

Microfluidic setup useful for research and teaching

Arranjo de microfluídica para pesquisa e ensino

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ABSTRACT

The aim of this work was the design, manufacturing and testing of a small, low cost setup useful for research and teaching on microfluidics. The setup was designed to be modular and was assembled using only of-the-shell parts and pieces. The final device is composed of a base, where all parts and pieces are assembled, accessories to provide continuous flow of gas (air) or liquid (water) fluids and two optical digital microscopes for diagnosis of fluid condition. The corresponding photos and videos are directly collected on a computer. The setup performs contact angle measurements even on rough surfaces and using small drops, up to 10 μL , and showed to be appropriate for microfluidic analysis of the mesostructures, such as microchannels with 100 μm on diameter.

Keywords: Microfluidics, Continuous flow analysis, Mesostructures, Contact angle measurement.

RESUMO

O objetivo deste trabalho foi o projeto, a construção e os testes de um arranjo experimental pequeno e de baixo custo, útil tanto em pesquisa como em ensino para testes em microfluídica. O arranjo foi projetado para ser modular e foi produzido com partes e peças facilmente encontradas no mercado. Esse arranjo é composto por uma base, onde todo o conjunto é montado, e acessórios que proporcionam um fluxo contínuo de gás (ar) ou líquido (água) enquanto dois microscópios ópticos digitais permitem a análise das condições do fluido. Fotografias e vídeos são automaticamente arquivados em um computador. Nesse conjunto é possível obter medidas de ângulo de contato mesmo em superfícies rugosas e utilizando pequeno volume de líquido, até 10 μL ; além disso, o conjunto se mostrou apropriado para análise de mesoestruturas, como microcanais com 100 μm de diâmetro.

Palavras-chave: Microfluídica, Análise em fluxo contínuo, Mesoestruturas, Medida de ângulo de contato.

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INTRODUCTION

Among the astonishing changes provoked by the microelectronic development on the last few decades, some are really unsuspected such as the appearance of new equipment and processes on Chemical Engineering and Chemistry. These new devices usually work on continuous flow, which requires new setups and leads to the important role of microfluidic tools in current research.

Furthermore, one of these rising areas that is not well perceived is the process intensification (PI) of Chemical Engineering processes¹⁻⁹, and a possible explanation for this, as pointed out by Sitter, Chen and Grossmann¹, is that the interest on PI research has increased only in this century. The definition of PI is broad, but the majority of the authors agree that PI, among others things, corresponds to reduction on equipment size that leads to reduction on energy consumption and waste formation, i.e., increase in sustainability. Another constant on PI research is the pursuit of multiple unit operations performing on a single device. One approach to achieve such gain on economical and environmental performance is the use of meso/microreactors, mainly due to the ability of such devices in mass and energy transfer and the high area/volume ratio that speed reactions. According to Tian et al², microreactors are one of the seven important PI technologies. However, the research on these small devices does not exclude worries regarding macroconditions; so, as described by Nikačević et al², there are two directions to ponder: the mesoscale that operates considering phenomenological approach and the macroscale that deals with process and plants. These two forces drive the future development towards molecular scale in one side and megascale on other. In this scenario, the use of simulation tools on fluid dynamics, such as COMSOL Multiphysics[®], is almost mandatory for the understanding of microsystems. Other important features are the low timescale for reactions and the possibility of using electromagnetic fields as driving forces.

Since PI deals a lot with mass and energy transfer, a further natural step is the use of microdevices, such as the ones developed by microelectromechanical systems (MEMS) research on PI design of new units. That is particularly true by mixing and microreactors, which are highly dependent on microchannels manufacturing¹⁰⁻¹⁴. Thus, for using or testing these microdevices¹⁵ the following unit operations must be provided by a test platform: a) fluid handling, b) sample handling unit, such as dilution or concentration and filtration among others, and c) fluid detection/measurement.

On the other hand, the actual dimensions of microfluidic setups/tools do not consider the requirements of PI optimization, since the devices on this area are still "big" (hundreds of microns or even few centimeters) if compared to conventional microfluidic dispositive. As stated by Fan & Luo¹³ in a review regarding critical dimensions, whereas microscale corresponds up to 100 μm , mesoscale goes up to 1 mm, for macroscale the upper limit is 6 mm but conventional scale is generally higher than it. Another important issue is the price of such commercial microfluidic

devices that, above all, tend to hinder undergraduate students to interact with these new concepts, equipment and tendencies¹⁶.

