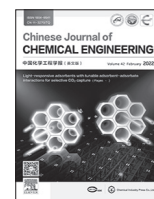




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Full Length Article

Determination of interfacial tension and viscosity under dripping flow in a step T-junction microdevice



Li Ma, Yongjin Cui, Lin Sheng, Chencan Du, Jian Deng, Guangsheng Luo*

State Key Laboratory of Chemical Engineering, Department of Chemical Engineering, Tsinghua University, Beijing 100084, China

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ABSTRACT

Microfluidic approaches for the determination of interfacial tension and viscosity of liquid-liquid systems still face some challenges. One of them is liquid-liquid systems with low interfacial and high viscosity, because dripping flow in normal microdevices can't be easily realized for the systems. In this work, we designed a capillary embedded step T-junction microdevice to develop a modified microfluidic approach to determine the interfacial tension of several systems, specially, for the systems with low interfacial tension and high viscosity. This method combines a classical T-junction geometry with a step to strengthen the shear force further to form monodispersed water/oil (w/o) or aqueous two-phase (ATP) droplet under dripping flow. For systems with low interfacial tension and high viscosity, the operating range for dripping flow is relative narrow whereas a wider dripping flow operating range can be realized in this step T-junction microdevice when the capillary number of the continuous phase is in the range of 0.01 to 0.7. Additionally, the viscosity of the continuous phase was also measured in the same microdevice. Several different systems with an interfacial tension from 1.0 to 8.0 mN·m⁻¹ and a viscosity from 0.9 to 10 mPa·s were measured accurately. The experimental results are in good agreement with the data obtained from a commercial interfacial tensiometer and a spinning digital viscometer. This work could extend the application of microfluidic flows.

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1. Introduction

The property of interfacial tension is one of the most important physical properties for liquid-liquid systems. This property could greatly determine the phase separation and droplet coalescence [1], and therefore influence mass/heat transfer [2–4], multiphase chemical reaction [5,6], functional materials [7,8] and pharmaceutical materials preparation [9,10], biomedicine and biochemical analysis [11–13], protein crystallization [14], and many other applications. Over the past two decades, various methods have been developed to measure the interfacial tension of liquid-liquid systems, such as the direct measurement technique using a microbalance and some typical techniques based on capillary pressure, capillary gravity forces or gravity-distorted drops [15]. Unfortunately, all the equipment are relatively complicated and hard to operate. Especially, it is not very reliable for the systems with low or ultralow interfacial tension. Therefore, more facile and reliable methods for the measurement of interfacial tension are required. With the growth of microfluidic technologies, many microdevices

and approaches have been developed for the preparation of highly monodispersed microdroplets [16,17], including T-junction microchannels [18], flow-focusing microchannels [18], coaxial microchannels, and so on. Droplets generation methods mainly include cross-flowing shearing [19,20], perpendicular flowing shearing [21], hydrodynamic flow focusing [22,23], and geometry-dominated breakup [24]. It has been proven that the droplet size can be controlled accurately in microdevices. Therefore, measuring the interfacial tension based on the droplet size [13], the droplet shape [25,26], and the pressure drop [25,27] has attracted widespread attention owing to the advantages of facile [28], reliable, high efficiency, and accuracy.

To measure the interfacial tension, the first microfluidic method was proposed and implemented by Hudson and co-workers [29,30], which is mainly based on the deformation and retraction dynamics of drops under an extensional flow in a microchannel. In this approach, the interfacial tension values can be drawn rapidly by calculating the rate change in the deformation of the droplet. Xu *et al.* [13] applied a coaxial microfluidic device to measure the interfacial tension based on the balance of the viscous shear force and interfacial tension during the droplet formation process. Nguyen *et al.* [31] proposed a microfluidic technique using

* Corresponding author.

E-mail address: gsluo@tsinghua.edu.cn (G. Luo).

a T-junction microdevice to measure some physical properties such as viscosity, surface tension, and interfacial tension. Additionally, some studies on test of interfacial tension and dynamic interfacial tension using microfluidic method by Luo's group have been reported [6,32–35]. All these microfluidic methods are considered to be facile and convenient. But there are some limitations regarding the measurement of systems with low interfacial tension or high viscosity. In these situations, all the techniques are hardly applicable or is unsuitable very much. Additionally, compared with normal oil-water systems, the interfacial tension of aqueous two-phase systems is extremely low, sometimes as low as 1–100 $\mu\text{N}\cdot\text{m}^{-1}$ [36]. The low interfacial tension with w/w systems tends to form all-aqueous jets rather than droplets due to the slow growth of the Rayleigh-Plateau instability [36]. Thus, it is a challenge to generate aqueous two-phase (ATP) droplets in microdevices. Therefore, microfluidic measurement techniques with more flexible and much wider applicability must be further developed.

At present, the microfluidic approaches used to measure the interfacial tension are mostly operated under liquid-liquid dripping flow, because compared with jetting flow, the generated droplets under dripping flow can be more stable with a highly uniform size. In this way, the measurement accuracy can be guaranteed. To the best of our knowledge, microdroplets with uniformity and monodispersity can be generated under a dripping flow for normal liquid-liquid systems in T-junction, coaxial and flow-focusing microchannels [18]. However, the operating range for generating microdroplets under a dripping flow is relatively narrow [37,17], and especially, liquid-liquid dripping flow is even hard to realize for systems with a low interfacial tension and high viscosity, such as ATP system. Therefore, it can be known that the limitations of all the reported methods are mainly come from the structures of microdevices. Therefore, specially designed microdevices must be developed and applied.

In this work, we attempt to measure the interfacial tension and viscosity of liquid-liquid systems by using a novel capillary embedded step T-junction microchannel, which was specially designed by Luo's group [37]. In this new microdevice an asymmetric flow field can be formed, and it can strengthen the shear force in the microdispersion process. Thus, liquid-liquid microdispersion process under dripping flow can be easily realized. In this way we may develop a more reliable microfluidic measurement technique, which could have more applicability for different systems in a much wider operating range. More than 8 liquid-liquid systems including water/oil (w/o) systems and an ATP system were selected as the working systems. A liquid-liquid microflow pattern and a mathematical model under dripping flow to calculate the interfacial tension and viscosity were provided. The experimental results were compared with the values measured by commercial instruments to validate the reliability of the microfluidic technique. In addition, the advantages of the new microfluidic technique were summarized.

