated by the computer application by assuming that the image area was directly proportional to the solid mass from which a plot of mass versus time could be obtained. These steps have been tested to solid (zinc, iron, calcium carbonate, and magnesium oxide)-liquid (acid solution reaction systems. Reaction of solid magnesium oxide with nitric acid solution resulted in the best images which were transferable to a plot of solid magnesium oxide mass as a function of time. This was used to explain rate of reaction concepts including average, instantaneous, and initial rate. Furthermore, the effect of concentration on reaction rate could also be explained. The generated data allows students to clearly and repeatedly visualize a solid-liquid reaction and relates rates of reaction concepts. The observation kit also allows teachers and students to extend its application into inquiry based experiments.

Keywords: *microscopic observation; solid liquid reaction; rate of reaction; inquiry learning*

ABSTRAK

Penelitian pengembangan perangkat dan kegiatan laboratorium ini didasari oleh pentingnya observasi dalam sains dan pendidikan sains. Perangkat pengobservasi reaksi kimia yang dikembangkan dapat digunakan dalam perkuliahan laju reaksi dalam kelas kimia dasar. Dari serangkaian reaksi kimia yang diuji coba, reaksi antara padatan magnesium oksida dengan larutan asam nitrat berhasil memberikan data berupa image yang dapat diolah menjadi informasi kinetik. Berdasarkan massa MgO sebelum reaksi, data image partikel yang diperoleh dalam video diubah menjadi data massa, sehingga diperoleh plot massa magnesium oksida sebagai fungsi waktu. Dari data yang diperoleh, mahasiswa diperkenalkan pada konsep laju reaksi, penentuan nilai laju, dan pengaruh konsentrasi terhadap laju reaksi dan orde reaksi. Set data yang diperoleh memungkinkan mahasiswa untuk secara visual mengamati reaksi padatan dengan cairan dan menghubungkannya dengan konsep laju reaksi. Perangkat pengobservasi ini juga, berpeluang untuk diintegrasikan dan dikembangkannya ke dalam pembelajaran berbasis inkuiri.

Kata Kunci: *pengamatan mikroskopik; reaksi padat-cair; laju reaksi; pembelajaran inquiry*

INTRODUCTION

The use of in-situ observation instruments to study various chemical processes has gained much attention for a long time. One example is the use of Environmental Scanning Electron Microscopy (E-SEM) to observe heat-induced change in material chemistry [1]. Another study [2] used an optical microscope to investigate the type of interaction between the catalyst material ($Cs_2SO_4.V_2O_5$) and soot material (mainly carbon) from room temperature to 350 °C. By using surface plasmon spectroscopy, a chemical reaction on the surface of a nano-sized gold particle was observed [3].

Those reported research activities are good examples that showed the importance of scientific observation in science investigation which is very important for both school and always on hand at many universities, for the sake of undergraduate chemistry classrooms, these scientific observation activities could be replicated in simpler and cheaper equipment.

Simplified replication of equipment or research instruments to become a classroom demonstration

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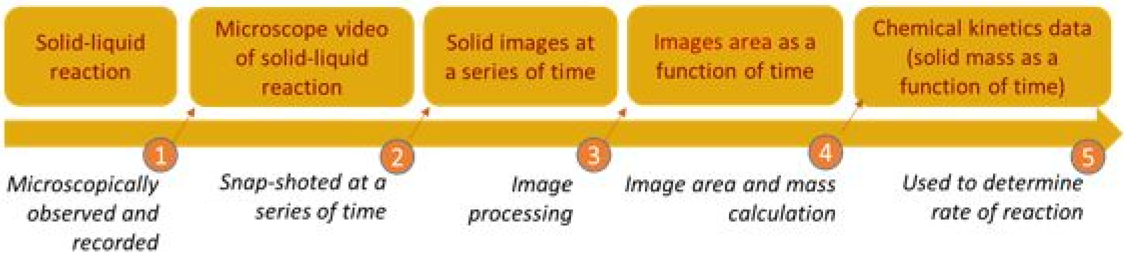

