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Short communication

A homogeneous chemical reactor analysis and design laboratory: The reaction kinetics of dye and bleach

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ABSTRACT

An experimental module for senior-level reaction engineering/reactor design students is described. The module is used to characterize the kinetics of dye (food coloring) neutralization by household bleach, and the reactor system is configurable for use in either batch reactor or continuous-stirred tank reactor (CSTR) modes. The reactor temperature, volume, reactant feed rates, and reactant concentrations may be adjusted to enable students to obtain a wide range of kinetic data. Dye concentrations in the reactor are monitored by absorbance spectroscopy, and the kinetic rate law is determined directly from the batch reactor performance data. Students use the completed kinetic rate law to compare experimental steady-state CSTR performance data to the mathematical models derived from reactor design equations. Finally, the students use the kinetic behavior of the system to design a hypothetical plug-flow reactor for the same chemical reaction and a set of stated operational goals.

1. Introduction

In recent years, the teaching faculty of the Chemical and Biological Engineering (CBE) department at the Colorado School of Mines (CSM) have endeavored to make a greater number of high quality, hands-on experiments available to undergraduate students enrolled in senior-level courses. The delivery of such laboratory modules are not always required alongside the lecture material normally covered in such courses, but the incorporation of a closely associated experimental project enhances the content in such a way that positive feedback is generated from both the students (who enjoy seeing instructional principles in action) and by those involved in the department's internal and external accreditation reviews. The reaction kinetics experiment described here may serve as a useful example for chemical engineering departments that are interested in adding a low-cost, and easily maintained, modular experiment to accompany undergraduate reactor design lectures.

One of the first published examples involving colorimetric methods for determining the kinetics of dye oxidation was the reaction of aqueous solutions of crystal violet and sodium hydroxide (Corsaro, 1964). The reaction of chemical dyes with commercial bleach to form colorless products (neutralizing or altering the dye's normal color) involves common household materials without any exceptional restrictions on handling or disposal. The fact that the reaction may occur visibly in an aqueous solution for a variety of dyes and food colorings has made it a very popular example for the instructors of chemistry and chemical engineering (Pickering and Heiler, 1987; Arce et al., 1998; Henary and Russell, 2007; Kalmatsky, 2013). Nearly every embodiment of the dye/bleach experiment, even

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those developed for chemical engineering students, involves simple batch reaction rather than reaction within a continuous reactor with feed and exit flows. One reason for this limitation is the difficulty in measuring dye or bleach concentrations (and thus reaction extent) in flowing streams, or within agitated reactors that have continuous reactant entrance and product exit flow streams.

Dye concentrations in the reaction solutions are conveniently measured by monitoring the absorption of the appropriate wavelengths of light passing through the solution both before and during reaction. For example, aqueous solutions of the food coloring FD&C Blue #1 (erioglaucine) absorbs visible light most strongly at about 628 nm. The absorption (or transmission) of this red-orange light through solutions of Blue #1 is typically measured using commercial visible light spectrophotometers, which are generally intended for use with samples contained in a glass or plastic cuvette. Further, optical scanning of the sample is typically carried out in a chamber that is shielded from external light. Light absorbance or transmittance may also be measured with a laser/photodiode combination for the wavelength of interest (Delgado et al., 1998), however this type of system introduces a potential hazard (laser radiation) and requires signal calibration, manipulation, and translation for the photodiode/media interface selected for data acquisition and recording.

In the present communication, a method for arranging a combination of inexpensive benchtop devices for measuring dye concentrations in batch and flow reactors is described. The model reactor designs are created for use with a visible light spectrophotometer, a fiber optic probe, and an electronic data logger. Students are provided with a choice of two food coloring dyes: FD&C Blue #1 (peak absorbance ~628 nm) and FD&C Red #40 (peak absorbance ~502 nm) for the experiment.

2. Experimental apparatus and operation methods

All dye and bleach solutions are prepared by laboratory teaching assistants prior to the start of student section laboratory sessions. The dye solutions are prepared from powdered dye sources (Sigma–Aldrich) and tap water. The bleach used is a concentrated commercial grade solution (Clorox Company; 8.25 wt% NaOCl), which is used both directly and in dilutions with tap water as required for the creation of reactor feed solutions.