2. Experimental

2.1. Materials and reagents

1-Butanol ($\geq 99\%$) and *n*-hexane ($\geq 97.0\%$) were obtained from Greagent. Chitosan with degree of deacetylation 80%–95% was purchased from Sinopharm Chemical Reagent Co, Ltd, *n*-octanol (99%), and Caprolactam (99.5%) were purchased from Shanghai Meryer. H_2SO_4 (AR) was purchased from Sinopharm Chemical Reagent Beijing Co, Ltd. $\text{NH}_3\cdot\text{H}_2\text{O}$ was purchased from Shanghai Titan Scientific Co, Ltd. Span 80 used as the surfactant dissolved in *n*-octanol purchased from Shanghai Meryer. Deionized water was obtained from an ultrapure water machine (Center120FV-S, ZEBULA Technology,

China). The composition and physical properties of liquid-liquid systems in this work are listed in Table 1. The viscosity and density of different liquid-liquid systems were measured by a commercial spinning digital viscometer (DV-IIa+P, Brookfield, USA) and an electronic densimeter (LC-MDJ-600G, LICHEN Technology, China), respectively. The interfacial tension of the liquid-liquid systems was measured by a commercial interfacial tensiometer (OCAH200, DataPhysics Instruments, Germany) based on pendant drop technology.

2.2. Fabrication of the microdevice

In this work, A capillary embedded step T-junction microchannels was specially designed and used to generate microdroplets, which was fabricated on a polymethyl methacrylate (PMMA) chip with a size of 20 mm $\times$ 20 mm $\times$ 3 mm. The main upstream channel was 400 μm in width (*W*) and 365 μm in height (*h*). In addition, the size of the main downstream channel and the side channel both in width and in height were 365 μm . A quartz capillary with an outer diameter of 365 μm and an inner diameter of 75 μm was embedded in the side channel and extended into the main channel. After the microdevice was fabricated, it was sealed using another PMMA chip at 75 $^{\circ}\text{C}$ for 3 min using a high-pressure thermal sealing technique. The structures of the microdevice are shown in Fig. 1.

2.3. Experimental schematic

The experimental schematic diagram is shown in Fig. 2. The dispersed phase was injected into the side channel by a syringe pump (LSP01-1BH, Longer, China). The continuous phase was injected into the main channel by a syringe pump (LSP01-1BH, Longer, China) simultaneously. The surfactants of Span 80 and Tween 20 were added into the continuous phase and dispersed phase to reduce the interfacial tension and stabilize the droplets. The surfactant concentrations were all kept at 2.0% (mass) in the experiment, which is much higher than the critical micelle concentration to eliminate the mass transfer influence. To minimize the measurement error, the flow rates of the two pumps were calibrated using deionized water. The droplet generation process was carried out with a microscope connected to a high-speed CCD video camera (i-SPEED TR, Olympus, Japan). The images were recorded with different frame rates adjusted to match the droplet generation frequency. The droplet size was measured by i-speed software. The entire process of droplet formation requires 30 s of equilibration time after changing the flow parameters. To ensure the measurement accuracy, 100 microdroplets were measured to determine the average size. All experiments were conducted at room temperature and atmospheric pressure. For the measurement of the viscosity of the continuous phase, different mass concentrations of chitosan were added to an aqueous solution of 2% (mass) acetic acid.

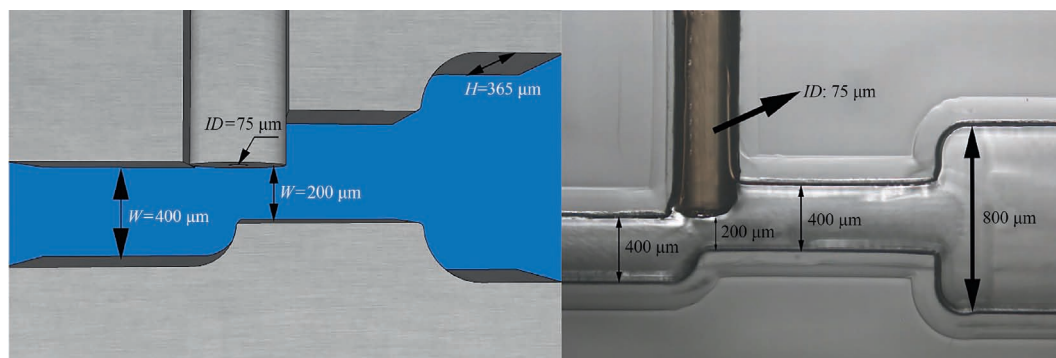
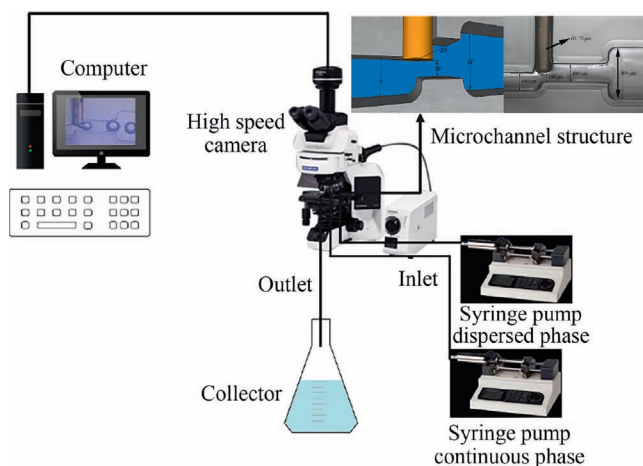
2.4. The measurement fundamentals and mathematical model

During the liquid-liquid microdispersion process under dripping flow, the interfacial tension and viscous shear force are the dominant forces [38–40]. Other forces, such as inertial force and buoyancy force can be neglected. In this capillary embedded step T-junction microchannel, there are two typical flow regimes: dripping and jetting. In the process of droplet formation, compared with jetting flow, the generated droplets under dripping flow are more stable, more uniform and highly monodispersed. In addition, droplet generation is mainly affected by the flow rates, the viscosity of the two phases, and the interfacial tension.

