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Microscale Implementation and Image Analysis of Fluid Processes

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Microscale fluidics offers new avenues for teaching CAD, prototyping, fluid mechanics, heat and mass transfer, process engineering, control, and image analysis. Miniaturized fluid systems are implemented in credit card-sized, clear plastic 'chips' that host a network of conduits, chambers, filters, manifolds, and flow control devices. The chips are connected to programmable syringe pumps. With colored and fluorescent dyes, images and videos of flow characteristics and mixing can be captured, processed, and analyzed with low-cost CCD cameras and along with image processing software (ImageJ or MATLAB®). Moreover, heats of mixing, heats of reaction, and convective, conductive, and radiative heat transfer in fluid systems can be analyzed using thermal image infrared (8-12 microns) cameras. Student projects include CAD of microfluidic chips, fabrication of chips using a CO2 laser cutter, 3D-printer, or CNC mill; experimental setup on a desktop with programmable syringe pumps, sensors, and CCD and thermal infrared camera, experimentation, and analysis of images. We study concurrent and countercurrent heat exchangers, various active and passive mixers, heats of mixing between alcohols and aqueous salt solutions, and acid-base neutralization reactions. These laboratory projects provide instructive and accessible hands-on experimentation, at levels ranging from intuitive and visual to more analytical treatments, in subject areas of fluid mechanics, heat transfer, reaction engineering, image processing and machine vision, engineering modeling, and rapid prototyping. We emphasize skills and concepts gained for their relevancy to energy efficiency, sustainability, and green manufacturing.

1. INTRODUCTION

Micro-scale experimentation can have substantial educational value in STEM curriculums as it provides highly visual, accessible demonstrations of fluid, thermal, and chemical phenomena. Further, students can design and prototype these experiments using commonly-available fabrication tools such 3d printers, laser and plotter cutters, and CNC mills. Moreover, these types of experiments allow a wide range of laboratory exercises with small demand on resources due to their compactness, inherent safety, low consumption of materials and generation of waste, and simple instrumentation. Finally, the experiments provide students with experience in 'lab on a chip' technology which is increasingly important is micro-scale chemistry, sensors, electrochemical processes, and medical diagnostics.

Process 'scale-down' and miniaturization are common themes in many fields of technology in order to reduce costs, increase safety, enable portability, and lower environmental impact by reducing waste, materials, and energy usage [1], thus fostering sustainability, "green manufacturing" and providing appropriate technologies for resource-limited areas of the world. Integrated circuits serve as the paradigm of miniaturization, but other areas ripe for similar scale-down include optics, sensors, and fluid-based technologies encompassing analytical chemistry, materials and chemical processing, biology, and medicine, the latter which fall under the broad category of microfluidics. For example, microfluidics enables miniaturization in medical diagnostics and biotechnology operations such as cell culture and separations of biomolecules, as well as small fuel cells to power portable electronics, sensors for environmental monitoring and chemical analysis and microscale

chemistry, (MEMS) (Micro ElectroMechanical Systems), micro-optics, and platforms for various nanotechnologies are also of increasing interest. Miniaturization can yield improved performance by exploiting effects that are prominent at the microscale such as reduced inertial effects, enhanced conductive heat transfer and utilizing various interfacial or surface energy-related phenomena such as capillary forces, and more options for controlling fluid and heat flow. Further, more efficient coupling with light and other radiation (e.g., for optical heating or detection) can be realized in such microsystems.

It is often problematic to instrument such systems with point sensors such as thermocouples, flow meters, and pressure gauges. On the other hand, imaging the microsystem during operation with (visible) CCD and (infrared) thermal cameras greatly aids analysis of microdevices [2]. Many microfluidic 'lab on a chip' applications have critical requirements for heating/cooling and temperature control [3-4]; various design features can be assessed with thermal imaging.

For STEM education, small-scale experimentation such as with microfluidics can alleviate demand on resources (lab space, consumables, safety and environmental compliances) and allow dissemination to institutions with constrained resources [5-8]. Various concepts in fluid mechanics, chemical reaction engineering, heat transfer, biotechnology, process modeling and process control can be demonstrated with microfluidic systems. Further, microsystems can include microcontrollers, rapid prototyping, CAD, finite element analysis sensors, image capture, processing and analysis, and including infrared imaging. Accordingly, microfluidics provides excellent case studies for teaching and integrating many engineering fields.

Microfluidic devices process small quantities (1 nl to 1 µl) of fluids in microscale (1 micron to 1 mm) structures. Typically, a credit-card sized plastic cartridge ('chip') hosts a microfluidic network of channels, conduits, chambers, filters, and flow control components [9]. Relative to traditional macroscale systems, 'lab on a chip' systems yield noteworthy advantages including more precise control of reactants faster reaction time, lower consumption of reagents, convenient disposal, effective containment of infectious agents or hazardous substances, portability, and compactness. Lab-on-a-chip applications such as polymerase chain reactions (PCR) to amplify nucleic acids, as well as cell cultures, need closely regulated heating and cooling with temperature control (often ± 0.5 °C) and fast thermal response times (> 5 °C/s) [4]. For such applications, infrared thermal cameras offer non-contact measurement of temperatures and two-dimensional temperature profiles of microfluidic systems.

Here, we describe thermal imaging of microfluidic systems with commercial infrared cameras (costing $1000 to $10,000) demonstrating that such thermal cameras provide a new level of analysis for such systems, facilitating more precise thermal management and temperature control. For STEM education, demonstrations of various thermal-fluid processes offer a new window on fluid flow and thermal phenomena. These tools are useful for applications of microfluidics to medical diagnostics applications, and more generally for microscale heat transfer and mixing and reaction engineering. Such thermal imaging has great instructional value [10,11]. In particular, microfluidics with thermal imaging allows visualization of combined heat transfer and fluid flow.