Fig 1. Logical framework for the development of this simple equipment

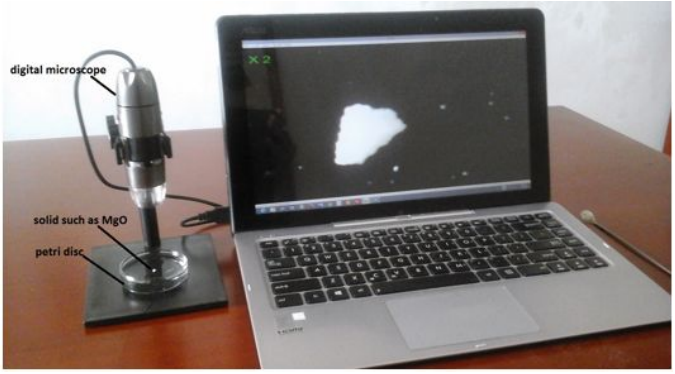

Fig 2. Arrangement of the solid-liquid chemical reaction observation kits

device has been carried out by many chemistry educators [4–11]. One example is a homemade spectrophotometer that can be made of inexpensive components [4]. This simplified spectrophotometer consists of, among others, flashlight as a light source, a compact disc as a diffractor net, and an LDR as a detector. Other examples of developing simple equipment are visible light spectrometer for use in secondary school [5] and a photochemical reactor used for five organic photochemical laboratory experiments [6]. To demystify the application of electrochemistry in sensor equipment, a simple oxygen sensor device using zinc-air electrochemical cells was developed [11]. To provide photochemical laboratory experiments, an inexpensive and commercially available ultraviolet light device, intended for “drying” gel-type fingernail polish, was developed by a group of researchers [6]. The fundamentals of UV spectroscopy, data handling, calibration, and sampling, can be learned by student to measure dissolution rates of tablets [7].

In this present study, we develop an observation kit that can be used for chemical kinetics experiments specifically for the teaching of reaction rate. The logical framework for the development of this simple equipment is presented in Fig. 1.

The kit consists of a digital microscope, a “chemical reactor”, and a tailor-made computer application. This was used to video-record a solid-liquid reaction to produce a series of two dimensional images of the solid that indicate the extent of reaction. The two dimensional image areas were assumed to be directly proportional to solid mass and were calculated by the computer application to produce the areas of the captured images to time data series. This present article also reports the test results of the kit for observing solid-liquid reactions and their data processing results. The data obtained were evaluated for their reliability based on their suitability with the reaction rate theory and their feasibility to use in the learning process of reaction rates.

EXPERIMENTAL SECTION

To implement the framework, three main activities were carried out: (a) developing of an observation kit for solid-liquid reaction, (b) developing the application for processing observation data, and (c) testing and improving the kit and its computer application. The reliability of the data was tested based on their feasibility to use in reaction rate analysis.

Materials

For the solid-liquid reactions tested, the solid materials were zinc, iron, calcium carbonate and magnesium oxide and the liquids were hydrochloric acid (2 M) and nitric acid (1 and 2 M). All chemicals were purchased from Merck.

Kit and Its Observation Procedure

The observation kit consists of a digital microscope equipped with a Petri dish that was used as chemical reactor which is schematically presented in Fig. 2. The digital microscope was connected to a computer that video recorded the chemical reaction. The solid-liquid reaction was carried out by putting about 1.5 mg solid in a Petri dish. Its initial appearance and size were then recorded. Subsequently, 5 mL acid solution was poured into the Petri dish and the digital microscope immediately started to record the process.