A second approach, equally important on PI area, rely on considerations of surface properties. Therefore, even for a well-designed and well-built device, issues can run from roughness to fouling, due to surface chemical characteristics¹¹. In order to address it, surface characterization uses several distinct analyses, with the contact angle measurement as one of them. This technique is useful for determining surface properties, such as hydrophilicity – if tests are carried out with water – organophilic or oleophobic materials, with organic and other compounds. Although there are many different setups for contact angle measurements, the simplest way to do it is the deposition of a small drop of liquid on the surface and the measurement of the angle formed on liquid/surface contact. Surface roughness has huge impact on contact angle with higher angles being obtained on rougher surfaces. Low cost equipment for contact angle measurements were already proposed, mainly based on image capture, even with basic digital cameras or cell phones¹⁷, and posterior angle determination by software; such tests were actually done in a physical chemistry course for Master students¹⁸, but that solution implies in low precision and a recent improvement was proposed by the use of a digital microscope¹⁹.

Thus, the aim of this work was the development of a low-cost microfluidic setup useful for research and teaching, especially on PI area.

EXPERIMENTAL

This work is based on project and manufacturing rules of mechanical devices. Thus, for all steps on this work, these requirements were followed:

1. Purchase of only commercially off-the-shelf products.
2. Easiness of assembly and disassembly.
3. Reliability with low cost.
4. Ability to carry out experiments on continuous flow using gas (air) or liquid (water) as fluids.
5. Use not only on undergraduate teaching but also as a research device, which means versatility and manipulation of small prototypes (mesostructures) mainly for surface analysis, respectively.

The setup was designed to be used over a common laboratory bench, lightweight and useful for analysis of 3D and planar dispositive. The plentiful use of plastic material, among others materials, assures long life and also allows the employment of acid and/or basic solutions during experiments. The project favor modular instruments in order to provide portability and versatility, however, although most parts are easily disassembled, due to the use of liquid reactants, the base of the apparatus is one single piece.

The mesostructures tested were manufactured on acrylics, which allows the use of optical microscopy and tracers (mainly aqueous solution of aniline) to provide a better fluid analysis. Moreover, since these devices are assembled by using silicone glue (3D structures) or double-sided adhesive tapes (planar structures), they are also easily disassembled. The mesostructures were previously extensively simulated and tested, not only for MEMS and sensors development²⁰ but also for educational purposes²¹, and represent the most common unit operations: mixers, impactors and reactors. Figure 1 shows the mesostructures used for testing the designed setup and some of them can present multiple unit operation; therefore, the reactor is also a mixer, the same occurring with the impactors, which means they are not only useful for remove particles but also mix different liquids. The 3D mixer is also a spray system. Reactants were proanalysis grade (Casa Americana S.A.) and environmentally correct.

