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Development of a new micromixer based on split/recombination for mass production and its application to soap free emulsifier

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Abstract

We fabricated two new types of multi-stage micromixers, YM-1 and YM-2. In these devices, mixing is achieved through the repeated splitting and recombination of the fluid flow. It was shown that the two types of micromixers presented here were capable to produce soap free emulsions at short contact times. From the investigation of the effects of the device wall material, the number of stages, etc. on the size of the droplets in the resulting emulsion, it was clarified that the emulsion was formed through efficient mixing achieved by utilizing the interactions which uniquely occur in microspaces, particularly between the device wall and fluid flow. The attained maximum production rates of emulsion in experiments were 5 l/h for YM-1 and 20 l/h for YM-2, respectively. To show the applicability of the mixers to other purposes, rapid extraction of phenol from dodecane by water was also conducted. It was found that YM-1 has a potential to continuously extract phenol even at extremely high flow rates (mixing times within 1 s). These results show that, although the mixers are extremely small, they have the capability to be used in fairly large-scale industrial plants.

Keywords: Micromixer; Splitting and recombination; Soap free emulsion

1. Introduction

The rapid production of a stable emulsion without using a surfactant is very attractive for the efficient extraction of valuable chemicals and the production of high performance materials such as dispersions of various pigments, nano-particles, and various polymers. Micromixing may become a promising approach to achieve such processing, if well-designed micromixers which take advantage of the unique dynamics of fluids in microspaces, are developed. It is, therefore, essential to understand the scaling effect induced by size reduction. Table 1 lists how the relative magnitudes of transport properties and forces change according to size. It can be noticed that in microspaces, the viscous force and interfacial tension become relatively dominant when compared with inertial force. Particularly, the ratio of interfacial tension to inertial force increases up to 10^{18} when sizes are reduced to the dimension of micron order. This suggests that operations utilizing interfacial properties, such as the production of emulsions, can be drastically improved by utilizing microspaces.

The mixing of two immiscible liquids for the production of an emulsion usually requires a lot of energy. Since the interfacial tension between liquids become dominant and also the volume of liquid droplets is restricted in microspaces, it is expected that the efficient production of emulsions can be achieved just by simply mixing two fluids in a microchannel. Indeed, Heinichen and Natelberg reported the development of a micromixer which showed a higher mixing efficiency when compared with a conventional stirred tank [1]. From this viewpoint, several attempts have been made for the development of new micromixers. The key factor for rapid emulsion production in a micromixer is to efficiently break down the fluids into small segments, and to let the formed segments efficiently make contact with each other. IMM GmbH has already developed a new type of interdigital micromixer for emulsion production in which two components divided into many streams are mixed with each other [2–5]. The IMM mixer consisted of layer type mixing elements which contain 18 sinusoidal-shaped fluid channels for each fluid with a width of 25 or 40 μm. Using this interdigital micromixer, they showed its high ability to produce emulsions

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Table 1
Scaling effect of transport properties

	nm	μm	mm	m
Length (L)	10^{-9}	10^{-6}	10^{-3}	1
Surface area (L^2)	10^{-18}	10^{-12}	10^{-6}	1
Volume (L^3)	10^{-27}	10^{-18}	10^{-9}	1
Specific surface area (L^{-1})	10^{9}	10^{6}	10^{3}	1
Rate ($\propto L$)	10^{-9}	10^{-6}	10^{-3}	1
Inertial force ($\propto L^4$)	10^{-36}	10^{-24}	10^{-12}	1
Viscous force ($\propto L^2$)	10^{-18}	10^{-12}	10^{-6}	1
Interfacial tension ($\propto L$)	10^{-9}	10^{-6}	10^{-3}	1
Viscous force/inertial force ($\propto L^{-2}$)	10^{18}	10^{12}	10^{6}	1
Interfacial tension/inertial force ($\propto L^{-3}$)	10^{27}	10^{18}	10^{9}	1

by successfully producing water–silicone oil emulsions having various droplet sizes by controlling the input flow rates of the two fluids [4]. The IMM mixer was also applied to the formation of cream [5]. Herwech et al. piled up four layers of plates, the 2nd layer being a broom-like mixer composed of 15 microchannels with widths of 60 μm and depths of 150 μm [6]. They visualized the flow pattern in the mixer, and clarified that the flow rate significantly affected the properties of the emulsion. Eventually, they showed that the device could produce an emulsion, which droplets are 190 μm in size, at a flow rate of 400 ml/h by mixing silicone oil and water. Schwesinger et al. developed a micromixer resembling a static mixer, based on multiple flow splitting and recombination [7]. They reported that a W/O emulsion, which droplets were 500 μm in size, could be produced by using five mixing units, and that the droplet size could be reduced by increasing the unit number to 16–20. They also developed a new device composed of a main channel and a partial channel to be used as an emulsifier [8].