2. CHIP FABRICATION, INSTRUMENTATION, AND OPERATION

For these studies, microfluidic chips (35 x 55 mm in area) are fabricated as bonded laminate structures [12] of three acrylic (PMMA) sheets (McMaster Carr, Elmhurst, IL). A microfluidic "circuit" is laid out with computer-aided design (CAD) software (SolidWorks™ or AutoCAD™). The CAD file programms a 30-W $CO2$ laser cutter (VLS 3.5 Universal Laser Systems, Scotsdale, AZ) to pattern an acrylic sheet (1.6-mm thick) with through-cuts defining the microfluidic circuit [13]. This patterned sheet serves as the middle layer of the chip. The channels are enclosed with a top and bottom acrylic sheets (0.25 mm thick) by using one of: 1) acrylic cement (TAP Plastics, Dublin, CA), 2) solvent bonding (acetonitrile), 3) double-sided tape (3M, St Paul, MN), or 4) thermal pressure bonding in a hydraulic hydrostatic press (Model 3851 Laboratory Press, Carver, Inc., Wabash, IN). The inlet and outlet ports of the chip were coupled to small-diameter (1.2 mm) teflon tubing by adhesive bonding and connected to syringes (0.5 ml to 5 ml) mounted on a programmable syringe pump (Model Fusion 200, Chemyx, Stafford, TX). In some cases, the liquids were preheated before entering the chip. A Peltier thermoelectric module (15 x 15 x 3 mm, 3 amps, 6.5 Watts, Digi-Key, Thief River, MN) under the chip channel is used for heating/cooling the fluids in chip channels. Some representative chips are shown in Figure 1. Flow rates range from 50 microliters/min to 5 ml/min.

Acrylic has many favorable attributes for microfluidic chips, but it is neither a good thermal conductor nor a good thermal insulator (heat capacity = 1.4 J/g·°C, thermal conductivity = 0.2 W/m·°C), thus, depending on the application, provides neither sufficient heat transfer or good thermal isolation. To accurately measure the temperature of fluids in underlying channels by a thermal camera, it is necessary to keep the top capping layer as thin as possible, and correct for the emissivity of the plastic. Emissivity corrections are relatively easy to incorporate with current thermal cameras. Although transparency of the chip is not required for thermal imaging, simultaneous visual CCD imaging of flow is often useful [14]. Flow visualization can be improved by adding food coloring or fluorescent dyes to the liquids (see Figure **1**).

Mixing in microfluidics is difficult since flow is laminar with Reynolds Numbers of < 10. We developed magnetic stirrers for microfluidics. For example, a 2-mm diameter circular chamber is formed at the junction of two channels and a common exit channel. A 1.5-mm long steel wire is placed in the chamber during the assembly and bonding of the chip. The chip is placed on a magnetic stirrer plate and the wire stirring rod is rotated at 500 to 1200 rpm to vigorously stir the fluids at their point of contact.

We use a FLIR T-360 IR camera (FLIR, Danderyd, Sweden) for the thermal imaging experiments, with the following specifications: detector type: Focal Plane Array uncooled microbolometer with 320 x 240 pixel resolution; spectral range: 7.6 to 13 μm; 26° x 19° field of view, minimum focus distance 0.4 m; and thermal sensitivity (NETD) 0.06 °C @ 30 °C. The IR camera also has a 1280 x 1024 pixel (visible light) CCD camera. Thermal and visible images are stored in a JPEG format for later analysis such as with the Image Processing utilities of MATLAB™ or ImageJ (public domain, Java-based image analysis software). These chip geometries and flow implementations, along with the thermal images, are conducive to finite element modeling.