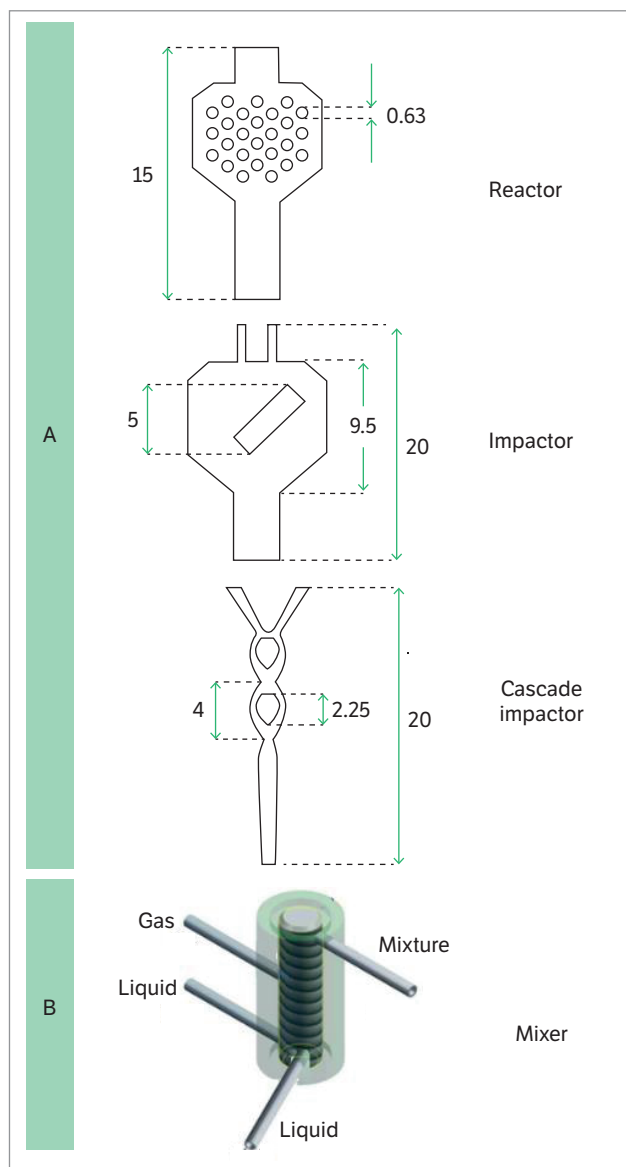


Figure 1: Mesostructures used for testing the designed setup (dimensions in mm); (A) planar²⁰; (B) 3D²¹

RESULTS AND DISCUSSIONS

This item describes the manufacturing of the proposed setup and the corresponding tests with mesostructures.

Setup manufacturing

The manufactured setup is mainly composed of three different parts:

- Base – where all parts and pieces that compose the modular instruments can be assembled.
- Modular pieces with several distinct functions.
- Diagnosis devices, which in this work are two optical digital microscopes, as explained later on the text, but other solutions can be adapted by users.

The base is composed of two parts: an aluminum recipient (approximately 1.0 m length $\times$ 0.5 m width $\times$ 5 cm height) and two drilled plastic pallets (0.5 m length $\times$ 0.5 m width $\times$ 2.5 cm height) disposed inside the aluminum container. Whereas the plastic pallets work as holder for microfluidic setup, the aluminum device avoids that any leak of liquids during tests hits researchers/students or other equipment. Furthermore, the aluminum choice for the external container favors to ground any electric equipment used on the setup; equally important, the use of aluminum leads to a lightweight dispositive. Since the pallets are regularly potholed, it is possible to machine fastening pins (in this case PVC was used as material, just for economic considerations) for fixing the modular pieces. Figure 2 shows these pieces in a completely disassembled setup.

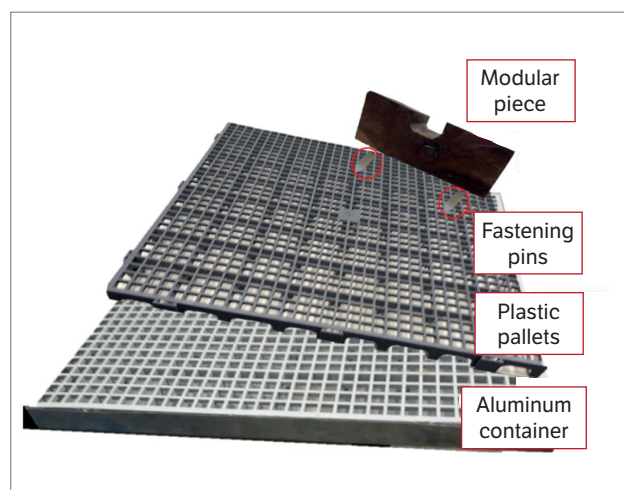


Figure 2: Image of setup base main parts.

The modular pieces are mainly:

- Air compressor (Vigo Ar 300, Brazil) and water pump (Sarlo S300, Brazil) for carrying on fluids, air and water, respectively.
- Plastic recipients, for water and reactants storage.
- Flow meters for air control.

- d. General connections, such as valves, usually used on medical care (Hartmann, Brazil) and capillaries for fluid transportation on a continuous flow.
- e. Machined modules.

Figure 3 shows a generic arrangement over the base that would attend gas and liquid tests. It is worth emphasizing that, due to the modularity, the layout can be quickly changed; furthermore, the fastening pin assures that each piece gets firmly stuck in the chosen position. The choice for air compressor is supported by previous tests carried out for different groups²¹⁻²³, that indicated air compressor as a reliable tool for continuous flow. The use of small water pump is possible due to the small pressure drop on the entire system; however, mesostructures with long channels or critical constrictions can be a hindrance.

