Discrimination of low-grade oil from edible oil by a microfluidic device

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Abstract. Illegal cooked oil is a serious food safety issue in China, while an effective authentication method is still lacking. In this paper, a microfluidic device was applied for the discrimination of low-grade oil from edible oil, by creating water droplets of different sizes in different oils.

Introduction

Recycled cooking oil, or gutter oil, has emerged as a serious food safety issue that challenges government authorities and consumers in China. Gutter oil is refined from kitchen waste, gutters, sewer drains, and slaughterhouse waste, as well as oils that have been repeatedly used to fry foods. By a series of simple processes, including collection, preliminary filtration and boiling, refining, and the removal of adulterants, illegal gutter oil can be cleaned up enough to meet the sanitation standards of cooking oil and resold as a cheaper alternative to normal cooking oil, to make huge profits. It is even estimated that up to 1/10 of the edible oil consumed in China is gutter oil.

Because of the same major chemical compositions of gutter oil and normal cooking oil, and lacking of typical chemical markers with strong characteristics and high accuracy, it is extremely challenging to identify gutter oil. Till now numerous detection methods appear for gutter oil [1]: (1) sensory test, (2) physicochemical inspection, (3) instrumental methods. Sensory tests preliminarily estimate the oil by its color, transparency and odor, which are intuitive and fast, but limited and suffer from low precision. Traditional physicochemical inspections use the quantitative difference of feasible parameters between gutter oil and normal cooking oil to inspect, e.g., acid value, refractive index, peroxide number, iodine value, carbonyl value and polar components. In addition, researchers also explored the application of numerous analytical instruments for screening gutter oil, including chromatography, mass spectrometry [2], spectroscopic methodology [3], nuclear magnetic resonance spectroscopy [4-6], Raman spectroscopy [7], and so on, which usually distinguish gutter oil from normal oil based on specific components, characteristic peak, and characteristic spectrum.

In this paper a microfluidic device capable of generating monodispersed droplets was applied to distinguish low-grade oil from edible oil, which is easy to use, portable and economic, and only consumes little sample oil.

Concepts and principles

Droplet formation, which provides an appealing way to produce emulsions with excellent uniformity, could be studied in various microgeometries, such as microchannel arrays, T-sections, hydrodynamic focusing, and concentric injection. In this paper it is studied in a symmetric microfluidic hydrodynamic focusing device, which consists of four microchannels with identical heights.

Hydrodynamic focusing is a common phenomenon that when one fluid flows into another immiscible fluid at certain flow rates, an instability would occur that the inner fluid was split by the
outer one, forming dispersed droplets, which is usually used for the production of micro-droplets [8], bubbles [9] and microparticles [10].

In this study, hydrodynamic focusing is applied to generate water droplets in different oils. According to previous research, sizes of the droplets will depend on the width of microchannel, the flow rates, the geometry, and the property of the oil and water. So with the other conditions remain constant, the difference in sizes of the droplet will reflect the difference in properties of the oil, which could be able to distinguish low-grade oil.

**Experimental procedures**

A. Materials and devices

Three types of oil are used in experiment, as shown in Figure 1(a). Oil A, the edible oil, is bought at the supermarket (Jinlongyu rapeseed oil), which is dark yellow compared with the others. Oil C is bought at a street stall, which is very cheap and almost colorless-apparently a type of low-grade oil. Oil B, also a type of low-grade oil, is the mixture of Oil A and Oil C (0.5:0.5), which is light yellow. Oil B must be shaken by a vortex mixer for 5min for thorough mixing of the two types of oil.

The microfluidic devices were fabricated using poly(dimethyl)siloxane (PDMS) and a silicon wafer by soft lithography. Briefly, a glass mask was designed and fabricated. The master structures of 29µm high SU-8 photoresist were prepared by mask photolithography on a silicon wafer. A mixture of PDMS prepolymer and curing agent (Sylgard 184, Dow Corning) was poured on the positive master structure on the wafer. After heat curing, the negative PDMS relief was peeled off. Holes were punched at the center of the reservoir areas and the relief was sealed onto a silicon wafer to form a closed channel, as shown in Figure 1(b).