Table 1

Physical properties of the liquid-liquid system (25 °C)

Continuous phase	Dispersed phase	Density /kg·m ⁻³ (continuous/dispersed phase)	Viscosity/mPa·s (continuous/dispersed phase)	Interfacial tension /mN·m ⁻¹
2% (mass) Span80 <i>n</i> -octanol	deionized water	820/998	8.20/0.85	4.30
2% (mass) Span80 <i>n</i> -hexane		654/998	0.95/0.85	2.99
<i>n</i> -octanol		827/998	8.06/0.85	8.26
<i>n</i> -hexane	deionized water +2% (mass) Tween 20	648/990	0.65/0.88	1.65
2% (mass) Span80 <i>n</i> -octanol		820/998	8.20/6.35	6.65
		820/1007	8.20/17.7	6.72
	1.2% (mass) chitosan	820/1009	8.20/41.83	6.94

**Fig. 1.** Structures of the capillary embedded step T-junction microchannel.**Fig. 2.** The experimental schematic diagram.

Under dripping flow, the dynamics of droplet breakup are dominated by the force balance between the interfacial tension and the viscous force [38–40], as shown in Fig. 3. Therefore, the equation during droplet generation can be written as Eq. (1):

$$F_D = F_\sigma \quad (1)$$

where F_D represents the viscous shear force during the process droplet formation induced by continuous phase flow, and F_σ represents the interfacial tension between the two phases.

2.4.1. Viscous shear force

According to the literature, the values of the viscous shear force can be approximately expressed by Eq. (2) at low Reynolds number Re [38,39,41,42]:

$$F_D = 3\pi(u_c - u_d)d_d\mu_c \quad (2)$$

where $u_c - u_d$ is the relative local velocity of the continuous phase flow u_c with respect to the velocity of the drop u_d , d_d represents the

average diameter of the droplets, and μ_c is the viscosity of the continuous phase. It is worth note that, the Reynolds number of continuous phase is in the range of $0.23 < Re_c < 58.44$ in experiment. For simplicity, we can use the average velocity of the continuous and dispersed phase flow $\bar{u}_c$ and $\bar{u}_d$ to replace the local velocity, then, Eq. (2) can be replaced by Eq. (3):

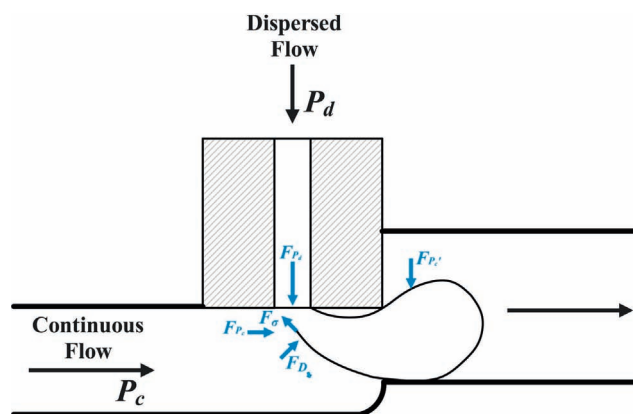
$$F_D = k_D(\bar{u}_c - \bar{u}_d)d_d\mu_c \quad (3)$$

where k_D is a constant for a fixed geometry microdevice. The values of $\bar{u}_c$ and $\bar{u}_d$ can be calculated by Eqs. (4) and (5):

$$\bar{u}_d = \frac{4Q_d}{\pi d_d^2} \quad (4)$$

$$\bar{u}_c = \frac{Q_c}{W \times H - \pi d_d^2/4} \quad (5)$$

where Q_d and Q_c in Eqs. (4) and (5) are the dispersed and continuous phase volume flow rates, respectively. W and H are the width and height of the main microchannel, respectively.

**Fig. 3.** Force analysis of droplet generation.

2.4.2. Interfacial tension

The interfacial tension can be calculated by Eq. (6) according to the literature reported [38,41,42]:

$$F_\sigma = \pi d_n \sigma \quad (6)$$

where σ and d_n are the interfacial tension and the inner diameter of the capillary, respectively. Eq. (6) based on the assumption that the Laplace pressure of the liquid droplet is at its maximum value when the diameter of the liquid drop curvature is equal to the capillary diameter, whereas in the actual situation, there will exist a departure of the interfacial from the rim of the capillary during the detachment process. Therefore, the interfacial tension is expected to be greatly reduced, and Eq. (6) is no longer invalid to predict the interfacial tension. Then, Eq. (6) was modified based on the equilibrium droplet diameter instead of the dispersed phase channel width by Husny and Cooper-White [43] and can be further described by Eq. (7): it should be noted that the applicable range for measuring the interfacial tension and viscosity based on Eq. (3) and Eq. (7) is under dripping flow. In our experiment, the measurement of interfacial tension and viscosity were carried out under dripping flow that is when the capillary number of the continuous phase is in the range of 0.01 to 0.7 as shown in Fig. 4.

$$F_\sigma = \frac{\pi d_n^2 \sigma}{d_d} \quad (7)$$

Combining Eqs. (1), (3) and (7), Eq. (8) can be obtained:

$$k_D(\bar{u}_c - \bar{u}_d)d_d\mu_c = \frac{\pi d_n^2 \sigma}{d_d} \quad (8)$$

Furthermore, we can obtain a mathematical model to predict the interfacial tension and viscosity:

$$\frac{d_d^2}{d_n^2} = \frac{\pi \sigma}{k_D \mu_c (\bar{u}_c - \bar{u}_d)} = k \frac{\sigma}{\mu_c (\bar{u}_c - \bar{u}_d)} \quad (9)$$

where k is a parameter of the fixed microchannel. In Eq. (9), the value of k mainly depends on the geometry of the microchannel, the degree of confinement, the surfactant concentration distributions and the Marangoni effects in droplet flow [6,13,44]. Since the surfactant concentration far exceeds the critical micelle concentration, thus, the adsorption of surfactant to the interfacial surface could be finished in a short time, and surfactant distributions and Marangoni effects could be neglected in this work [6,33]. Moreover, the wetting condition can also be neglected in this step T-junction

microchannel. Thus, k can be regarded as a constant for a fixed microchannel. Then, according to Eq. (9), k can be easily obtained by a standard system if we know the value μ_c of the continuous phase and σ of the system.

