Use of Stereolithographic 3D Printing for Fabrication of Micro and Millifluidic Devices for Undergraduate Chemical Engineering Studies

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Abstract

Undergraduate STEM student performance is greatly benefited by supplementary, hands-on laboratory experience. Micro and millifluidic devices provide a multitude of opportunities for interactive study of concepts and phenomena encountered in nearly every field of engineering, as well as in chemistry, biology, and other disciplines. However, due to the cost and difficulty of standard micro and millifluidic device fabrication methods, many undergraduate students do not have access to these versatile educational tools. Fortunately, 3D printing offers an inexpensive and simple solution to this issue. This work aims to demonstrate the capability of stereolithographic 3D printing for the fabrication of micro and millifluidic devices for future use in undergraduate engineering studies at the University of Tennessee at Chattanooga (UTC); the ultimate goal of the work is to enhance student academic performance though the active study of concepts encountered in courses. A secondary goal is the enhancement of related undergraduate research projects.

Keywords

Microfluidics, millifluidics, 3D printing, fluid mixing, droplet generation

Introduction

Micro and millifluidic devices have been applied in a variety of areas including pH sensing[1], particle separation[2], fluid mixing[3], chemical synthesis[4], and droplet generation[5]; as such, these devices offer abundant opportunities for education.[6] Microfluidics have typically been fabricated from a variety of materials using photolithography, soft lithography, and milling techniques; however, many of these fabrication processes are expensive or limited in their application.[7, 8] One form of microfluidic device fabrication that offers cheap, rapid production, along with the ability to produce more complex 3D features is 3D printing.[9, 10] Due to the increasing commercial availability of 3D printers, many researchers and educators are exploring the possibility of producing microfluidic devices for a variety of applications.[3, 11, 12] Both fused filament fabrication (FFF) and stereolithography (SLA), two of the most common 3D printing types, have been used for fabrication of microfluidic devices, but the viability of both printing types is dependent on the design and purpose of the devices being produced. SLA is, in general, capable of producing smooth, small channels, yet is limited to only UV curable resins or photopolymers as a printing material. FFF cannot typically produce channels as smooth or small as SLA, but is compatible with a wider range of printing materials.[10] The work presented in this paper details the fabrication and analysis of various micro and millifluidic devices produced with a Formlabs Form1+ SLA printer, the initial results obtained through the use of the printed devices, and future plans for the continued integration of this technology into courses and undergraduate research at UTC. The student learning objectives (SLOs) for this project – and future implementation of its products – are aligned with the Accreditation Board for Engineering and Technology's SLOs[13] to: 1) "develop and conduct appropriate experimentation, analyze and interpret data, and use engineering judgement to draw conclusions," and 2) "acquire and apply new knowledge as needed, using appropriate learning strategies." Initial experiments with these devices were completed by a single undergraduate student and were intended to demonstrate 3D printing capabilities and potential for utilization of the printed devices in studies and demonstrations related to fluid mixing, biodiesel production, and droplet generation. These applications were chosen for their relatability to topics covered in various engineering courses and potential for use in research at UTC, as detailed below.

Fluid mixing: Due to low fluid flow velocity and the hydraulic diameter seen in micro and millifluidic devices, laminar flow and limited fluid mixing can often be easily observed within micro and millifluidic channels. However, fluid mixing can be encouraged through the addition of various features to the channels of the devices, among other methods.[3] Demonstration of both laminar flow and mixing, topics often discussed in fluid mechanics engineering courses, can be a useful, hands-on supplement to standard undergraduate coursework.