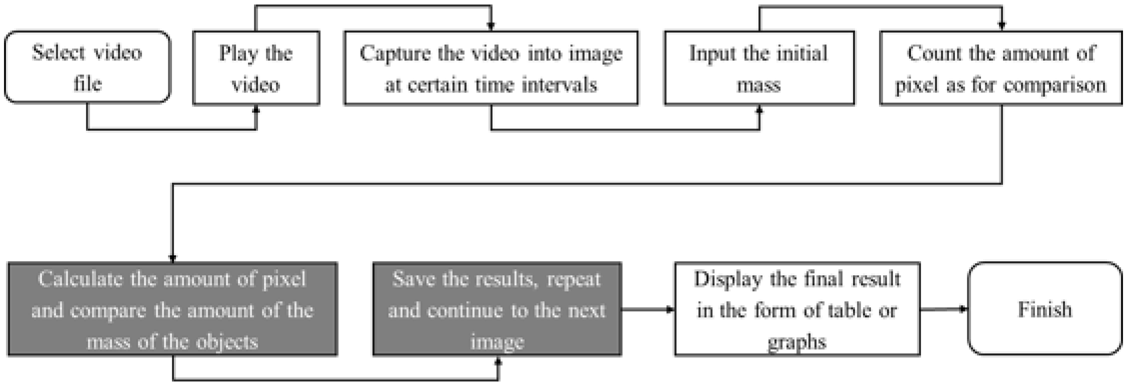

Fig 3. Application work flow to process the video of chemical reaction in to chemical kinetics (mass vs. time) data

Table 1. Summary of solid-liquid reaction observation test

No	Reaction	Notes on the video recording
1.	Zn + HCl	• The particle image was difficult to analyze due to the position changes • The solid plate edges with the bubbles is not clear • Two-dimensional shape of solid zinc (plate) did not represent the zinc quantity • The size took a long time to change
2.	Fe + HCl	• The particle image was difficult to analyze due to position change and the color of Fe image which resembled the image of gas bubbles • The iron grain moved from its position for several times due to the high gas production • The size took a long time to change
3.	$CaCO_3$ + HCl	• The particle of $CaCO_3$ split into several parts resulting in difficulty in analyzing the image – due to the size
4.	MgO + HNO_3	• The color of MgO solid was relatively in contrast with that of the very little bubbles that could still be observed • The MgO grain was relatively intact so that the size of the remaining MgO could be analyzed. • Changes in size took place within a reasonable time interval

(a) (b) (c)

Fig 4. The screen shots of the reaction video between: a) zinc and hydrochloric acid, b) iron and hydrochloric acid, and (c) calcium carbonate and hydrochloric acid after 30 sec reaction time

Application for Processing Observation Data

For easy transformation of a solid grain image to mass data, a software application to process observation videos of chemical reaction into kinetic data was developed. The application has features and data processing steps as summarized in Fig. 3. As shown in the figure the main steps in data processing are to capture image at given time intervals, to calculate image area by comparing the number of pixels, to then calculate the solid mass and plot it as a function of time.

RESULT AND DISCUSSION

Solid-Liquid Reaction Observation Test

The observation kit was applied to several solid-liquid reactions namely; zinc- hydrochloric acid, iron-hydrochloric acid, calcium carbonate-hydrochloric acid and magnesium oxide-nitric acid reactions. The results are summarized in Table 1, while examples of captured image are shown in Fig. 4.

Table 2. The screenshots of the observation results: the reaction of solid MgO

Time (sec)	MgO Image	Remarks
Before reaction		The image of an MgO grain before reaction. The grain was around 2 mm in size and 1.5 mg in mass.
0		The image of the MgO grain soon after the adding of 2 M nitric acid solution.
60		The image of MgO grain at 60, 120, and 240 sec after the addition of nitric acid solution. The particle size decreased compared to the initial.
120		
240		MgO fully reacted in 320 sec.