Moreover, the capillary number Ca_c of the continuous phase and Weber number of the dispersed phase (We_d) can be described by Eq. (10) and Eq. (11), respectively.

$$Ca_c = \frac{u_c \times \mu_c}{\sigma} \quad (10)$$

$$We_d = \frac{\rho u_d^2 l}{\sigma} \quad (11)$$

where μ_c is the viscosity of continuous phase, ρ and l in Eq. (11) are density and characteristic size of the dispersed phase, u_c and u_d can be calculated by Eq. (12) and Eq. (13).

$$u_c = \frac{Q_c}{W \times H} \quad (12)$$

$$u_d = \frac{4 \times Q_d}{\pi d^2} \quad (13)$$

Combining Eqs. (9) and (10), the droplet size can be approximated by a power law that is described as Eq. (14).

$$\frac{d_d}{d_n} \sim Ca_c^{-1/2} \quad (14)$$

Then, according to Eq. (9), the interfacial tension can be gained if the viscosity of the continuous phase and the droplet size are known. If the interfacial tension and the droplet size are known, then the viscosity of the continuous phase can be calculated.

3. Results and Discussion

3.1. Flow regime in the step T-junction microchannel

As illustrated in Fig. 4, the liquid-liquid microflow regime can be mainly divided into squeezing, dripping and jetting flow. Compared with jetting flow, dripping flow has a much stronger stability and the size of the generated droplets has a very narrow distribution. Therefore, we carried out the measurements under the dripping regime in this work. Dripping flow can be observed when $0.01 \leq Ca_c \leq 0.70$ in this step T-junction microdevice. Because the asymmetric flow field with this microdevice strengthen the

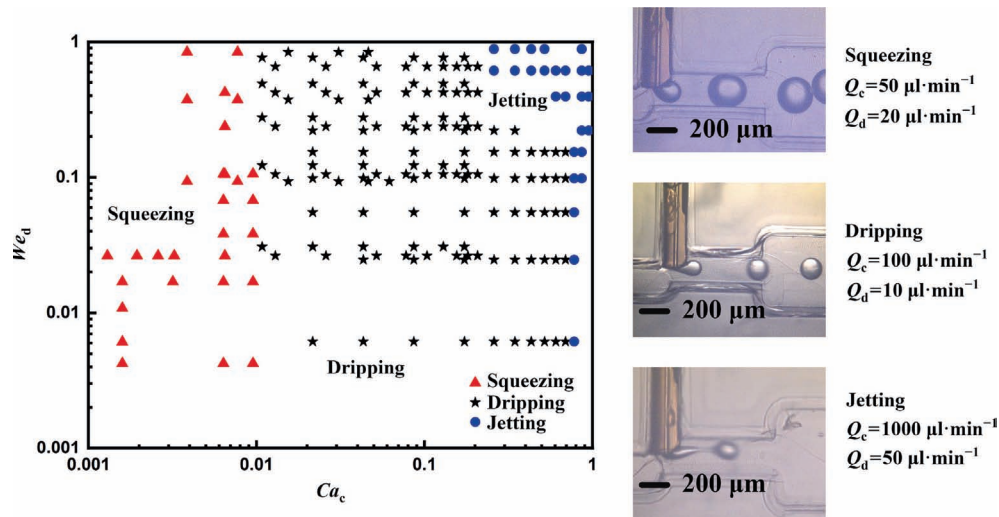


Fig. 4. The flow pattern in this microdevice.

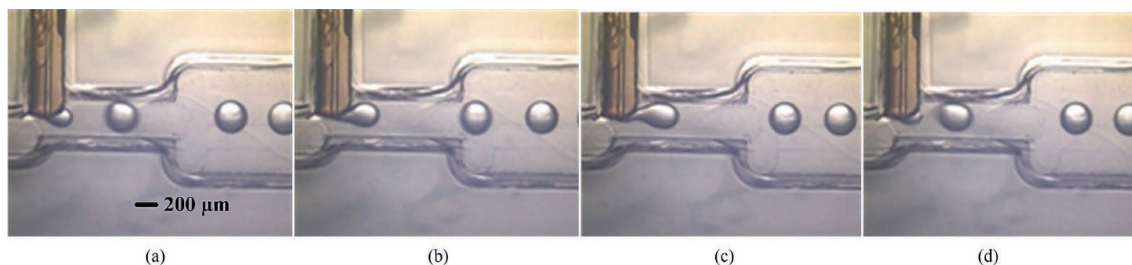


Fig. 5. Process of droplet generation (continuous phase: deionized water-saturated butanol, dispersed phase: deionized water; $Q_c = 100 \mu\text{l}\cdot\text{min}^{-1}$, $Q_d = 10 \mu\text{l}\cdot\text{min}^{-1}$).

shear force during the droplet formation, stable dripping flow can be easily realized even with low interfacial tension systems as shown in Fig. 4. The experimental operating range is much larger than that of other microdevices, such as flow-focusing and T-junction microdevices [43,45]. The wider operating range facilitates the measurement of the interfacial tension in a wider range, even for systems with low interfacial tension.