Aside the conventional pieces that allow a constant flow inside the mesostructures, two identical modules were machined to provide broad changes in the focus during the use of the microscope to support:

- a. Planar samples: these samples can be as smooth as silicon wafers, or rough – for instance, plastic material. It is usually used on contact angle measurements, evaluation of thin films, etc.
- a. Mesostructures: in this situation, the structures are attached to the module with two flexible hooks, i.e., in a similar way of the “third hand” on a soldering electrical setup. In such conditions, the microscope will be held by its own support or the correct focus will not be achieved; on the other hand, this arrangement supplies a 3D vision on fluid behaviors. Figure 4 shows the assembly of both

configurations; however, it is worth noticing that, due to the need of stiffness, the modules are not interchangeable (i.e., the second module is not designed to be disassemble), but the microscopes can be. The main advantage of digital microscopes is that photographs or eventually videos are directly collected on a computer, in other words, online information is provided. This work used two distinct digital microscopes: one with amplification up to 200X and other up to 500X (VC68U, Akkord, USA). While the 200X microscope is useful on contact angle measurements, the 500X microscope is more appropriate for fluidic analysis on the mesostructures.

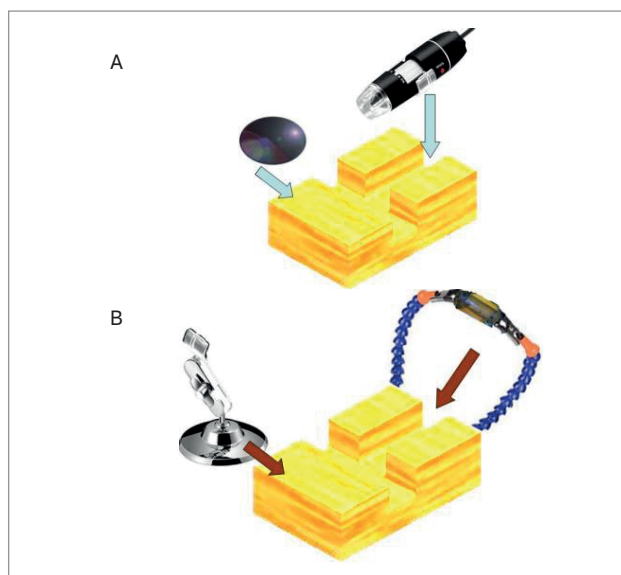


Figure 4: Module for (a) contact angle measurement and (b) fluidic analysis.

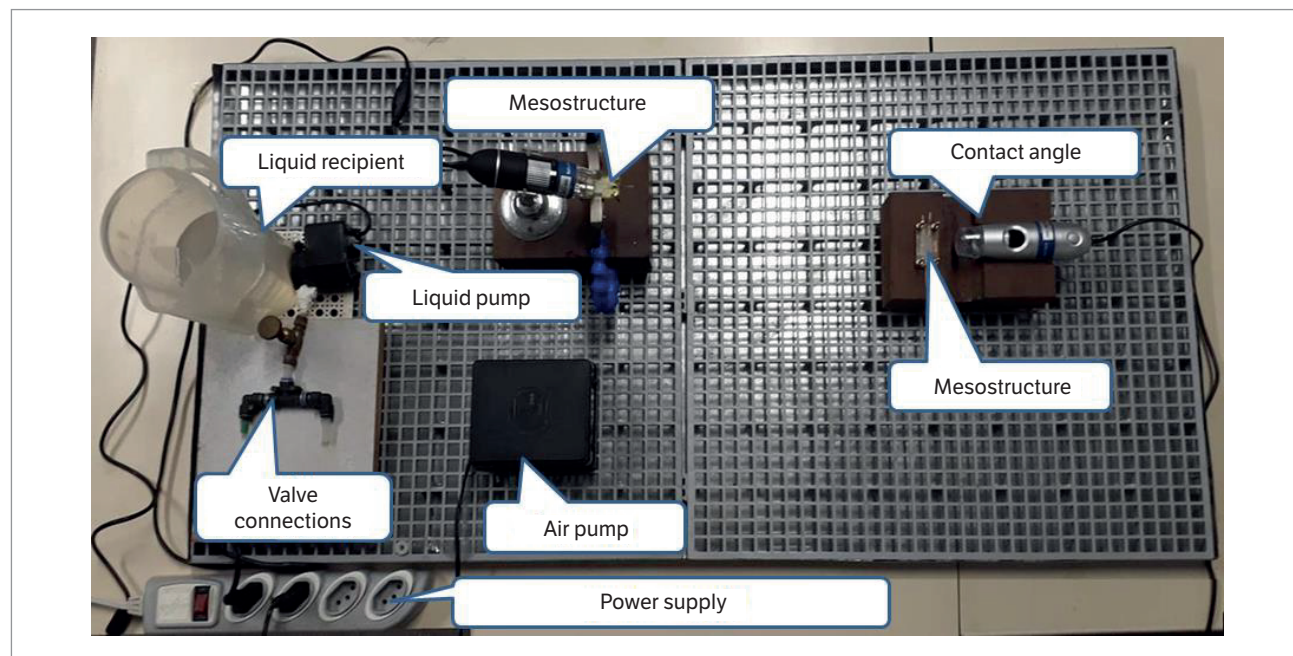


Figure 3: Setup assembled and modular pieces installed (plumbing not shown to improve visualization).