Representative desktop experimental setups are shown in Figure **4**.   The costs for this system are relatively modest: IR camera (~$10,000), programmable syringe pump (~$1000), consumable materials (< $200). The chips can also be made in other materials (e.g., polycarbonate, polystyrene, cyclo olefin copolymer (COC), or PDMS) and by other means (CNC, soft lithography, desktop engraving, plotter). Also, thermal imaging camera prices continues to fall, and the latest generation of cellphone-based thermal cameras (e.g., Seek™ and Therma-App™) range in price from $250 to $2000.
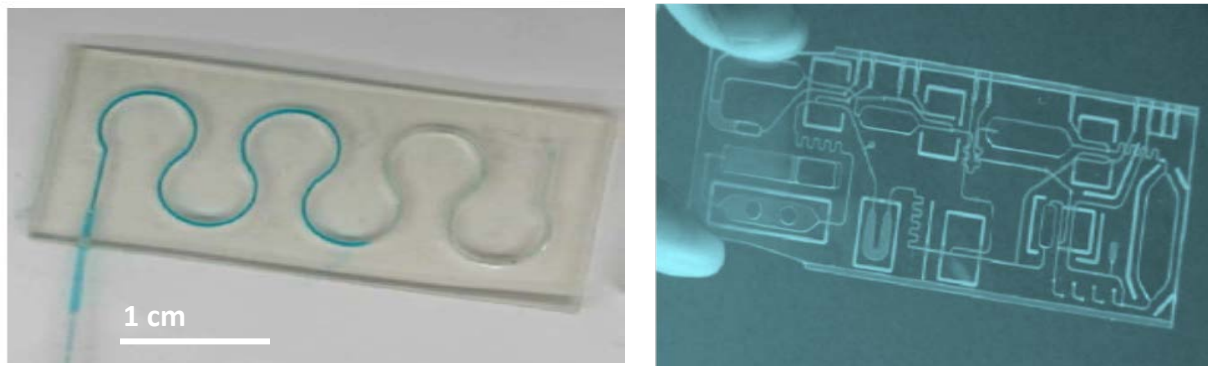
Figure 1. (left) simple chip with single serpentine channel; (right) chip with more elaborate fluidic circuit.
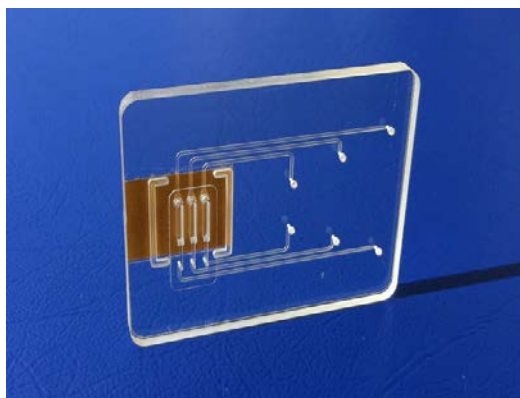
Figure 2: Chip with three parallel 20 microliter   reactors.
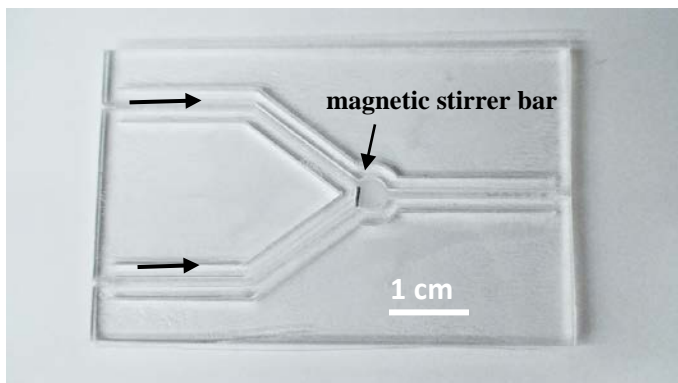
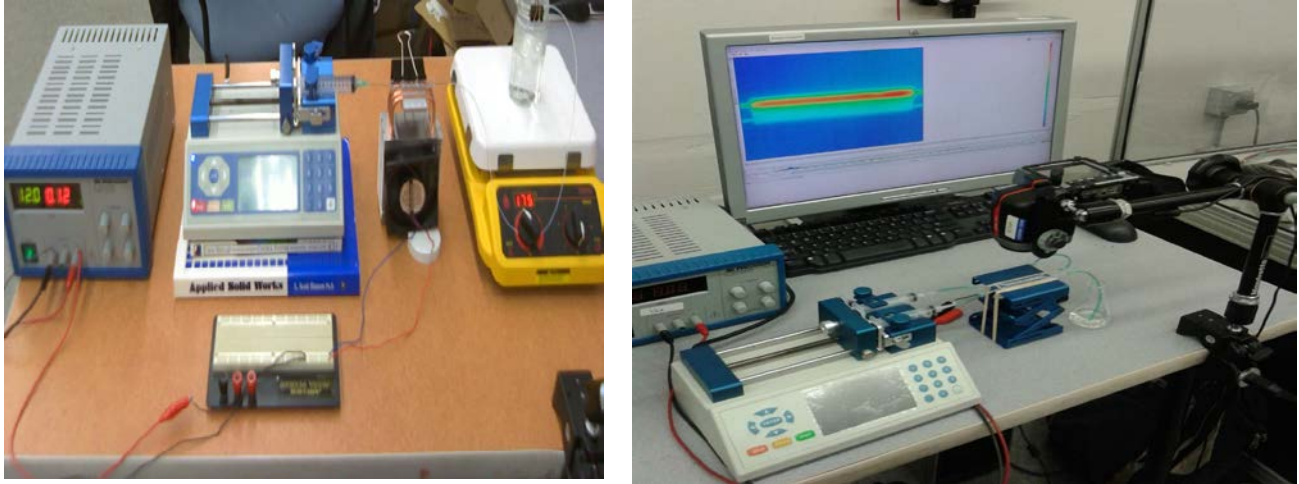**Figure 3:** Y Junction Chip with magnetic stirrer at junction.

Figure 4. Desktop and tabletop setups for thermal imaging of microfluidics experiments.

Infrared cameras can readily assess the uniformity of heated surfaces and spatial temperature variation in microfluidic chips.   For example, Figure **5** shows a top view of a stage heated by chemical heating [15]. Similar evaluations of Peltier and resistive heating elements.
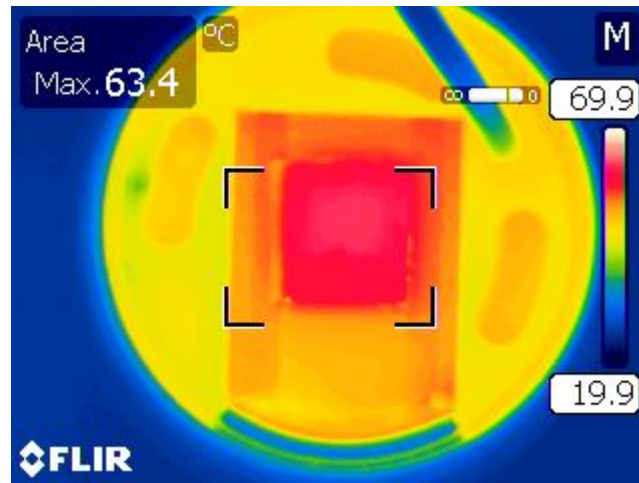

Figure 5. Surface temperature profile of heated surface for microfluidic chip.
Heated area is approx. 1 cm x 1 cm.

3. HEATING AND COOLING OF FLOW IN MICROCHANNELS AND MICROFLUIDIC HEAT EXCHANGERS

Microfluidic heat exchangers comprised of two parallel channels were fabricated. Water streams of different temperatures and flow rates are pumped through the channels, either concurrent or countercurrent. The streams are characterized by the inlet and outlet temperatures and flow rates. Figure **6** shows the temperature of the chip between the cannels as a function of flow rate. A difference of approximately 4 °C is observed for the range of flow rates used. Figure **7** shows a temperature gradient in the plane of the heat exchanger chip: with temperatures measured at 1 mm intervals. Evidently, efficient heat transfer is difficult to achieve due to the 1) laminar flow and 2) the relatively poor thermal conductivity of the chip material.
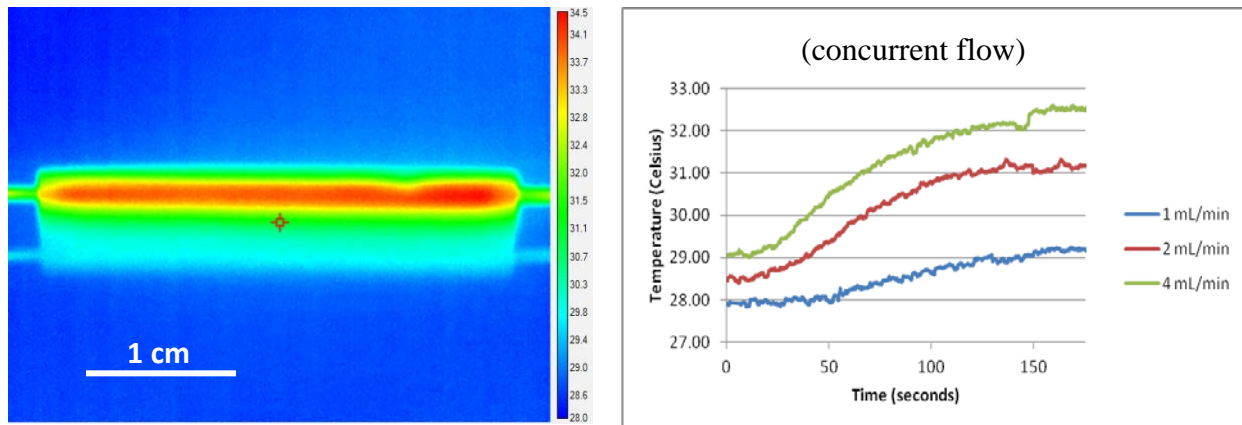