The specific design features of the experimental apparatus for each reactor type (batch and CSTR) were developed over the course of three semesters of student use, leading to the final (unified) apparatus described here. A schematic illustrating the combined batch reactor/CSTR vessel is shown in Fig. 1.

The reactor body consists of a square fiberglass-reinforced plastic (FRP) tube section, closed at one end (the reactor bottom) by edge-joining a square aluminum (alloy 5052) plate with a two-part, waterproof epoxy adhesive (J-B Weld Company; JB-KWIK). Both the aluminum plate and FRP tube have edge lengths of 7.62 cm. The FRP tube section is approximately 20 cm in length, with a wall thickness of 6 mm. This creates a maximum functional reactor volume of approximately 750 mL. The aluminum plate has an approximate thickness of 3 mm. Aluminum alloy 5052 was chosen for its high thermal conductivity and for its generally high resistance to corrosion by bleach solutions.

The reactor was used above a heating/cooling plate and a magnetic stirring assembly. The heating/cooling plate consists of an aluminum (alloy 1100) base with an integrated copper fluid tube. The plate is approximately 13 mm in thickness, and is supplied with water from a circulating heater/chiller with digital temperature control. The reactors are agitated with a Teflon®-sheathed magnetic stir bar 5 cm in length. Agitation of the solution within the reactor assists in maintaining the temperature of the reactor contents (the FRP is an excellent thermal insulator), and also generates sufficient solution blending to maintain well-mixed conditions in the case of CSTR operation. The square cross section of the FRP tube simplifies the placement and gasket sealing of the tubing and optical ports, and also enables enhanced mixing versus a cylindrical geometry; the sharp corners naturally baffle the stirred reactor (Meyers et al., 2002), mixing the contents while reducing vortex formation in comparison to a cylindrical tank without baffling (Kresta et al., 2006).

A total of five ports exist in the reactor's sidewalls. Two of these are tubing connections for the dye and bleach feed solutions, located above the free liquid surface. The plastic feed lines connected to both of the feed ports each include a small (ca. 40 cm) upstream segment, designed with stainless steel tubing and compression fittings as a double-pipe heat exchanger. This segment also shares a fluid connection to the circulating heater/chiller which enables simultaneous control of the reactor and feed temperatures. Two aligned ports near the bottom of the reactor sides are fitted with watertight brass glass sights. These sights serve as the mounting points for fiber optic connections from both the halogen light source (Dolan-Jenner Industries; Fiber-Lite MI-152) and the fiber optic detector probe (Vernier Software & Technology; SVIS-FIBER). The final port, centered near the bottom of the front side of the reactor, serves as the drain tubing connection. This drain connection includes an isolation valve attached to a segment of flexible plastic tubing. The end of the flexible drain tubing may be fixed at various elevations with a ring stand clamp, enabling hydrostatic control of the reactor volume. Feed and drain connections are only used during CSTR operation, and the illumination and detection ports are used for every experimental run. Solution absorbance is measured by incorporating the fiber optic probe into the detection port of the miniature spectrophotometer (Vernier Software & Technology; SVIS-PL). All absorbance data is collected and recorded with a hand-held electronic logger (Vernier Software & Technology; LABQ2).

2.1. *Batch reactor operation*

When testing the reactor in batch mode, students first fill the reactor partway with tap water, covering the glass sights within. The spectrophotometer is set to monitor absorbance at the wavelength of interest (628 nm or 502 nm), and the intensity of the light source is manually adjusted so that the absorbance reported on the logger display is very nearly zero. This value is recorded as the dye-free absorbance reference, representing the absorbance of water and the fiber optic detection cable at the selected wavelength. At this point, students may add a small dose of commercial bleach solution to the water in order to observe that there is no absorbance caused by the bleach itself. The reactor is then drained.

Dye solutions are made available to the students at premixed concentrations (2–6 μM for FD&C Blue #1, and 20–60 μM for FD&C Red #40) in insulated 18 L dispensers. Typically, two different dye solution concentrations are available for each dye