3.2. Generation of the monodisperse droplets

Before the measurement, it is very important to determine the flow pattern between the two phases, which determines whether Eq. (9) can be used to measure the interfacial tension within the operating range. To ensure an accurate relationship between the

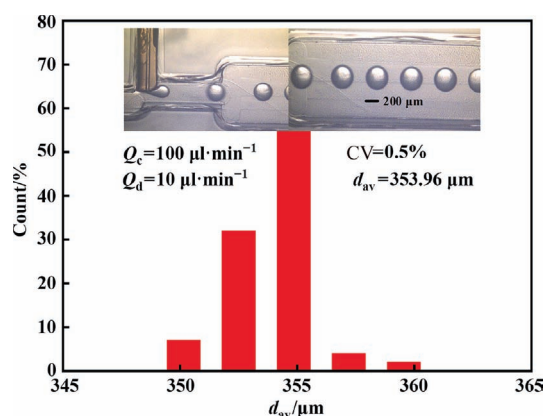


Fig. 6. Distribution of droplet size (continuous phase: deionized water-saturated butanol, dispersed phase: deionized water; $Q_c = 100 \mu\text{l}\cdot\text{min}^{-1}$, $Q_d = 10 \mu\text{l}\cdot\text{min}^{-1}$).

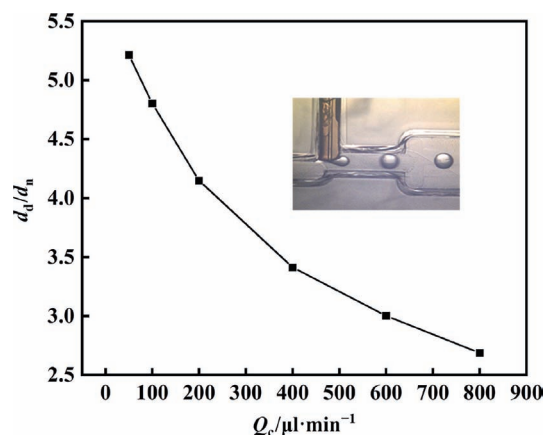


Fig. 7. Effect of the continuous phase flow rate on the droplet size ($Q_d = 5 \mu\text{l}\cdot\text{min}^{-1}$).

average size of droplet and the interfacial tension, *n*-butanol/deionized water was served as the controlled experimental system because of constant interfacial tension. The flow rate of the two phases is a significant parameter in liquid-liquid microdispersion. By changing the two-phase flow rate, monodispersed microdroplets with high uniformity under a dripping flow can be generated as depicted in Fig. 5. Dripping flow can be realized when the

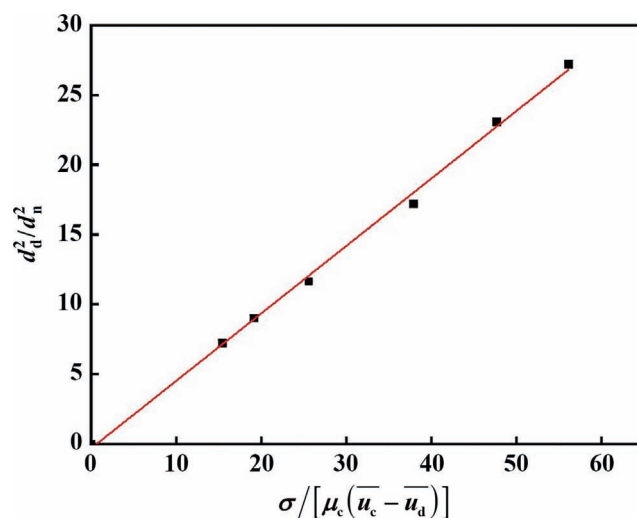


Fig. 8. Determination of the value of k , $(d_d/d_n)^2$ is a function of $\sigma/[\mu_c(\bar{u}_c - \bar{u}_d)]$, the slope is equal to the parameter k .

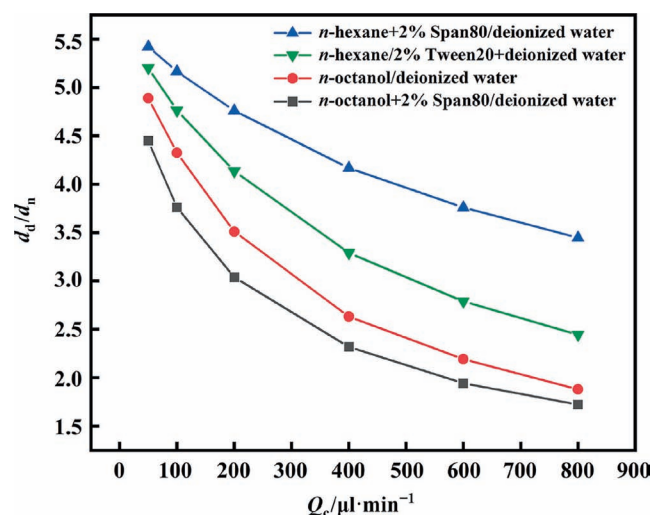


Fig. 9. Effect of the continuous phase flow rate on the droplet size for the different systems. ($Q_d = 5 \mu\text{l}\cdot\text{min}^{-1}$).

dispersed phase flow rate was $5\text{--}50\ \mu\text{L}\cdot\text{min}^{-1}$ and the corresponding continuous phase flow rate was $50\text{--}1400\ \mu\text{L}\cdot\text{min}^{-1}$. The coefficient of variation (CV) of the droplet size is defined as $\text{CV} = \delta/d_{\text{av}} \times 100\%$, where δ is the standard deviation and d_{av} is the average droplet diameter. As shown in Fig. 6, through the statistics of 100 droplets diameter in the main channel near the step structure, the CV value is less than 5%, thus, the droplet generation processes can repeat quite well. The Stable droplets with highly monodispersity, uniformity generation in a wider operating range under dripping flow can provide the fundamentals for the measurements of interfacial tension and viscosity.