Figure 6. (left) Thermal image of heat exchanger chip, cursor shows temperature measurement point.
(right) Temperature at cursor point as a function of flow rate and time of flow in heat exchanger channels.
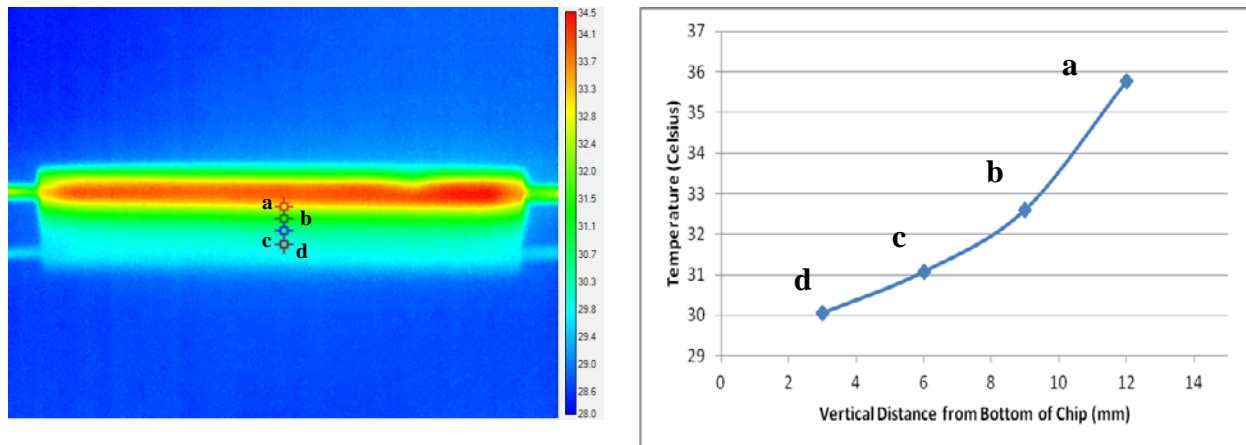

Figure 7. (left) Lateral temperature gradient due to hot water flowing in channel. Temperature at different points from channel (right).

In a second set of experiments, chips with a long channel traversing the length of the chip are placed directly over a Peltier module to heat or cool water flowing in the channel. Figure **8** shows the temperature variation along a serpentine channel heated by the thermoelectric module with experimental setup on left, and thermal image on right. Figure **9** is time-lapse thermal images of

starting from powering the thermoelectric at time $t = 0$ seconds. The advancing fluid can be halted upon freezing in the channel with sufficient Peltier cooling. This is the basis of so-called 'phase change' or 'ice' valves [16].
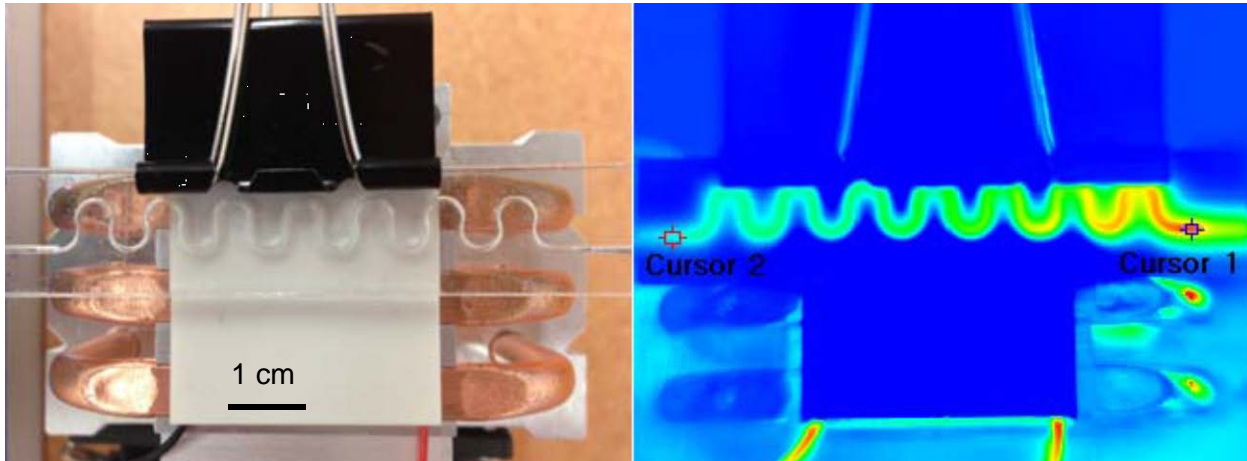