Reaction between zinc and hydrochloric acid resulted in grain images which were difficult to analyze. As can be seen in Fig. 4 (a) gas bubbles were evolved from the zinc surface causing difficulties in detecting solid image boundaries. Furthermore, the zinc size changes were hardly observed for a long reaction time. Similar results were shown by the reaction between iron and hydrochloric acid. Another type of results was shown by the reaction between calcium carbonate and hydrochloric acid where the grain was splitted into several parts resulting in difficulties for image analysis. Therefore, the results of zinc, iron, and calcium carbonate containing reaction system were not discussed further.

Promising results were produced by the reaction between solid magnesium oxide and nitric acid. This reaction produced images with relatively clear shapes and solid boundaries. The screen shots of the MgO-HNO_3 reaction observation are summarized in Table 2. In this experiment, 1.5 mg of solid MgO fully reacted with 5 mL of 2 M HNO_3 in 312 sec. The observation screen shots of the reaction during 312 sec reaction time are highlighted in Table 2. As can be seen in the table, an approximately 2 mm size grain of MgO decreased within 1 min time. This decrease continued further until the solid MgO was completely reacted in 312 sec.

Small bubbles were still seen in the reaction between magnesium oxide and 2 M nitric acid, probably caused by the reaction of the acid with small amounts of magnesium carbonate impurities (this could result from incomplete calcination of magnesium carbonate during manufacture or reaction of the MgO with carbon dioxide in the atmosphere).

These reaction observations provide chemical kinetics information which is very useful for learning reaction rates. The information can be summarized as follows: (a) The observations showed that a chemical reaction occurred in the system as indicated by the solid MgO size decreases, (b) The time for MgO to react with HNO_3 reflect the relative reaction rate; the higher the concentration of acid, the faster is the rate of the solid-liquid reaction, and (c) if the remaining solid MgO at a time interval is assumed to positively correlate with grain volume, then it is directly proportional to the image area of the solid. The reaction rate is comparable to the decreased area of the image per time unit that can be identified using equation 1.

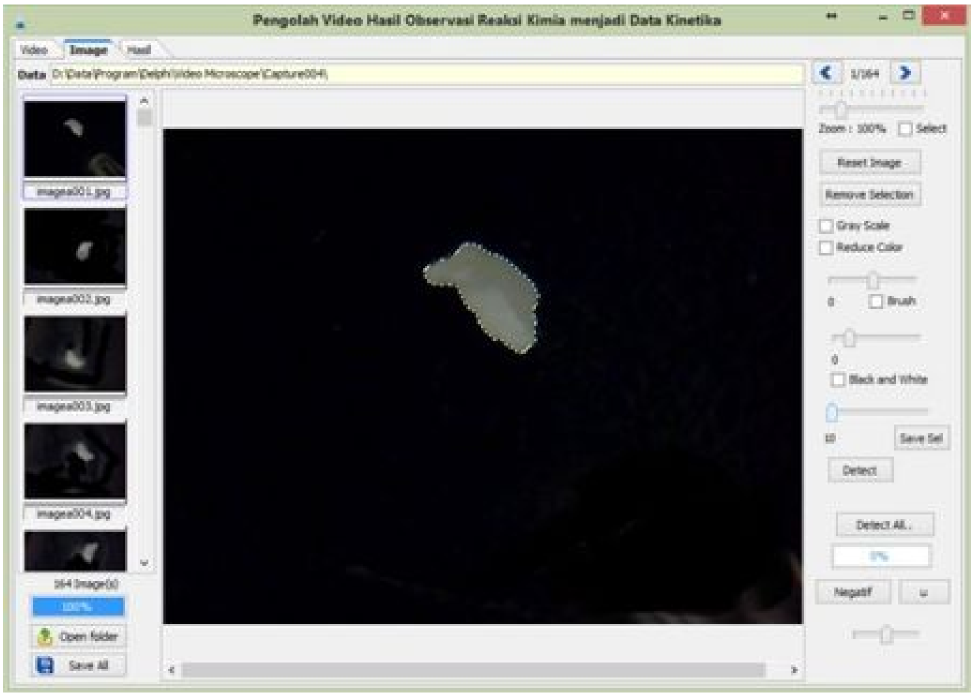

Fig 5. The application screen shot displaying a video image being played

$$\left(\frac{\Delta m_{MgO}}{\Delta t} = -\frac{\Delta A_{MgO}}{\Delta t}\right) \quad (1)$$

where: m_{MgO} = mass of MgO, A_{MgO} = area of images, and t = reaction time.