![Figure 1 (a) Three different oil samples; (b) The microfluidic device.](image)

B. Establishment of the experimental system

The microfluidic device was connected to the oil and water sources on syringe pumps by plastic tubing. The microscope, video camera and computer recorded the experimental process when it reached stable, as shown in Figure 2.

C. Experimental process

In this experiment, oil was used as the continuous phase, while deionized water was used as the dispersed phase. Oil was injected into the two sheath flow inlets, and deionized water was injected into the middle sample inlet. Each sheath flow was set as 0.2mL/h, while the water flow was adjusted from 0.05mL/h to 0.4mL/h. After each adjustment step, the flow is allowed 3min to get stable before taking images.

Three types of oil were in turn injected into the microfluidic chip with water. Based on the above conditions, and with the combining effect of inner viscous forces and interfacial tensions between water and oil, the water flow will go through the cap displacement, cap squeezing, pinching, tail stretching and completely breaking up process[11], and be split into dispersed droplets by the continuous oil phase, as shown in Figure 3.
Discussions

A. Formation of water-in-oil droplets

A wide range of instabilities would arise when streams of immiscible fluids get in touch. The various dynamic instabilities depend on the thermodynamic properties of the fluids, the driving stresses, and the flow geometries. In our case, the surface tensions drive an interfacial instability, ultimately overcoming viscosity to cause the water thread to break up into droplets. The size of the droplet strongly depends on the geometry of the microchannel, the flow rates, and viscosity of the oil and water.

One of the advantages of this approach is that, it does not need surface treatment on the microchannel walls, i.e., the microfluidic device could be used as fabricated. And it could be reused as long as it is cleaned before each step and remains intact.

B. Discrimination of oil by droplet sizes

As seen in Figure 3, the oil flow rate is constant, as the water flow rate increases, the droplet will become larger in dimensions, and its distribution density will increase as well. As the sheath flow changes from oil A to oil C, the size of the droplet will also be different. The relationship between the normalized size of the droplet and the flow ratio of water to oil was summarized in Figure 4, where the normalized size of the droplet is the ratio of the size of the droplet to the width of the enveloping microchannel (1mm); the size of the droplet is the average of the length and width of the rightmost droplet in each figure. It is defined this way because the droplets did not evolve into a perfect round shape throughout the imaging zone.

From Figure 4 it can be seen that, the droplet will enlarge as the flow rate ratio of water to oil increases; and with the same flow rates, as the oil is different, the dispersed droplet size will also be distinctly different. Computation results implicate that the difference of droplet sizes for Oil A and
Oil C of the same flow condition could be up to 30%, which means, the microfluidic droplet-generating approach could be a feasible way to distinguish different oil.

Setting Oil A as a reference, Oil B and Oil C could be distinguished. However, a quantitative standard is required, the resolution, linear characteristic, reliability of this approach needs to be identified. What is more, the compositions and sources of oil are very complex and variable, our current work only covers two types of common oil. Further work needs to be done to testify this approach for widespread oil discrimination.

C. Selection of flow model and rates

The syringe pumps used in the experiment are mechanical pumps, which often suffer from pulsatile flow, i.e., the flow is not smooth, and changes abruptly, which will in turn cause the droplets to suddenly enlarge or shrink. In the contrary, the microfluidic device will burst if the flow rate is too high. Combining those concerns, the oil flow rate is set as 0.2mL/h (Its minimal programmable flow rate is 0.025mL/h).

For the same reason, water-in-oil mode was selected rather than oil-in-water mode, since the latter one requires a quite high flow rate to happen.

Conclusion

A microfluidic hydrodynamic focusing device was implemented to generate water-in-oil droplets, and by the sizes of the droplets, oil of different grades could be distinguished. Completely different with common spectroscopy methods, this approach is beneficial since it is economic and could be portable and disposable. Future work could be the establishment of reference standards for special type of oil.

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