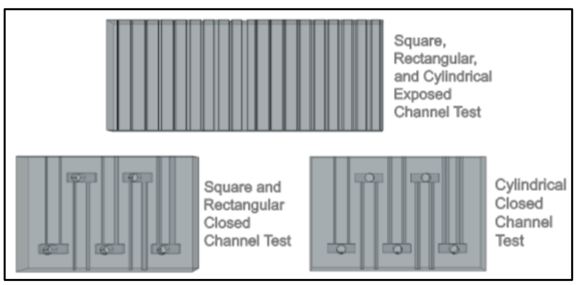
Biodiesel production: Previous researchers and educators have also studied biodiesel production in microfluidic devices; in comparison to standard batch reactors, microfluidic devices are able to significantly reduce the processing time of biodiesel, per unit volume, from hours to minutes. This reduction in processing time is mainly attributed to the devices' high surface area to volume ratio and increased mass and heat transfer rate.[14-17] Compared to petroleum diesel, biodiesel produces less carbon monoxide, sulfur dioxide, and unburned hydrocarbons, and because of its similarity to petroleum diesel, it is a worthy alternative and additive for use in many of the areas where diesel is currently predominately used, including transportation.[16] Because of this, research into both traditional and non-traditional biodiesel production methods at various scales is highly applicable for chemical engineering students entering industry focused on alternative energy and fuels.

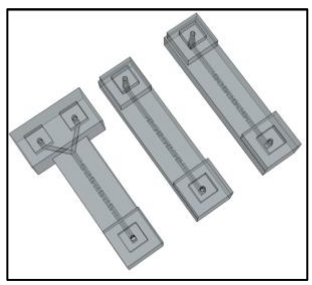
Droplet generation: Microfluidics further enable droplet generation for numerous applications; for example, water in oil droplets can be used to encapsulate cells or create emulsions.[12, 18, 19] Droplet sizes can be influenced by a variety of factors including device design and flow rate, both of which can be easily studied and manipulated by students.[20] Furthermore, the ability to encapsulate cells could enhance current bioengineering research being conducted at UTC.

Methodology

Device design and fabrication: AutoCAD 2018 for Mac software was used for all modelling in this study, and Formlabs PreForm software was used to interface with the printer. A Formlabs Form 1+ printer and Formlabs High Temp Resin (a clear photopolymer) were used to fabricate all devices at a resolution of 50 microns. After printing, all parts were agitated in isopropyl alcohol (IPA) for two minutes, followed by three minutes of undisturbed soaking in IPA; this wash process was repeated once more in fresh IPA. For devices containing channels, pressurized air was used to force unpolymerized resin from the channels. After clearing the channel with pressurized air, IPA was pumped through the channels to remove any remaining resin. All parts were then post-cured for 30 minutes in a post-cure chamber consisting of a string of 395-405 nm lights wrapped inside a cylindrical chamber. In order to attach devices in a modular fashion, ports were connected and unpolymerized resin was coated over device interfaces. The unpolymerized resin was then irradiated by a 405 nm laser, effectively bonding the devices.

Printing evaluation: The objective of this initial work was to demonstrate the capability of the Formlabs 1+ printer to fabricate both covered and exposed micro and millifluidic channels; the knowledge gained through this study was intended to inform subsequent design of micro and millifluidic devices for utilization at UTC and beyond. Channel test pieces (Figure 1) were designed using AutoCAD software. These designs consisted of a series of five channels of varied cross-sectional area in square, rectangular, and cylindrical geometries. Covered channel tests included a port for flushing of unpolymerized resin. The exposed channel test pieces were modeled using the channels from covered channel tests, but with no top to the channel for rectangular and square geometries. In the case of the cylindrical channels, only half of the channel was printed such that the cross section of the channel was a semicircle. The resultant prints were analyzed using optical microscopy, and printed channel dimensions were compared to that of the AutoCAD design. In order to obtain multiple representative cross-sectional measurements, the printed channel test pieces were sanded perpendicular to the channels. Five to seven cross-sectional images were taken and analyzed using Micron imaging software over a 3mm length for all channel tests.