Several attempts have also been made to produce mono-dispersed droplets. As mentioned above, the effect of interfacial tension and viscous force are significantly dominant in microspaces. Sammarco and Burns microfabricated a new device which takes advantage of these features, and produced discrete droplets by interval pumping [9]. Sugiura et al. have developed an original emulsifier with a special microhole of 17.5 μm in width [10]. The fluid of the dispersion phase was enforced as to penetrate into the flow of the continuous phase through the rectangular penetration hole. Droplets were formed in the continuous stream as the ratio of interfacial tension to inertial force was extremely high, and mono-dispersed droplets of various vegetable oil fats, 32.5 μm (1.5% of variation coefficients) in diameter, were successfully produced in a water flow of 10 ml/h.

Thus, several remarkable new concepts for efficient mixing using microspaces have been presented. However, the development of new micromixers which can be used for mass production must also be challenged. In microchemical processing, the numbering-up concept is usually applied for mass production. Stable and precise operation of the devices is required for numbering-up, but is usually difficult because of the high sensitivity of the microdevices. Therefore, the development of a microdevice which has the stability to be easily numbered-up is also desired. In addition, a microdevice can be directly attached to conventional macroscale equipment if it has a fairly high processing capacity. The development of such devices will certainly accelerate the popularization of microdevices in the industrial world. Returning to the production of emulsion, a surfactant is inevitable when a conventional macroemulsifier is used. A soap free emulsion is very attractive, especially when an environmentally benign emulsion is desired. As the interactions between fluid and wall is dominant in microspaces, the electric charge of the fluid induced by friction between fluid and wall may be utilized to form an emulsion. From these observations, we fabricated two types of multi-stage micromixers for mass production, based on repeated splitting and recombination of the fluids. Using these micromixers, we examined the effects of device wall material, number of stages, and velocity of fluids on the formation of emulsion to clarify the basic potential of the micromixers, and then verified their performances as on-site devices for rapid surfactant-free emulsion production and rapid extraction.

2. Experimental

2.1. Fabrication of micromixers

Two types of micromixers, YM-1 and YM-2, were fabricated as shown in Fig. 1. Both mixers consist of 11 stages of mixing elements, where splitting and recombination of the fluids simultaneously occur in each element. Stainless steel, aluminum, and acryl resin versions of both mixers were prepared. The mixing element of YM-1 consists of two inflow holes and two outflow holes of 400 μm fabricated by micromachining. The lowest stage has 11 mix elements, and the number of elements in each stage was decreased one by one as the stage number increases. As shown in Fig. 2(a), in each element, two liquid flows enter the element from the two inflow holes, and are respectively split into two streams. These four streams are recombined into two streams which respectively flow out of the two outflow holes. These two streams

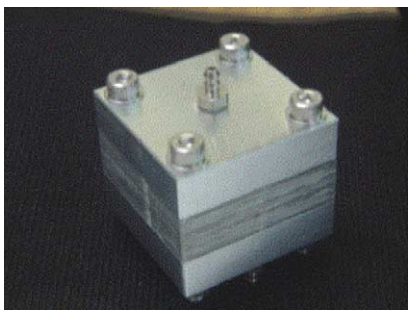
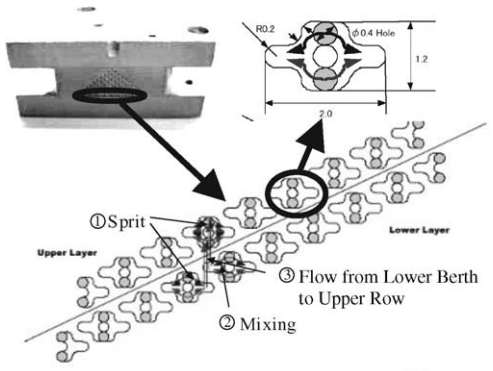
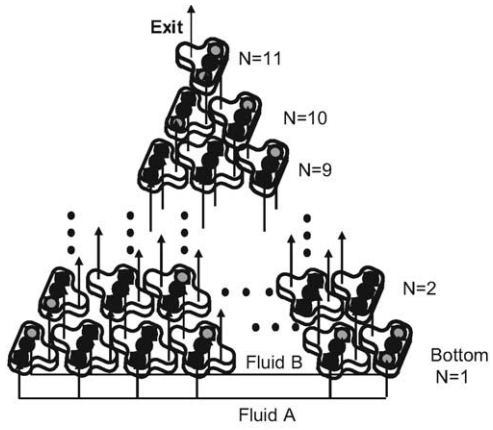


Fig. 1. Overview of micromixer.



(a) Structure of element in YM-1



(b) Flow pattern in YM-1

Fig. 2. Structure of micromixer, YM-1.

are separately provided to two neighboring elements of the upper stage. As shown in Fig. 2(b), a regulating flow was created from the bottom to the top by repeating the motions described above, and the two liquids were finally mixed into one stream at the exit of the mixer. YM-2 is also composed by the same mixing element but the number of elements, which number was 36, was constant in each stage as shown in Fig. 3. Two liquids were respectively distributed to the 1st stage through the inlet sheet, and splitting and recombination of the flows as in YM-1 were repeated in each element up to the 11th stage, and the flows were finally gathered as to flow out of the center exit of the top outlet sheet.