3.3. Determination of the value of parameter k

The standard system, deionized water/1-butanol, was used to determine the value of parameter k of the capillary embedded step T-junction microchannel. It is worth noted that 1-butanol and water are mutually saturated which excludes mass transfer during the droplet formation process [46]. Fig. 7 shows that under a given dispersed phase flow rate of $5\ \mu\text{L}\cdot\text{min}^{-1}$, the droplet size decreases with the increase of the continuous phase flow rates. It is because an increase in the continuous phase flow rate means an increase in the shear force, which leads to a continuous decrease in the droplet size.

Combined with Eq. (9), according to the linear fit of $\sigma/[\mu_c(\bar{u}_c - \bar{u}_d)]$ to $(d_d/d_n)^2$, the slope is the value of parameter k , as shown in Fig. 8, and the value of parameter k is 0.483 for the capillary embedded step T-junction microchannel.

3.4. Measurement of interfacial tension σ

Several liquid-liquid systems and an ATP system with different interfacial tension were selected as the working systems. Firstly, four w/o systems (2% (mass) Span 80 in *n*-octanol/water, 2% (mass) Span80 *n*-hexane/water, *n*-octanol/water, and *n*-hexane/2% (mass) Tween 20 in water) with constant interfacial tension were tested. The organic phase was used as the continuous phase, and the aqueous phase was used as the dispersed phase in these working systems. Fig. 9 represents the effects of the continuous phase flow rates on the droplet size at a given dispersed flow rate of $5\ \mu\text{L}\cdot\text{min}^{-1}$. The trends of the four systems are similar when the continuous phase flow rates range from 50 to $800\ \mu\text{L}\cdot\text{min}^{-1}$. Then, Fig. 10 shows $(d_d/d_n)^2$ as a function of $k/[\mu_c(\bar{u}_c - \bar{u}_d)]$, and the slopes of the fitting lines are the interfacial tensions for different systems. The measured interfacial tensions of the four systems are (4.43 ± 0.04) , (2.94 ± 0.10) , (8.20 ± 0.30) , and $(1.68 \pm 0.05)\ \text{mN}\cdot\text{m}^{-1}$, respectively.

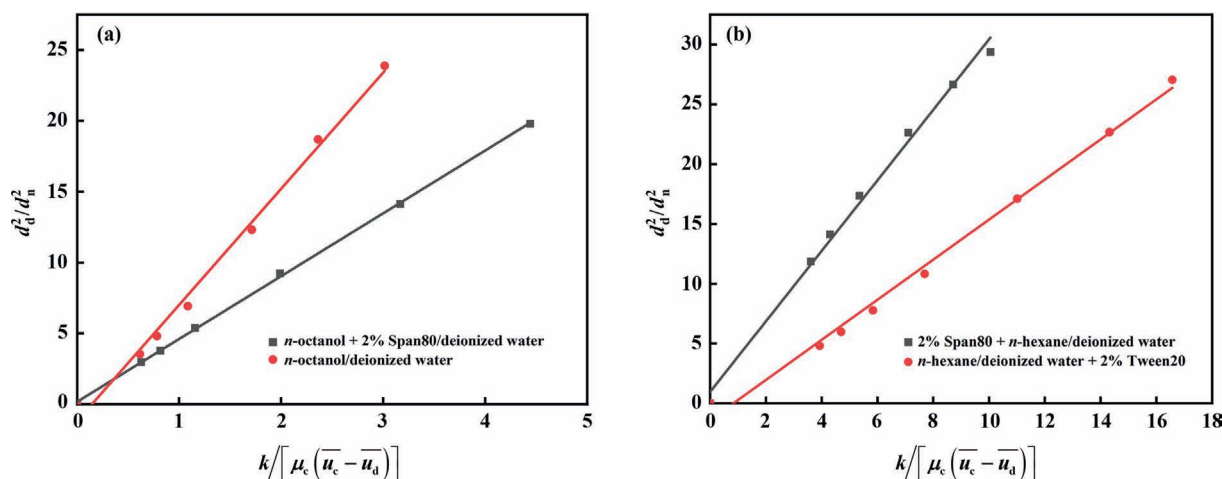


Fig. 10. Determination of the values of σ : $(d_d/d_n)^2$ as a function of $k/[\mu_c(\bar{u}_c - \bar{u}_d)]$. The slope is equal to the interfacial tension σ ($\text{mN}\cdot\text{m}^{-1}$). ((a): black square, 2% (mass) Span 80 in *n*-octanol/water; red circle, *n*-octanol/water; (b): black square, 2% (mass) Span80 *n*-hexane/water; red circle, *n*-hexane/2% (mass) Tween 20 in water).