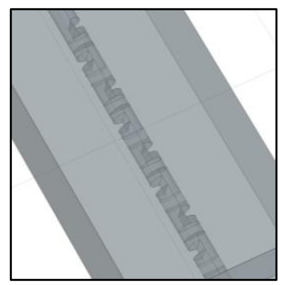
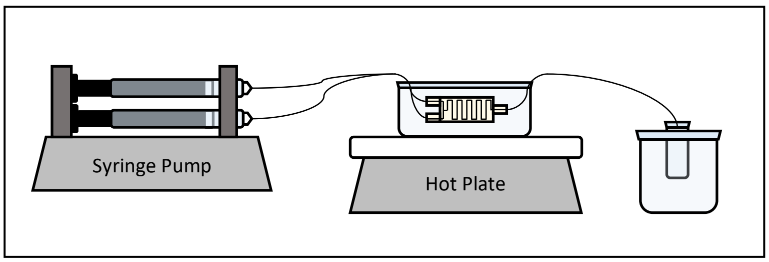


Figure 1 (left). Models of all three channel test pieces. **Figure 2 (middle).** Models of various modular fluid mixers. **Figure 3 (right).** Close up of channel with spiraling baffles rotated clockwise relative to flow of liquid.

Fluid mixing: The first device application demonstrated was fluid mixing. Initially, a device with a straight, unobstructed, cylindrical channel was designed and printed to demonstrate laminar flow. After this, devices with various obstructions within a cylindrical channel were designed to show diffusive mixing of fluids. Highlighted in Figure 2 are three devices designed for fluid mixing. All three devices contain obstructions covering half of the cylindrical channel. Each obstruction was rotated 90 degrees relative to the last obstruction to create a spiraling baffle effect (Figure 3) in order to encourage fluid mixing and diffusion. The obstructions of the middle device in Figure 2 were rotated counterclockwise relative to the direction of flow, as opposed to clockwise as in the other two devices. When physically connected in series, a long channel is created with the desired mixing effect. Water colored with blue and red commercial food coloring was used for all demonstrations of fluid flow and mixing, which was observed visually. A Cole Parmer KDS Legato 210 syringe pump was used to pump fluids through the various devices at a set flow rate.

Biodiesel production: Before biodiesel product experiments were conducted, the High Temp resin was determined to be sufficiently resistant to chemicals involved in the biodiesel transesterification reaction. After this, modular fluid mixing devices were printed, cured, and assembled for use in biodiesel production experiments. The experimental setup is shown in Figure 4. The biodiesel reactants, a 0.24 M sodium hydroxide and methanol solution and commercial, food-grade vegetable oil, were drawn into separate syringes and placed on the syringe pump. The printed mixing device was submerged in a stirred hot water bath at 60˚C. The fluids were pumped through the device at a rate of 2ml/hour and collected in a container submerged in a cold water bath to terminate the biodiesel reaction. The products were allowed to settle, and the top phase was removed and transferred to a new container by pipette and subsequently washed with 1ml of deionized water. The product was then tested qualitatively using a 3:27 biodiesel test often used as a pass or fail test for large scale home biodiesel production and in educational labs.[21]



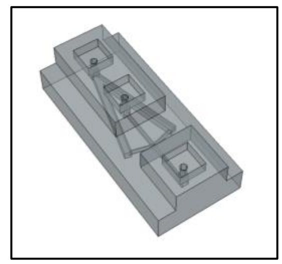
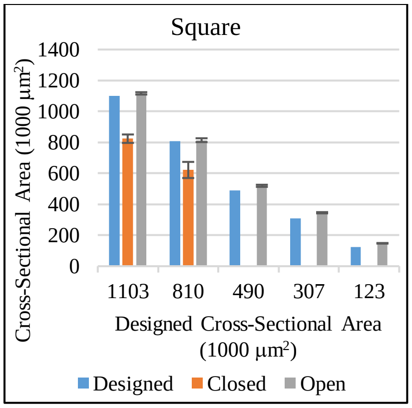


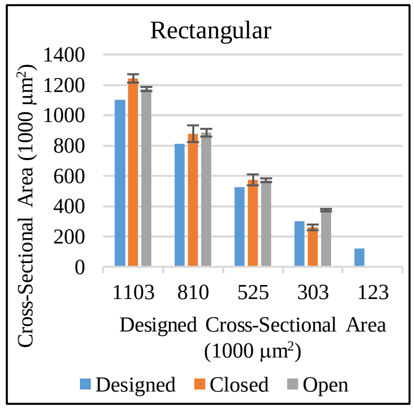
Figure 4 (left). Setup for biodiesel production experiment. The syringe pump (left) feeds reactants into the mixing device which is submerged in a hot water bath (middle). Products are then collected in a vial submerged in a cold water bath (right). **Figure 5 (right)**. Model of droplet generator.