Figure 8. Thermoelectric heating of water flow in serpentine channel, flow rate = 1 ml/min: (right) top plan view of chip; (left) thermal image of flow.
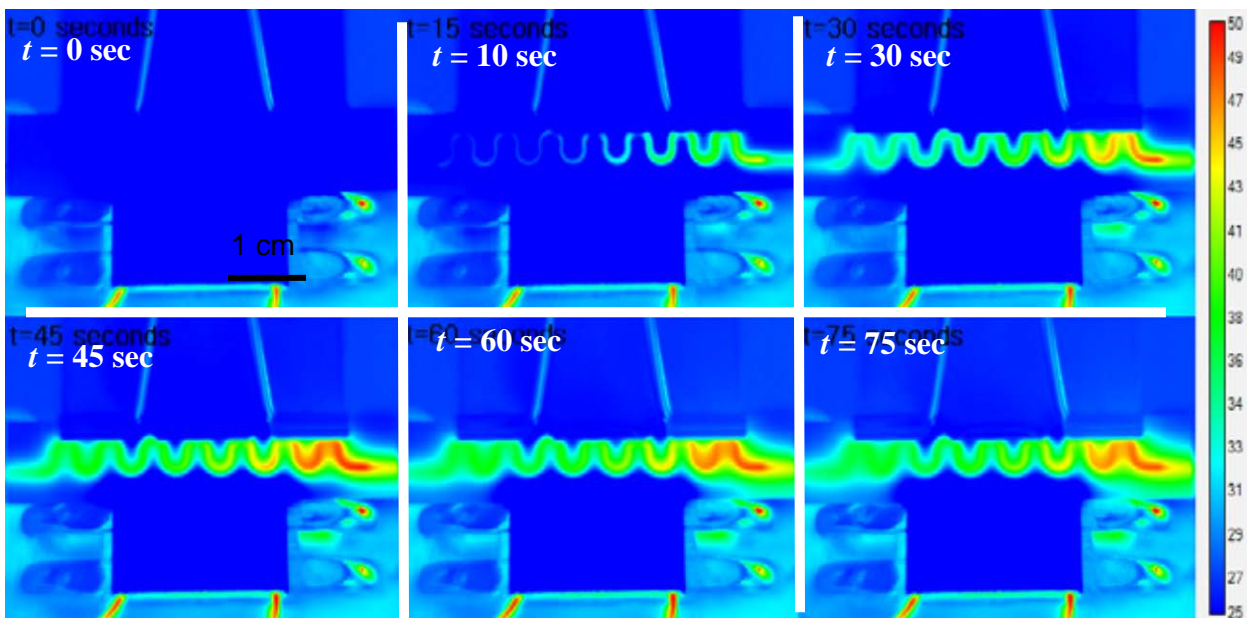

Figure 9. Time-lapse thermal images of Peltier heating of fluid flow in serpentine channel as in Figure 8.

4. MIXING OF HOT AND COLD WATER IN MICROCHANNELS

Warm and cold water pumped into each of the inlets of the fluidic chip by a syringe pump is mixed at the outlet (Figure **10a**).   Figure **10b** shows the temperature plot for this mixing. The top and bottom panels show the mixing *with* and *without* stirring. The resulting temperature was found to be average of the temperatures of the two inlet channels.   The effect of stirring was found to be negligible based on similar temperature profiles, and resultant outlet channel temperatures. This shows that magnetic stirring does not introduce significant energy into the system to heat the fluid, but rather just enhances mixing between the laminar streams. This observation is helpful in concluding that outlet channel temperatures in the following mixing experiments are the result of better mixing of inlet streams due to magnetic stirring, rather than extraneous heating due to magnetic stirring.
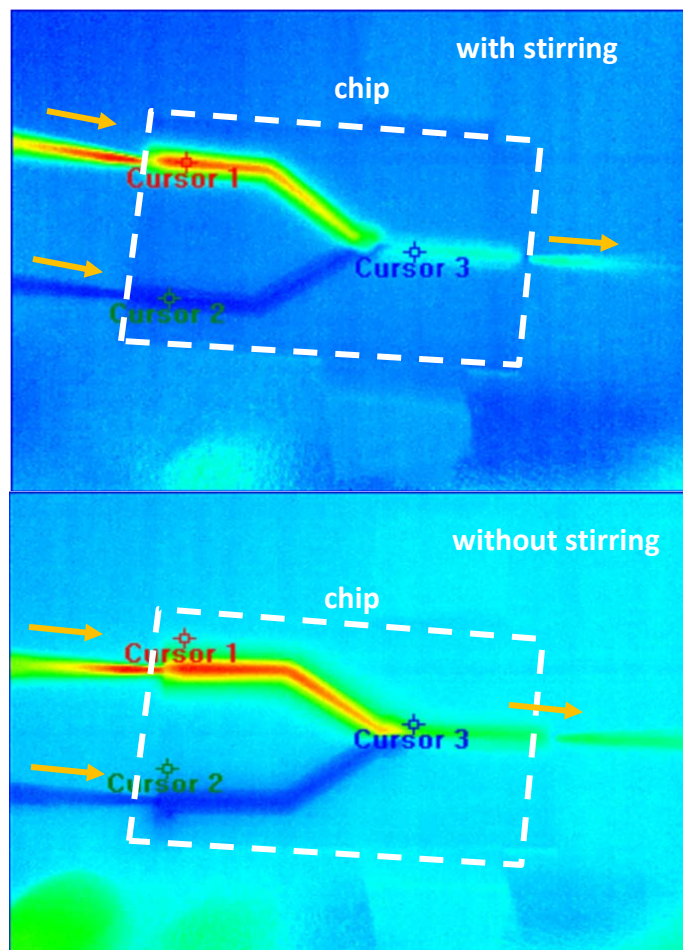

Figure 10a. Mixing of cold and hot water in Y junction of chip. (top plan view of chip). Top: with magnetic stirring (85 seconds after streams meet at Y junction), Bottom: without stirring (90 seconds after streams meet at Y junction.