Setup testing

The main achieved results are shown in two different forms, with or without fluid flow, i.e., PI and contact angle measurements, respectively.

Contact angle measurements

Measurements of contact angle can be done with water or any reactant, even volatile ones, if a fume hood is used (Fig. 5). The depth of focus is capable of showing drops separated from each other by several centimeters (Fig. 5A). Due to the area of the sample the microscope can analyze, thickness is also not a concern (Fig. 5B). The accuracy on this technique is quite dependent on the amount of liquid used to produce the drop; 1 μL drops can be recorded by the microscope on maximum amplification (Fig. 5C) and in a system with several other near drops. In this image (906×506 pixels) and considering the 10

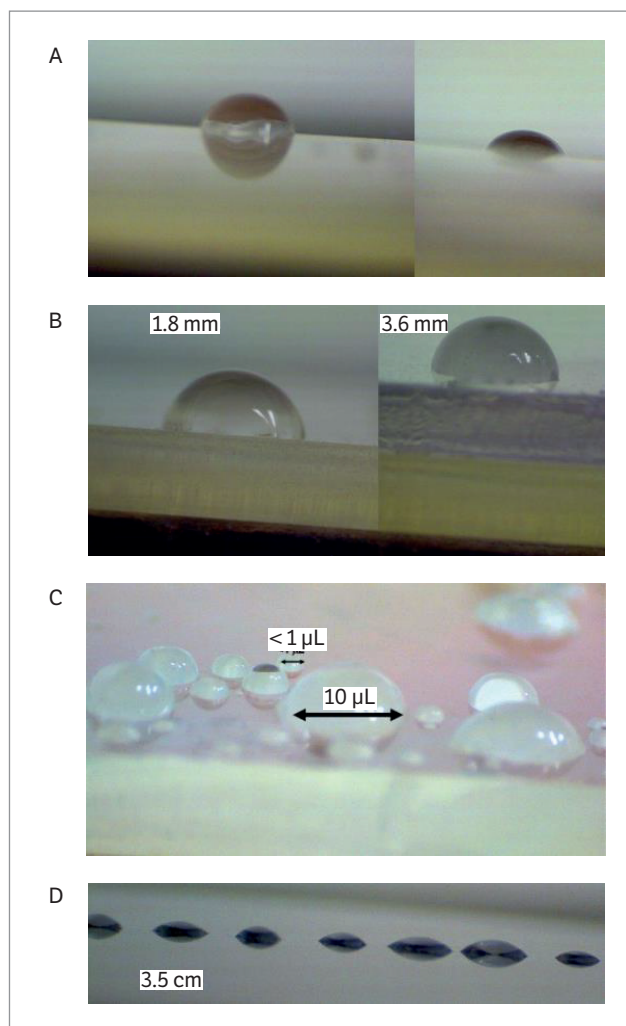


Figure 5: Contact angle measurement: (A) Drop nearest and farthest of the microscope; (B) Smooth and thin, rough and thick substrates, the dimensions in mm refer to substrate thickness; (C) different volumes of drop over a PVC substrate; (D) 10 drops reacting on the silicon surface and far from the microscope, the dimension in cm refers to the linear distance of the analyzed sample.

μL drop as reference, the nearest drop on the left is ca. 0.15 mm apart but the contour is still clearly observed; thus, resolution of 0.1 mm can be expected. On minimum amplification, 10 μL drops are still visible, and at least 10 of these drops can be recorded simultaneously, which means the system has good depth of field. Moreover, and more important, if these drops present different concentrations of a specific reactant, for instance, recording them as a function of time allows the study of surface changes and determination of chemical reaction rates (Fig. 5D). Due to the good focus, any of the recorded drop shown in Fig. 5 can have their contact angle determined by basic software, such ImageJ; thus, the system has good performance for research and educational developments.