However, the accuracy of this method would really depend on the regularity of MgO shape. To improve accuracy, a more sophisticated assumption should be applied as in reality MgO has three dimensional regularities. This reflects the limitation of the method.

The Functionality of Data Processing Application for Observation Result

The main function of the built application is to extract the images from the video and subsequently calculate their area. An example of solid MgO and HNO_3 reaction image and video extracts being played is shown in the screen shot of the application in Fig. 5. The left hand side partition screen in Fig. 5, showed the extracted MgO particle images at several points of times. The results of this image extract were then processed into the reaction rate data namely the mass of MgO as a function of time. The result of the next processing can be displayed in a table and graphic forms. The obtained data series can also be exported for processing using a spread sheet program.

The sample of data processing results, image pixel, and estimated MgO mass, in the reaction experiment of 1.5 mg MgO with 2 M HNO_3 produced from the application is presented in Table 3.

Table 3. Examples of data processing result of solid-liquid reaction observation application

Reaction time (sec)	MgO image (pixel)	MgO mass estimated (mg)
0	8151	1.50
30	7657	1.41
100	5285	0.84
200	1688	0.31

These data were further presented as mass of MgO as a function of time as shown in Fig. 6. As can be seen in the figure, in both the concentration of 1 M and 2 M of HNO_3, the MgO mass decreased as a function of time. The general trend of depletion in several points might be due to the changing position of solid MgO against the microscope position. The shape of solid MgO which was not fully spherical made it possible for this phenomenon to take place.

Curve fitting of the results revealed an exponential trend line curve for the reaction of 1 M HNO_3 concentration, and a second order equation for reaction data with 2 M concentration. Even though factors affecting the trend were still unexplainable, the reaction rate of higher concentration was faster as indicated by the slope. Furthermore, the curves show the effect of concentration on reaction rate.

Implication to Chemistry Learning

The observation results of MgO reaction with HNO_3 can be used to give an overview about solid-liquid

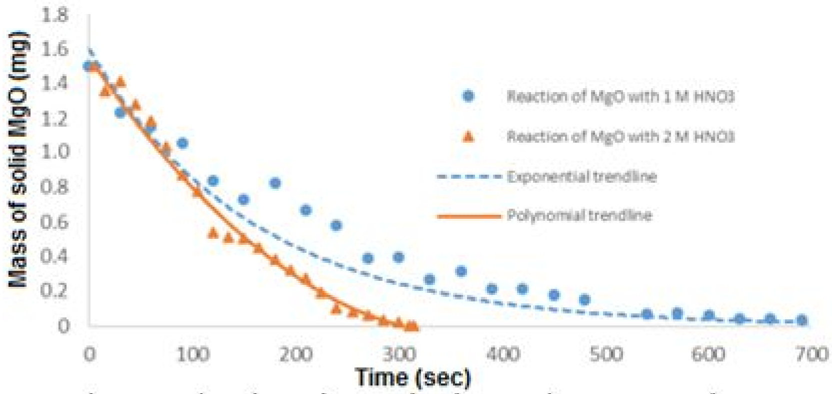

Fig 6. Plot of MgO mass against reaction time observed using a microscope and processed using the application with different concentration of HNO_3

reactions which are rarely used as examples in chemical kinetics teaching. Furthermore, the image of solid MgO at the various time intervals can be processed into data on the mass of MgO against time for further development of chemical kinetics concepts, namely the definition of reaction rates based on the MgO mass curve against time. The reaction video produced in this study can also be used to show the influence of concentration on reaction rates.