5. MIXING OF ETHANOL AND WATER IN MICROCHANNELS

Figure 11 shows thermal images of exothermic mixing of ethanol and water on the chip. Pure ethanol in channel **1** and dyed water in channel **2** were used to observe the exothermic reaction. Unfortunately, pure ethanol resulted in delamination of the top layer from the base layer, as acrylic is not chemically compatible with pure ethanol. However, there was not a substantial difference in the temperatures measured at channel **3** between pure ethanol and 1:1 ethanol:water case in the absence of any stirring. Diluted ethanol (50% water: 50% ethanol) did not result in any visible degradation of the chip. Therefore, a 1:1 ethanol : water was used to demonstrate exothermic mixing between alcohol and water. The fluid in channel **1** (red) is a 1:1 mixture of pure ethanol and water, channel **2** (blue) contained water and channel **3** (brown) is the mixed liquid. At time, t_0, the liquid in channels **1** and **2** made contact at the junction, and after 10 to 12 seconds the thermal camera indicated a temperature rise at t_1. We surmised this delay is due to the time for heat transfer from the liquid to the surface of the chip. In time period **A**, the flow rate in the feed channels of **1** and **2** was 100 µl/min, and that in channel **3** (combining the ethanol and water streams in channels **1** and **2**) was twice of that level. In this case, the magnetic stir bar rotated at 500 rpm. At around 150 s, the flow rates of the syringe pump (channels **1** and **2**) were increased to 200 µl/min with the magnetic stirrer rpm rate unchanged, and this was maintained until $t = 190$ s, represented by time period **B** in the plot. Time period **C** in the plot represents the conditions starting from t = 190 s, where the flow rate is unchanged from region **B**, at 200 µl/min, but magnetic stirrer rotation rate is increased to 1200 rpm. Reynolds number for the streams in these experiments ranged from about 2 to 5, which implies laminar flow in the absence of active magnetic stirring.
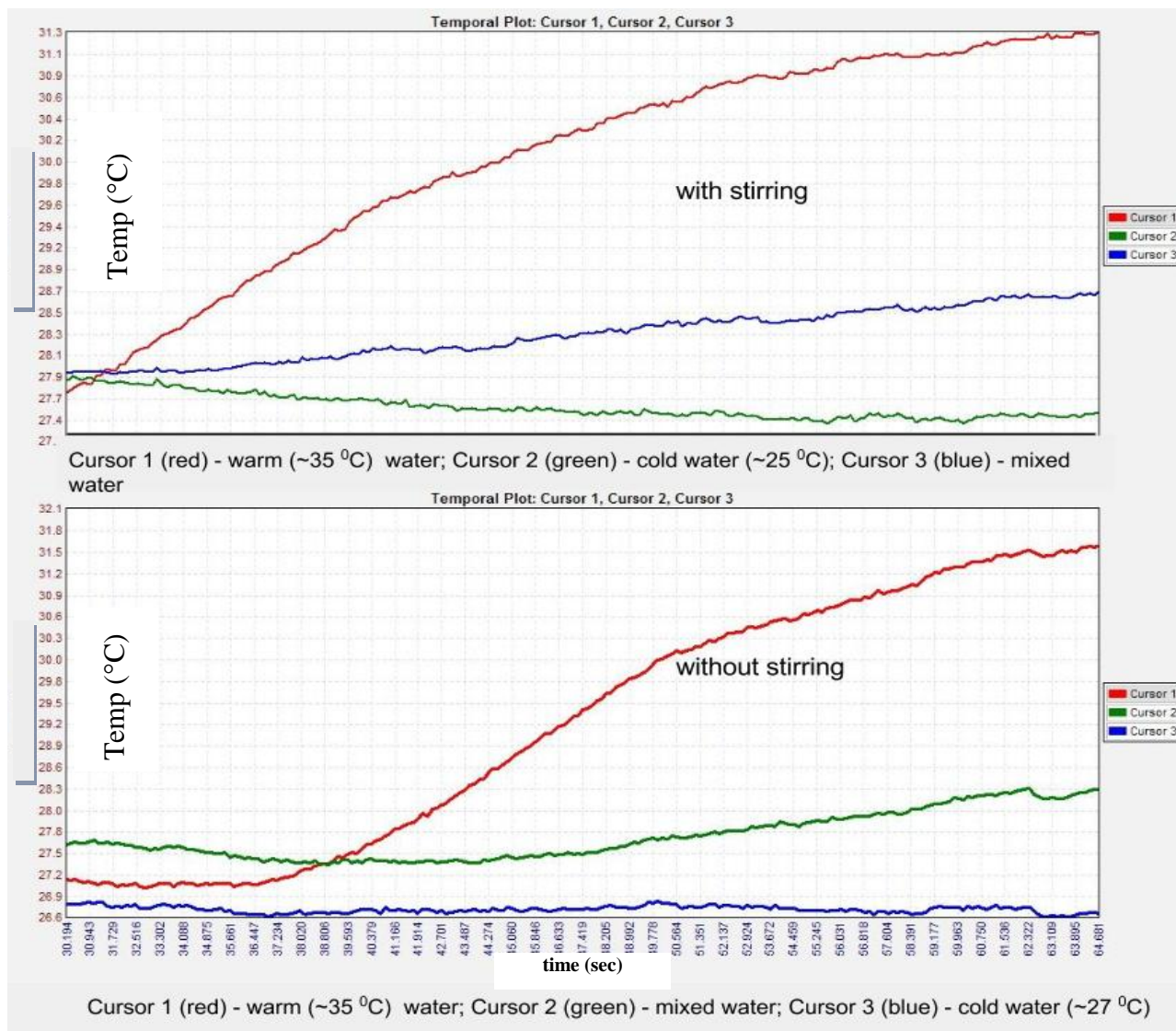
Figure 10b. Temperature measurements of mixing of cold and hot water in microfluidic chip as in Figure 10a, with (top) and without (bottom) magnetic stirring.) as a function of time from streams meeting at Y junction.

