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# Teaching reaction kinetics with chemiluminescence

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## ARTICLE INFO

## Article history:

Received 25 August 2017

Received in revised form 4

December 2017

Accepted 6 December 2017

Available online 13 December 2017

## Keywords:

Reaction kinetics

Rate law

First-year undergraduate/general

Chemical engineering

Hands-on learning/manipulatives

Reaction engineering

## ABSTRACT

An experiment to aid the transition from secondary school chemistry to introductory chemical engineering in higher education is described. The phenomenon of chemiluminescence observed during the oxidation of luminol has been successfully employed to study the kinetics of the reaction. Using inexpensive light sensors the effects of temperature on rate of chemical reactions can easily be quantified through their associated kinetic parameters.

The experiment gives reproducible results and allows the measurement of the rate constants of the reaction and its order with respect to luminol at different temperatures in one three hour laboratory session. From these, the activation energy of the reaction can be determined. Experimental skill and supervisory requirements are minimal making the setup ideal for first year undergraduate or final stage secondary school students.

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## 1. Introduction

The transition from the final year of secondary education to the first year of higher education in chemical engineering can be difficult for students because their mind-set is rooted more in the pure sciences, i.e. physics, chemistry, mathematics and biology. To them, chemical engineering represents an eclectic mixture of these sources which need to be forged into a different paradigm of thought.

One of the crucial facets of this change is to think about chemical reactions from a scale-up standpoint. To enable industrial scale production, reaction chemistry must be studied and understood to arrive at a reaction model which mathematically represents the underpinning process.

It is critical that chemical engineers and chemists are trained in reaction kinetics to maximise yields of desired products, reduce unwanted by-products and ensure that systems operate safely (Campbell, 1963). The ultimate aim of a kinetic teaching experiment is to familiarise students with the process of acquiring experimental data to develop a mathematical model for the reaction kinetics by extracting key information such as rate constants and activation energies. This scientifically rigorous approach forms the basis of successful scale-up from the bench to industrial scale (Zlokarnik, 2006) and is one of the cornerstones of chemical engineering education.

In industry, kinetic data is most commonly determined by *in situ* real time monitoring of the reaction using analyt-

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<sup>1</sup> At the time practical research was carried out.

<https://doi.org/10.1016/j.ece.2017.12.001>

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ical techniques such as Fourier Transform Infra-Red (FT-IR) spectroscopy (Blackmond, 2005), ultraviolet-visible (UV/Vis) spectroscopy (Choquette et al., 2011), or nuclear magnetic resonance (NMR) spectroscopy (Mathew et al., 2006) to directly determine the concentration of the reaction components over the course of the reaction. These techniques are expensive and require a level of training which by far surpasses the level of first year undergraduate students. Less infrastructure-intensive analytical methods such as gas or liquid chromatography may be viable in a teaching environment but require samples to be taken from the reaction mixture, thereby disturbing its composition and resulting in less precise time resolution, especially if the reaction is rapid.

There are a variety of established ways to qualitatively and quantitatively demonstrate kinetics in a teaching laboratory. For example, in schools across the U.K. and many other parts of the world, kinetics of the “sulphur clock” reaction (Nyasulu and Barlag, 2010) are investigated using visual observation of the reaction mixture: sodium thiosulphate and hydrochloric acid are mixed at constant temperature in a conical flask placed on a paper marked with ‘X’. The time elapsed for generation of the product sulphur is estimated by visual observation. As sulphur is produced, the solution becomes less clear and the ‘X’ mark cannot be viewed through the solution. The experiment is repeated at different temperatures and reactant concentrations to study their effects on the rate of reaction. Despite being visually easy to follow and simple to perform, this experiment lacks quantification of the kinetic parameters. Another way of visually demonstrating and also quantifying a reaction has been reported by Mendes et al. (2004), where kinetic parameters of a saponification reaction were determined with the help of a pH sensitive dye, i.e. indigo carmine. While visually attractive, the colour change itself is not used for determining kinetics, and only qualitatively indicates the reaction’s progress. The analysis by conductivity requires calibration, which introduces a source of error in the kinetic parameters, particularly at high conversions. In addition, the timescale of the reaction only allows three experiments over a 10 °C temperature range in a single three hour session.