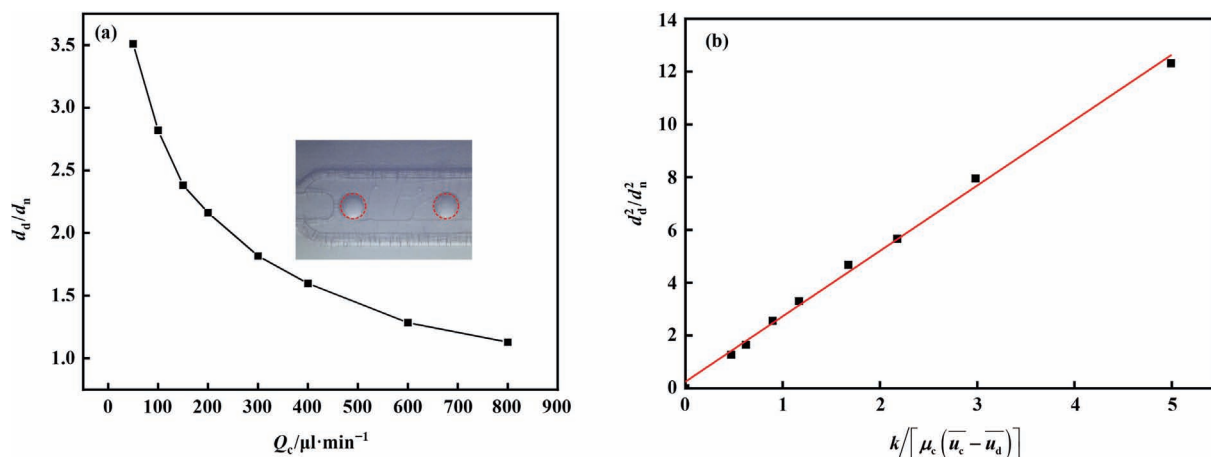


Fig. 11. (a) Effect of the continuous phase flow rate on the droplet size ($Q_d = 5\ \mu\text{L}\cdot\text{min}^{-1}$, ϵ -caprolactam aqueous solution as the continuous phase and $(\text{NH}_4)_2\text{SO}_4$ aqueous solution as the dispersed phase); (b) Determination of the values of σ : $(d_d/d_n)^2$ as a function of $k/[\mu_c(\bar{u}_c - \bar{u}_d)]$. The slope is equal to the interfacial tension σ ($\text{mN}\cdot\text{m}^{-1}$).

Table 2The experimental values of the interfacial tension σ for different systems

System	Dispersed phase	Continuous phase	$\sigma_{\text{exp}}/\text{mN}\cdot\text{m}^{-1}$	$\sigma_{\text{mea}}/\text{mN}\cdot\text{m}^{-1}$	Ref. [13,35]
1	deionized water	2% (mass) Span80 <i>n</i> -octanol	4.43 ± 0.04	4.30 ± 0.07	4.30 ± 0.04
2	deionized water	2% (mass) Span80 <i>n</i> -hexane	2.94 ± 0.10	2.99 ± 0.09	2.80 ± 0.03
3	deionized water	<i>n</i> -octanol	8.20 ± 0.30	8.26 ± 0.06	8.60 ± 0.05
4	deionized water +2% (mass) Tween 20	<i>n</i> -hexane	1.68 ± 0.05	1.65 ± 0.04	1.77 ± 0.03
5	(NH ₄) ₂ SO ₄	ϵ -caprolactam	2.48 ± 0.05	–	–

Then, Caprolactam-(NH₄)₂SO₄-water with mutually saturated could form liquid-liquid two phase system, and was selected as an ATP system to prove the effectiveness and feasibility of this microfluidic method. The microdroplets also can be generated under a dripping flow in the step T-junction microdevice, and then the interfacial tension can be obtained. Fig. 11(a) shows the droplet size of the aqueous two-phase system. According to Eq. (9), $(d_d/d_n)^2$ was fit to a linear function of $k/[\mu_c(\bar{u}_c - \bar{u}_d)]$, and the value of the slope of $(2.48 \pm 0.05) \text{ mN}\cdot\text{m}^{-1}$ is the value of the interfacial tension of the system (Fig. 11(b)).

In Table 2, the experimental data were compared with the data acquired from commercial interfacial tensiometer. It can be seen they are in good consistency. Additionally, the experimental values are in good agreement with the literature reported [13,35]. There-

fore, the effectiveness and accuracy of this equipment to measure interfacial tension have been further proved.

Subscript mea represents the values of the interfacial tension σ were measured by the commercial pendant-droplet interfacial tensiometer

3.5. Measurement of the continuous phase viscosity

To determine the viscosity of the continuous phase, several systems were selected for testing in this work. First, 2% (mass) span 80 in *n*-hexane/deionized water was initially used as the standard experimental system to ensure an accuracy and fundamentals of other system's measurements. Then, we chose chitosan aqueous solution with different mass concentrations used as the dispersed phase and *n*-octanol, with 2% (mass) Span 80 added to stabilize the droplets, as the continuous phase to prove the repeatability of the method. Fig. 12 illustrates that the microdroplets size varies with the continuous phase flow rates at a given dispersed flow rate of $5 \mu\text{L}\cdot\text{min}^{-1}$. Similarly, with increasing continuous phase flow rates, the size of the microdroplets decreases, according to Eq. (9), $k\sigma/(\bar{u}_c - \bar{u}_d)$ is a function of $(d_d/d_n)^2$, and the slope is equal to μ_c (mPa·s). The measurement results are shown in Fig. 13(a). Eventually, we chose 2% (mass) span80 in *n*-octanol/deionized water and *n*-octanol/deionized water to confirm the applicability and suitability of this microfluidic method and the measurements results are shown in Fig. 13(b).

Eventually, we compared the experimental results with the data obtained from the commercial spinning digital viscometer and the results are given in Table 3. From Table 3 we can conclude that both are in good agreement.

Subscript mea represents the values of the viscosity μ were measured by the spinning digital viscometer.

Through this microfluidic equipment, the interfacial tension and the viscosity of different systems were tested with high accuracy, good repeatability and a small sample volume. The proper operating condition range for interfacial tension in this microdevice have been provided and have compared the operating range

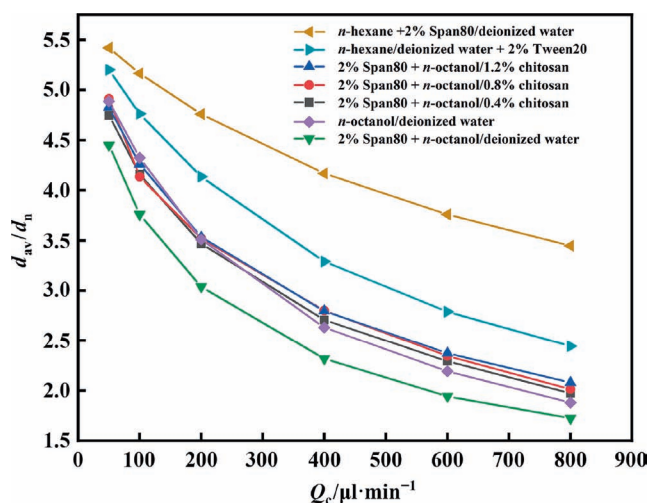


Fig. 12. Effect of the continuous phase flow rate on the relative droplet size for the different systems.