The reduction of the temperatures stream combining ethanol and water) temperature from periods **A** to **B** indicates lower overall enthalpy of mixing due to lesser degree of mixing. This appears reasonable because the time required for the fluids to mix, due to increased flow rate, is larger. However, since the distance from the junction to the outlet is still constant, this results in reduced mixing and thus, lower measured temperature in channel **3**. Going from region **A** to **B**, the stirring rpm remains unchanged. From time period **B** to **C**, the stirring rate (rpm) is almost doubled, while maintaining constant flow rate. The measured temperature in channel **3** increases, owing to increased stirring. This increased temperature indicates the enhancement of mixing on stirring. Moreover, the temperature measurements in channel **3** (brown) follow those measured at the junction (green) consistently indicating that there is negligible heat transfer along channel **3**, i.e., most of the mixing and evolution of heat of neutralization occurs near the junction.
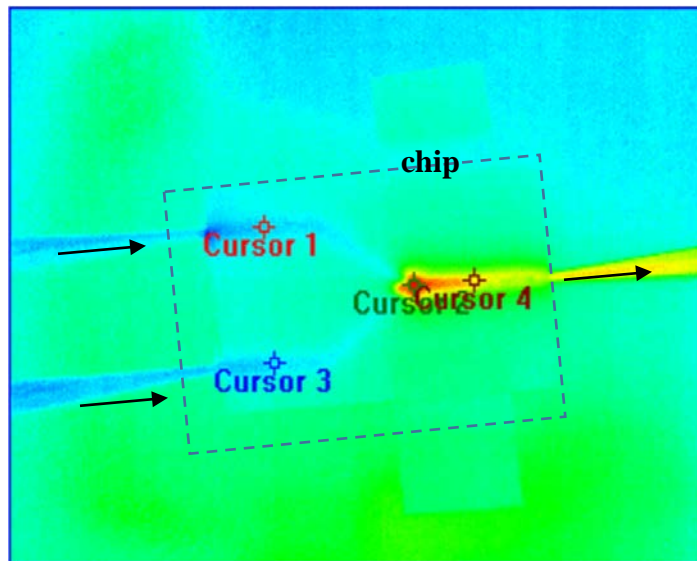
Figure 11a. Mixing of ethanol and water in microfluidic chip (top plan view of chip), thermal image at 230 seconds after streams meet.
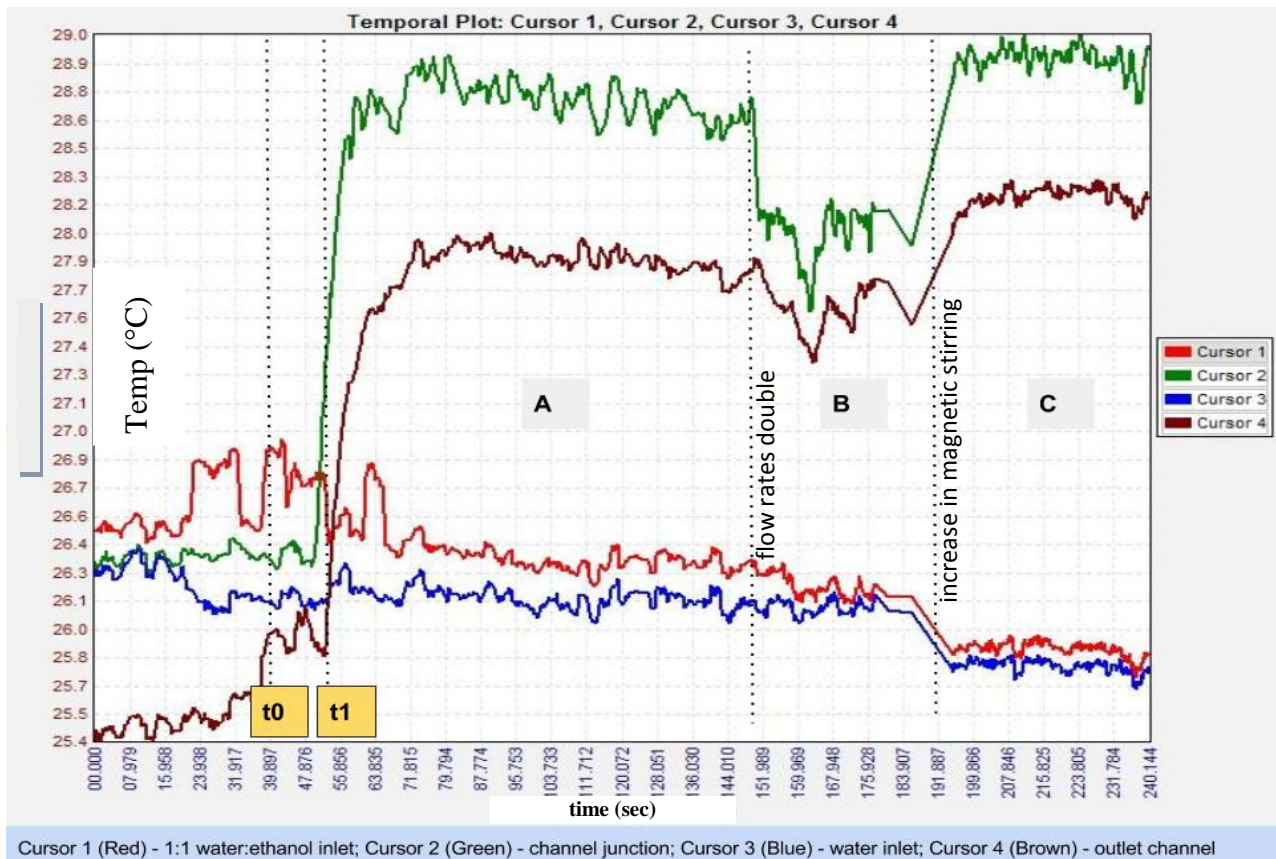

Cursor 1 (Red) - 1:1 water:ethanol inlet; Cursor 2 (Green) - channel junction; Cursor 3 (Blue) - water inlet; Cursor 4 (Brown) - outlet channel

Figure 11b. Temperatures in ethanol : water mixing at Y junction of chip measured at four points as per **Figure 11a**. t_0 = streams meet, t_1 = IR camera measures rise in temperature. Flow rates change between A, B, and C. (see text for details).