Droplet generation: In droplet generation studies, water droplets in a continuous oil phase were created. The droplet generator (Figure 5) consisted of an entry port for oil, an entry port for water, and an exit port for oil and water droplets. The channels were designed such that the oil approached the water flow from two sides as to facilitate droplet breakoff. Droplets were collected in oil on a microscope slide fitted with a 3D printed sidewalls used to contain the liquid. Droplets were generated with varied water to oil volumetric flow ratios, including 1:1, 1:2, and 1:2.8, by use of various sizes of syringes. Resultant water droplets were imaged using optical microscopy, after which droplet uniformity at varied flow ratios was analyzed.

Results

Printing evaluation: All exposed channels were successfully printed aside from the smallest rectangular and cylindrical channels which contained some regions of over cured resin. The largest four sizes of cylindrical channels, four sizes of rectangular channels, and two sizes of square channels were successfully flushed in the closed channel test pieces. For all geometries of closed channels, the heights of the channels were consistently less than the designed height, while the width was typically larger than that of the designed width. Overall, the cross-sectional area of the closed cylindrical channels most closely aligned with that of the design, though the cross section was elliptical rather than circular. Open channel dimensions much more closely aligned with that of the design, leading to the conclusion that, by closing the channel, over curing of resin within designed channels occurs; indeed, difficulty in printing closed designs at small scales was the largest challenge identified in the device fabrication process. Because cylindrical channels most closely agreed with designed dimensions and experienced less variance, cylindrical channels were mostly used for later design of micro and millifluidic devices. The measured cross-sectional areas of all channels are shown in Figures 6-8.





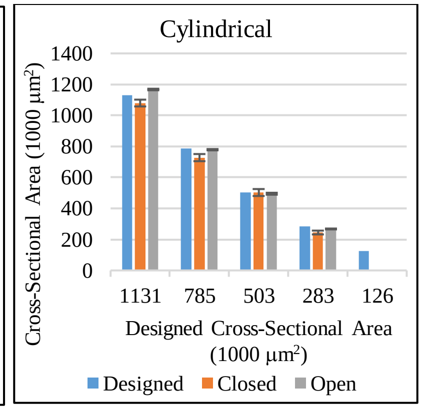


Figure 6, 7, and 8 (left, middle, right). Measured square, rectangular, and cylindrical channel cross-sectional areas, respectively. Note that channels that could not be flushed are not included in these figures.

Fluid Mixing: A printed device containing a straight cylindrical channel of designed radius 0.5 mm is shown in Figure 9. Two different colored water solutions enter the ports on the left of the device, and no significant mixing of the two fluids is apparent throughout the channel. This device provides an excellent visual representation of laminar fluid flow.

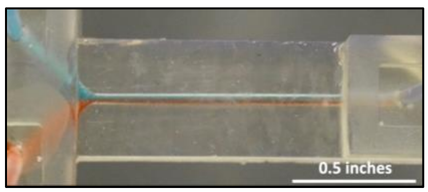


Figure 9. Printed device with straight cylindrical channel of radius 0.5 mm.

Figure 10 shows the modular devices designed for fluid mixing. In the first device separate red and blue regions can be easily observed. As the fluid continues through the device, the rotated baffles encourage mixing and diffusion, and, by the last device, the liquid appears to be well-mixed and regions of blue and red water are not observed, as detailed in Figure 11.

Figure 10 (left). Connected devices during fluid mixing demonstration. **Figure 11 (right).** Close up of last section of device in Figure 10. Note the lack of blue and red colored regions.