Microfluid behavior

All mesostructures shown in Fig. 1 were easily visualized on the setup, as can be noticed in Fig. 6, with minimum amplification, which means approximately 50X. For the 100 μm 3D channels, an arc of almost 90° of circumference is perceived during analysis, which means ca. 10 mm of depth of focus. Furthermore, by increasing the amplification some of these channels can be better scrutinized (detail in Fig. 6), i.e., this change does not require disassembly, only change in amplification, and approx. 100 μm depth of focus has been achieved. In addition, to evaluate the different portions of these structures, a slight change in the position of the flexible hook is all that is required.

Some simple fluid manipulation was carried out on such structures, as can be seen in Fig. 7, which shows snapshots of videos taken during mesostructures use on the setup. On the microreactor, the use of two different water flows, one of them tainted with green aniline, allows to infer the flow path and behavior, as can be noticed in Fig. 7A. It is possible to observe in this figure the pillar that compose the flow constraints (yellow circles) and some shadows, which – on maximum amplification – corresponds to flow mixture as the distinct green shades denote. The addition of oil sample on water carrier flow can lead to similar results and Fig. 7B shows the exit of a cascade impactor after the use of tainted oil. The regular color on the mainstream and the strong shade on the walls indicate that it also has preferential paths although some mixing occurs. The 3D microchannels were designed to provide mixing of two flows with high gap on viscosity; Fig. 7C shows glycerol, fluid highly soluble in water, added to the water flow. Since water and glycerol are colorless liquids, glycerol was dyed in blue to allow visualization. The small blue drops are indication of good mixing, the same occurs with mineral oil (Fig. 7D). All these results pointed out for the usefulness of such setup for educational and research works.



Figure 6: Mesostructures visualized on the setup with minimum amplification.

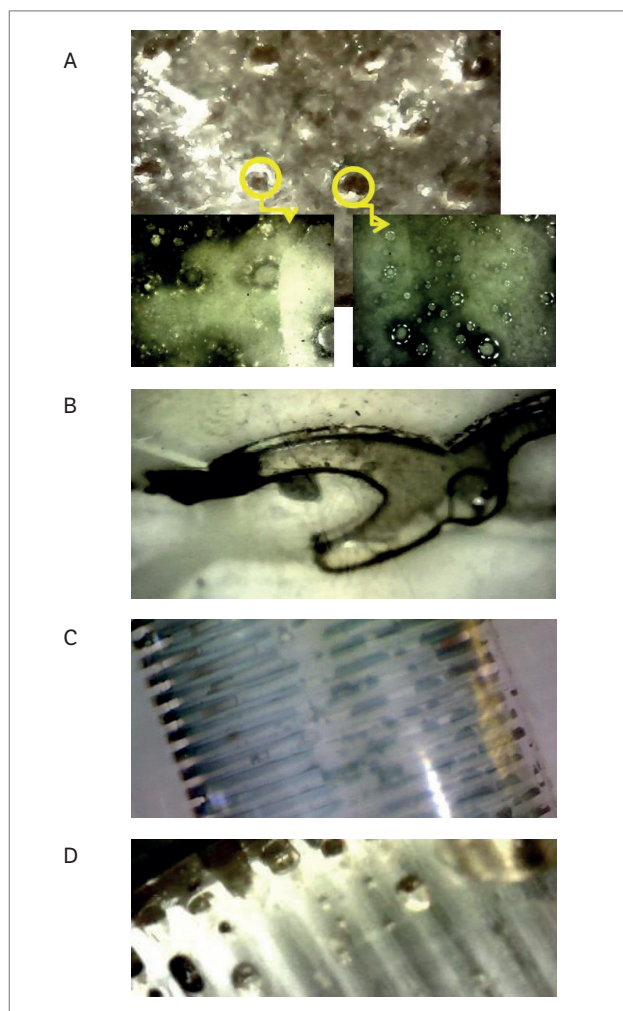


Figure 7: Microfluid behavior: (A) Mixing of two flows in the microreactor, (B) Cascade impactor and oil exiting the structure, (C) Glycerol tainted with dye and (D) Small drops of oil tainted on the 3D microchannel.

CONCLUSIONS

This work shows a simple setup for contact angle measurements and fluidic research. The contact angle can be measure with small drops, such as $1\ \mu\text{m}$, and realized even during surface reactions. Fluidic behavior can be easily revealed on microchannels as narrow as $100\ \mu\text{m}$, even in three-dimensional structures; thus, several unit operations can be simulated on this setup. It is also possible to infer differences in fluidic behavior within these channels by controlling the depth of focus. Considering all its capabilities and that the whole set cost less than US\$300.00, excluding labor costs, this setup is a useful way to conduct experiments in the PI process, and even microfluidics, not only in research but also in education.

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