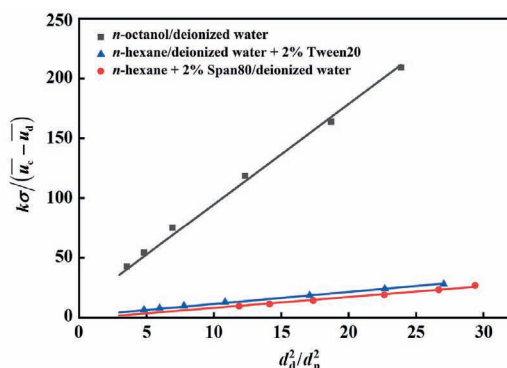
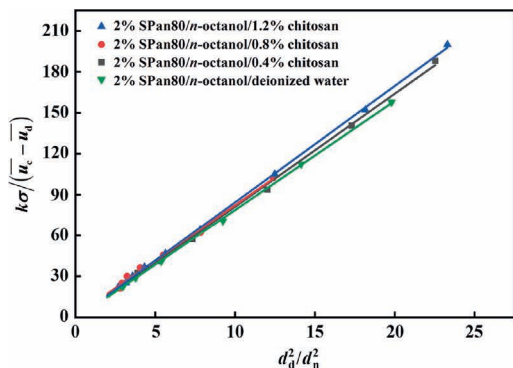


Fig. 13. Determination of the viscosity μ : $k\sigma/(\bar{u}_c - \bar{u}_d)$ as a function of $(d_d/d_n)^2$. The slope is equal to μ_c (mPa·s). ((a) blue triangle, red circle, black square: represents three systems with same continuous phase that 2% (mass) Span80 in *n*-octanol, 1.2, 0.8, 0.4% (mass) chitosan aqueous solution used as dispersed phase; green triangle represents 2% (mass) Span80 in *n*-octanol/deionized water system; (b) black square, *n*-octanol/deionized water; red circle, 2% (mass) span80 in *n*-hexane/deionized water; blue triangle, *n*-hexane/2% (mass) Tween 20 in deionized water).

Table 3

The experimental values of the continuous phase viscosity for the different systems

System	Dispersed phase	Continuous phase	$\mu_{\text{exp}}/\text{mPa}\cdot\text{s}$	$\mu_{\text{mea}}/\text{mPa}\cdot\text{s}$
1	deionized water	2% (mass) Span80 <i>n</i> -hexane	0.90 ± 0.03	0.95
2	deionized water	2% (mass) Tween 20 <i>n</i> -hexane	0.99 ± 0.05	0.91
3	0.4% (mass) chitosan	2% (mass) Span80 <i>n</i> -octanol	8.29 ± 0.14	8.20
4	0.8% (mass) chitosan	2% (mass) Span 80 <i>n</i> -octanol	8.13 ± 0.21	8.20
5	1.2% (mass) chitosan	2% (mass) Span 80 <i>n</i> -octanol	8.54 ± 0.06	8.20
6	deionized water	2% (mass) Span 80 <i>n</i> -octanol	8.01 ± 0.07	8.20
7	deionized water	<i>n</i> -octanol	8.40 ± 0.31	8.06

Table 4

The proper operating range of different microdevices

Methods	Operating range Ca_c	Accuracy	Advantages
pore array micro-structured device [34]	0.007–0.05 [34]	~1%	facile and practical
T-junction [34]	0.005–0.03	~3%	easy to control and accurate
co-axial structure [13]	0.05–0.289	~3%	accurate, facile
this work	0.01–0.7	~5%	repeatability, accuracy, a small volume sample

with other microdevices, the comparison results are shown in Table 4.

4. Conclusions

In this work, we designed a novel microfluidic device that is a capillary embedded step T-junction microchannel to measure the interfacial tension of two immiscible liquids including several different w/o systems and an ATP system. Additionally, the viscosity of the continuous phases was also tested using the same microdevice. All the measurements were based on the force balance between the interfacial tension and the viscous shear force during the droplet generation under the dripping regime. The fabrication of the microdevice and the mathematical derivation process of the measurement fundamentals are provided in this work. Several systems with interfacial tension from 1.0 to 8.0 mN·m⁻¹ and the viscosity of the continuous phase from 0.9 to 10 mPa·s were accurately measured with this microdevice. Then we compared the experimental data with data acquired from the interfacial tensiometer and the spinning digital viscometer, the comparison results show that they are in good agreement. Finally, the accuracy, effectiveness, applicability and suitability of this microfluidic approach used to test low interfacial tension and viscosity have been proved.

CDediT Authorship Contribution Statement

Li Ma: Writing – original draft, Investigation, Methodology. **Yongjin Cui:** Investigation, Validation. **Lin Sheng:** Validation, Formal analysis. **Chencan Du:** Writing – review & editing, Conceptualization. **Jian Deng:** Writing – review & editing, Conceptualization. **Guangsheng Luo:** Writing – review & editing, Conceptualization, Supervision.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Nomenclature

Ca_c	capillary number of the continuous phase
d_d	diameter of droplet, μm
d_n	inner diameter of capillary
F_{pc}	inertia force of the continuous force, N
F_{pd}	inertia force of the dispersed phase, N
H	height of the microchannel, μm
Q_c	volumetric flow rate of the continuous phase, $\mu\text{L}\cdot\text{min}^{-1}$
Q_d	volumetric flow rate of the dispersed phase, $\mu\text{L}\cdot\text{min}^{-1}$
u_c	superficial velocity of the continuous phase, $\text{m}\cdot\text{s}^{-1}$
u_d	superficial velocity of the dispersed phase, $\text{m}\cdot\text{s}^{-1}$
W	width of the microchannel, μm
We_d	weber number of the dispersed phase
w	width of the orifice, μm
μ	viscosity of different system mPa·s
σ	interfacial tension between two phases, $\text{mN}\cdot\text{m}^{-1}$

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