Biodiesel production: A biodiesel production experiment was conducted using a device similar to that shown in Figure 10. During the experiment, bubbles and two phase flow were seen within the device channels, indicating limited mixing of the reactants. Despite this fact, the resultant products experienced color change compared to the reactants, indicating some extent of reaction. After separation and washing, the resultant product passed the 3:27 test, indicating production of biodiesel in the device. Rigorous analysis of the product following ASTM D6584-17 using gas chromatography methods is recommended for future experiments to quantitatively and accurately determine extent of reaction, yield, and tunability of this production method.[22] Finally, visual inspection of the device did not reveal degradation of the device over the course of the experiment.

Droplet generation: The printed droplet generation device is pictured in use in Figure 12. Droplet generation studies demonstrated successful production of various sizes of water droplets (stained with blue food dye) at different oil to water flow rate ratios. 20 droplets were analyzed for each flow ratio. At a 1:1 oil to water ratio the mean droplet radius was determined to be 597 ± 38.8 μm. Droplets produced at a 2:1 oil to water ratio were much more uniform with a mean radius of 451 ± 5.9 μm. Droplets generated at a 2.8:1 oil to water ratio were slightly less uniform with a mean radius of 455 ± 13.5 μm, and it is theorized that this variance was a result of inconsistent flow rate of both liquids due to lower quality syringes being used during that portion of the study. This technology may be applied to coursework in chemical and biomolecular engineering, as well as research projects involving biomimetic membranes.[19, 23]

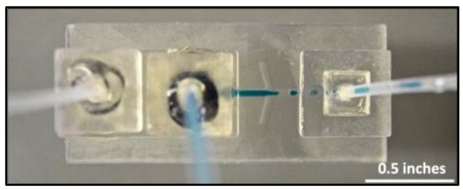


Figure 12. Printed droplet generator device during droplet generation experiment.

Conclusion and Future Work

This work demonstrated the ability to directly print micro and millifluidic devices with potential for use in undergraduate engineering courses and research using a commercial SLA 3D printer and photopolymer. Laminar flow and fluid mixing were first shown qualitatively in two different printed devices. After this, a similar mixing device was used during a biodiesel production experiment, the products of which passed a common qualitative biodiesel test. Finally, a droplet generator was printed and used to generate various sizes of water droplets in a continuous oil phase. Future work will focus on improvement of 3D printing device fabrication, the evaluation of 3D-printed micro and millifluidic device use for other applications, and integration of these devices into courses and research at UTC. Specifically, videos depicting laminar flow, diffusive mixing, and droplet generation will be created for use in engineering lecture courses. Hands-on experiments will also be developed to complement this lecture material in associated laboratory courses. These hands-on experiments will involve design and 3D printing of devices and experimental design and execution to achieve a stated objective. For example, a student team may be tasked to design a device for demonstrating and verifying laminar flow through calculation of a Reynolds number. Such experiments will ensure that the previously described SLOs are met through exploration and learning of new techniques related to 3D modeling and printing, microfluidic device operation, and general experimental design and interpretation. Feasible targets include: fluid mechanics and fluid mechanics laboratory, unit operations laboratory, and chemical process operations and chemical processes laboratory. Current research at UTC in bioengineering will also seek to incorporate this technology into undergraduate research projects.

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Cooper Thome

Cooper received Departmental Honors for work related to that presented here in his senior undergraduate year at the University of Tennessee at Chattanooga. Cooper is now a graduate student at the University of Colorado Boulder and is working toward obtaining a Ph.D. in biological engineering. He has conducted research in a variety of settings and at several institutions including Georgia Institute of Technology and the National Institute for Materials Science (Japan), and his interests include acoustofluidics, biosensing, and immunology.

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Dr. Bradley Harris

Dr. Harris is an Assistant Professor in Civil and Chemical Engineering at the University of Tennessee at Chattanooga. His research interests are in bioengineering: the application of engineering principles to biological problems. He is passionate about undergraduate research and seeks to maintain a laboratory offering opportunities for chemical engineering students interested in bio-related research.