We noticed that the quality of data obtained from this equipment should be treated as educational purposes data and not as a "research quality" data. The students should be reminded that the calculated mass of the solid particle was just an estimation based on the image area, and not the actual mass. Furthermore, no account has been taken of changes in surface area of the particle. Therefore, the data, as used here, can only show the concept of reaction rate and a trend that reaction rate is influenced by reactant concentration. An example showing the limitation of this procedure can be seen when the order of reaction was calculated using the data; the order is one for 1 M HNO_3, and the order is two for 2 M HNO_3. For an advance research purpose, this method should be enhanced and validated for example by improving the assumptions.

The potential advantages of using this observation apparatus for learning chemistry are as follows: (1) the experimental data obtained can be digitally recorded and replayed, (2) a library of experimental data can be readily obtained, (3) the cost of the digital microscope is low and (4) the experimental data is processed using an easy to use application to produce kinetics information. The data has relevance to the students because they watch the reaction occur and they can relate to the origin of the data (it is not simulated). The experiment helps students directly link the observable (macroscopic) phenomena with the abstract (sub-microscopic and symbolic) theoretical concepts. This chemical representation can be considered as a science modeling skills as defined by [12-13]. In addition, a small quantity of chemicals is required for an experiment that can be observed by all students in the classroom.

To some extent, this reported research showed that the experimental procedure mimicked the activity of using an "environmental microscope" where a chemical reaction or a physical process can be observed *in-situ.* This type of experiment helps to link school chemistry with current technology and/or chemistry research experiments as recommended by [14] and shows how technology can be used and designed a lab experiment a resource as also done by [15-16].

To ensure that the developed approach is strongly learner focused we adapted the ASELL (Advancing Science by Enhancing Learning in the Laboratory) educational analysis (see http://www.asell.org/Publications/Document-Library) to document the intended learning outcomes. This educational analysis is part of an educational template first developed by Barrie and co-workers [17] and has been used, for example, by other group'[18]. In doing the experiment, the students will develop scientific and practical skills as defined by [19–21], in this case: (i) observing and recording a solid-liquid reaction (ii) using image processing software (iii) tabulating and graphing data and (iv) handling of acids safely. They will also develop thinking and generic skills in data analysis, critical thinking and report writing. Ultimately, the students will increase their theoretical and conceptual knowledge in the area of kinetics and rates of reactions. Specifically they will gain a better understanding that the rate of a reaction is a bulk property which, in this case, refers to the change in mass of the MgO with respect to time. By changing the parameters (concentration of acid, temperature and or particle size) they will also gain first hand experience of how these affect the rate of reaction.

For the purpose of educational practices, the approach might be extended to other possible chemistry learning activities. One of these might be that the procedure is used in an inquiry setting where the student are asked to do a chemical kinetics experiment in which they explore the impact of temperature, concentration and/or particle size on the reaction of MgO with HNO_3. Another option may be, instead of using the image processing software for analysis of area, the students can find the mass of MgO by cutting the printed image, weighing the paper followed by calculating the MgO mass. A variety a results will be obtained from every student, thus introducing the concept of sampling and measurement uncertainty. Other applications which may be used could include the dissolution process of soluble solids, precipitation and crystal growth. These learning activities might be implemented in an inquiry setting such as using Process Oriented Guided Inquiry Learning (POGIL) as described by [22].

CONCLUSION

A digital microscope is a simple piece of lab equipment that can be used to video record and demonstrate the rate of non-gas producing solid-liquid reactions such as magnesium oxide and nitric acid. The video processing application (available from the corresponding author) for chemical observation developed in this study is able to extract images of solid MgO at several time intervals that can further be used to explain the concepts, definition of reaction rates, and the influence of concentration on chemical reaction rates. The procedure has the potential to be used in an inquiry learning setting to explore students' creative thinking.

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