6. NEUTRALIZATION OF ACID AND BASE IN MICROCHANNELS

The temperature plot in Figure **12** was obtained from mixing an acid and a base in the mixing chip using the experimental setup described earlier. Hydrochloric acid and sodium hydroxide were selected as the acid-base pair because acrylic is chemically resistant to these reagents and the heat of neutralization between this strong acid and base is comparatively high.   Cursor **1** (red) corresponds to the temperature in channel **1**, in which 20% (v/v) hydrochloric acid (HCl) has been injected from one of the syringes. Likewise, cursor **2** (green) corresponds to the temperature in channel **2** for the stream of aqueous NaOH (~0.6 M). Cursor **3** (blue) shows the temperature of the outlet channel in which the acid and base react, releasing the heat of neutralization. In region **A**, the flow rates in channels **1** and **2** are 100 µl/min, and in region **B** of the plot, the respective flow rates are 200 µl/min. There was no stirring in this experiment. At twice the flow rate, the temperature in outlet channel **3** decreases indicating lesser time for chemical reaction to occur between the two laminar streams, as was expected. In the trial experiment, with conc. HCl (35%), the heat released was much higher and the temperature difference between inlet and outlet streams was greater than 5 °C. For this reason, and for safer handling, 20% HCl was used.
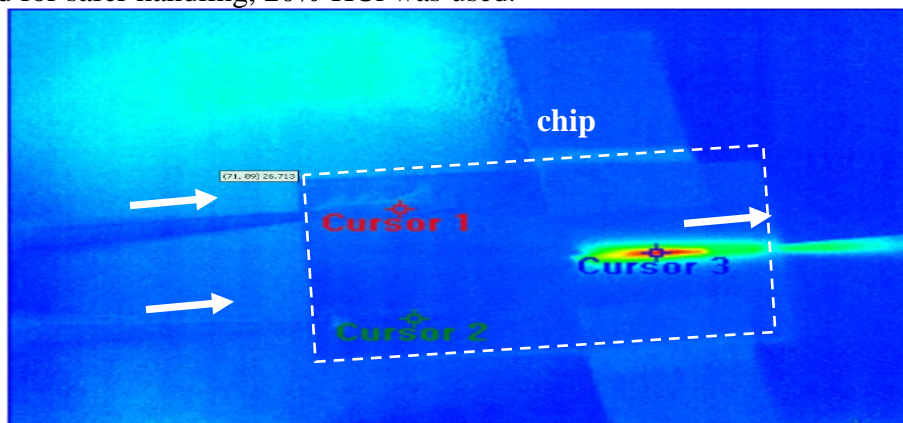

Figure 12a. Neutralization of HCl and NaOH solutions in Y junction chip. (top plan view of chip). Thermal image at 87 seconds after streams meet.
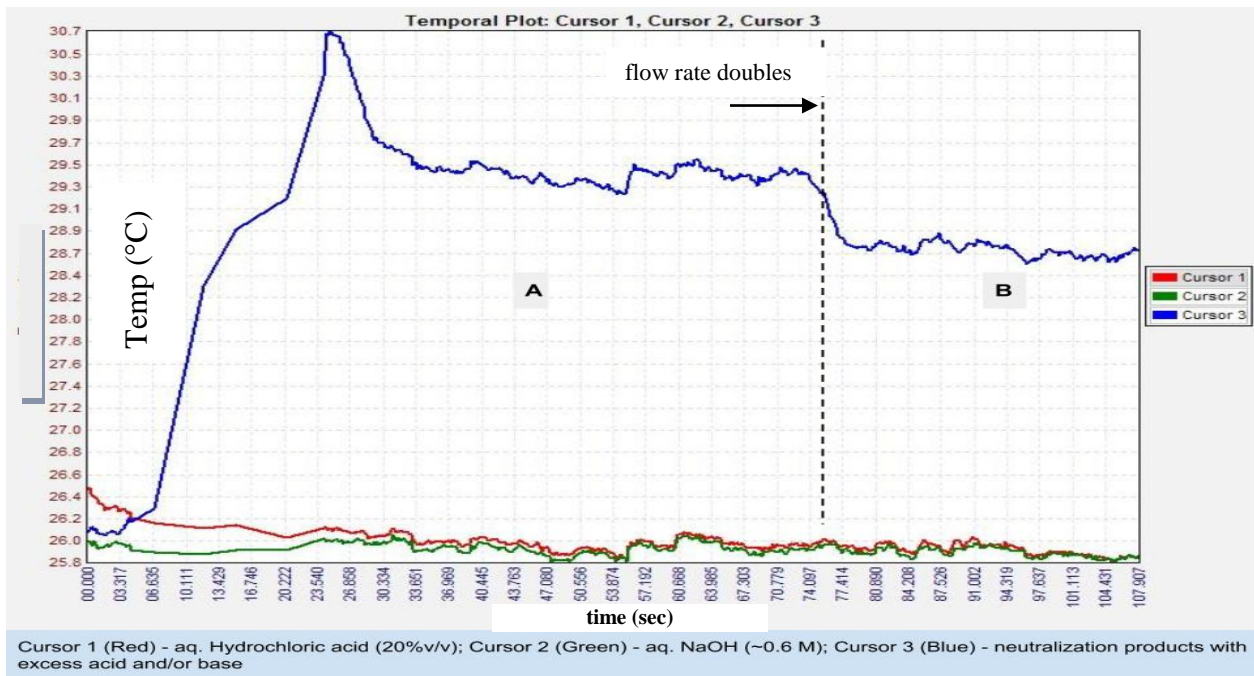
Figure 12b. Temperatures measured in neutralization of acid and base at Y junction of chip, as in Figure 12a.

7. DISCUSSION

We have developed a series of educational experiments on thermal imaging of simple microfluidic-based flow processes. The chips are simple to design, fabricate, and instrument and are typical with respect to materials (acrylic), dimensions (~0.5 mm channel widths) and operations (e.g., syringe pump fluid actuation, thermoelectric heating/cooling with small Peltier modules). The chips are representative of those used for medical diagnostics, microscale chemistry, and microfuel cells. Further, these microfluidic chips can be rapid prototyped with a CO_2 laser cutter, combined with adhesive, solvent, or thermal bonding. CCD camera imaging of fluid travel through the channels of the chip can be used to characterize flow rates (flow profiles and flow rates), as well as mixing of streams that are colored with dyes. Thermal imaging adds a new dimension in analysis of microscale phenomena in chips. Heat exchangers comprised of closely-spaced parallel channels (with either concurrent or countercurrent flow) can be evaluated quantitatively. Thermal mixing at Y junction can also be evaluated. In addition, three thermal mixing experiments were studied: mixing of hot and cold water, mixing of ethanol and water, and mixing of an acid and base. In the latter two cases, the heat of mixing and the heat of acid-base neutralization reaction could be observed and quantified, and a magnetic stirrer at the junction was added to improve mixing. These experiments provide representative examples of instructive microscale phenomena for heat transfer, fluid mechanics, and process engineering courses in the engineering curriculum. The ability to visualize thermal and flow effects complements analysis techniques. Further, these experiments highlight important design issues in microscale systems.

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