At an advanced level, the activation energy of hydrogen peroxide decomposition (Abramovitch et al., 2003) can be readily calculated by plotting temperature increase against time in a calorimeter. The drawback of this method is that it assumes negligible heat loss during the course of the reaction and instantaneous, perfect mixing, which is difficult to realize. Another, popular approach to follow reaction progress is to monitor the pressure of a gas phase reaction (Abramovitch et al., 2003; Nyasulu and Barlag, 2010). This requires specialized pressure vessels with the associated expense and safety considerations. Varying the oxygen concentration in solutions containing different amounts of methylene blue has been measured using oxygen sensing electrodes (Anderson et al., 2012; Delgado et al., 2014) to follow reactions but this method requires a lot of sample handling as well as the exclusive use of a photometer. Students can also be taught kinetics through computerised (da Silva et al., 2014) or partially computerised methods (Loyson, 2010). However, in these cases they do not develop the essential practical skills associated with lab work. For undergraduates and students in their final years of secondary education, there needs to be a balance between the complexity of the chemistry and associated instrumentation with the ease of conducting reproducible experiments which achieve a positive learning experience.

The phenomenon of chemiluminescence is very popular in teaching and outreach activities due to the striking visual effects (Cambrea et al., 2013; Kuntzleman et al., 2012) which capture the attention and imagination of the observers. Thus far, chemiluminescence demonstrations have been purely qualitative (Dean et al., 2011). However, with increased availability of affordable light sensors, it is possible to convert the qualitative data into quantitative, meaningful information by measuring lux values (Huntress et al., 1934; Seitz, 1975; White and Bursey, 1964; White et al., 1964). The system described in this study uses a light sensor connected to a commercial data logging software for measuring the chemiluminescence arising from the oxidation of luminol by sodium hypochlorite solution under basic conditions (Seitz, 1975). Most kinetics experiments reported in the literature involve measuring reactant or product concentrations over time. In contrast, the experiment developed in this study measures a rate dependent variable (the intensity of the evolved light) which is used as a proxy for the reaction rate. It allows for the determination of the order and the activation energy of the reaction, which reaches completion in a matter of seconds. This makes several repetitions at different conditions possible within a single three-hour lab session.

The experiment developed in this study enables introductory level chemical engineering and final stage secondary school students to quantitatively investigate reaction kinetics. The simplicity, safety and visual appeal of this experiment gives it an advantage over more advanced kinetic experiments.

## 2. Materials and methods

### 2.1. Chemicals

Luminol (3-aminophthalhydrazide) ( $\geq 98\%$ ) was purchased from Molekula, UK. Sodium hydroxide (AnalaR NORMAPUR pellets,  $\geq 98.5\%$ ), sodium hypochlorite solution (GPR RECTAPUR, 12%  $\text{Cl}_2$  in aqueous solution) was purchased from VWR, UK.

### 2.2. Equipment

A schematic of the setup is shown in Fig. 1(a), next to a photograph in Fig. 1(b). It consists of: a Vernier silicon photodiode light sensor (LS-BTA; the light sensor contains a Hamamatsu S1133 silicon photodiode, producing a voltage directly proportional to the intensity of light it is exposed to), Vernier ‘Logger Lite’ data collecting software (Vernier Software and Technology LLC, Beaverton, USA), installed on a personal computer, an IKA RW overhead stirrer (IKA Works Inc.) with a 45° pitched blade impeller, a 500 mL stainless steel flanged vessel with a flanged polytetrafluoroethylene (PTFE) lid.

### 2.3. Hazard and safety precautions

Chemicals used in this experiment are corrosive and can potentially be hazardous. Sodium hypochlorite is an irritant and can cause severe skin burns and eye damage if splashed. When in contact with acid, it can release chlorine, a toxic gas. Sodium hydroxide can also cause skin burns. Luminol is also an irritant and can cause skin and respiratory irritation. To reduce the likelihood and consequences of hazardous exposure, the concentrations of chemicals in the experiment are limited to the smallest possible amount. All students work-

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