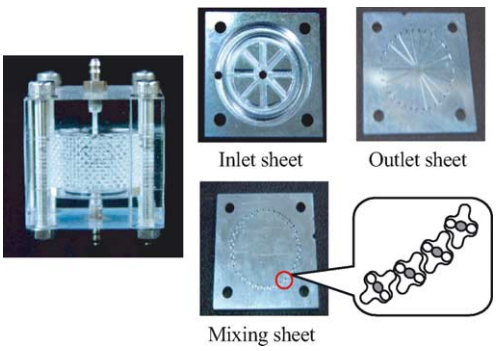


Fig. 3. Structure of micromixer, YM-2.

2.2. Production of emulsion using micromixers

Salad oil and distillated water were supplied to the micromixers using two sets of microsyringe pumps (IC3210, Kd Scientific Co.) at 293 K. The flow rates of oil and water were, respectively, changed in the range of 0.06–1.2 l/h and 0.6–3.6 l/h for YM-1, 0.06–6 l/h and 0.6–12 l/h for YM-2, and 0.06–0.36 l/h and 0.06–0.36 l/h for IMM single mixer. Emulsions were produced using micromixer YM-1 made of different materials (Stainless steel, aluminum, and acrylic resin), and micromixer YM-2 having different stage numbers, in order to clarify the mechanism of emulsion forming.

2.3. Measurement of the properties of the emulsion

The dispersion was observed by means of a digital microscope (VH-8000, Keyence, Japan). The number density distribution of droplet size was estimated from the microphotograph by Harverkamp's method [4]. The ζ-potential of the emulsion immediately after production was also measured by the laser-Doppler method with 10 mW He–Ne laser using a Zeta Potential Analyzer (Otsuka Electronics Co. Ltd., ELS-800).

3. Results and discussion

3.1. Performance for emulsion production

First, we examined whether the micromixer, YM-1 made of aluminum, had a potential to produce a soap free emulsion or not. Fig. 4(a) compares the changes of the emulsification behavior of YM-1 when the flow rates were varied. The oil concentration was fixed to 10 wt.% in this series of experiments. An oil in water emulsion did not form at low flow rates, but stable emulsions were formed without a surfactant when the total flow rate exceeded 1.2 l/h. This clearly shows that a high flow rate is required to produce a sufficiently uniform emulsion. When the total flow rate was 4.5 l/h, the mixing time of the micromixer was about 0.3 s. Fig. 4(b)

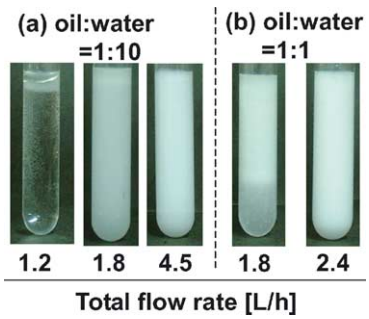


Fig. 4. Photograph of emulsion produced at various flow rates by YM-1.

shows the emulsions obtained when the oil flow rate was increased to be equal with the water flow rate. The emulsion became cloudy as the oil flow rate was increased. Thus, an equivalent mixture of oil and water can also be emulsified without a surfactant at a high flow rate that only allows 0.1 s of mixing time. The same results were obtained in experiments using YM-1 made of stainless steel. From these results it was confirmed that the YM-1 type mixers made of metal have a potential to continuously produce emulsions at short mixing times.

On the other hand, emulsions could be produced by YM-2 made of aluminum only at extremely high flow rates. The maximum production rates of emulsion achieved in this work were, respectively, 5 l/h for YM-1 and 12 l/h for YM-2, under 0.2 MPa of the discharge pressure. This means that the capacities of the mixers for emulsion production is several to a hundred tons per year, and therefore they can be utilized as efficient mixers in mass production systems.

3.2. Size distribution of droplets produced by YM-1 and YM-2

Considering there usages, the size distributions of droplets in emulsions are extremely important. We, therefore, examined the size distribution of droplets in the emulsions produced by YM-1 and YM-2 made of aluminum. Fig. 5 shows the effect of the total flow rate on the number density distribution of size of droplets in emulsions produced at different oil concentrations using YM-1. The distributions were estimated from microphotographs of the emulsions following Harverkamp's method [4]. For all oil concentrations, emulsions could be formed only when the total flow rate exceeded 1.8 l/h. This means that a high flow rate is required to form emulsions when YM-1 is used. With the increase in the total flow rate, the distribution, which was initially bimodal, shifted to a smaller size region, and finally became almost constant and unimodal as shown in Fig. 5(a) and (b). From these results it was assumed that the large droplets were broken down into smaller ones by increasing the flow rate. These results strongly suggest that the flow rate is a crucial factor to make a stable emulsion by YM-1.

Interestingly, the peak droplet size of the constant distribution attained at high flow rates decreased with the decrease in oil concentration. Their values were ca. 0.08, 2, and 40 μm for 10, 20, and 50% oil concentrations, respectively. This tendency coincided well with the results reported previously [3–5]. Thus, it was verified that the YM-1 mixer shows an excellent performance for the mass production of emulsions within a few seconds, without using a surfactant. In addition, the droplet size of the emulsion could be easily controlled by simply adjusting the total flow rate.

3.3. Influence of the number of stages

Since the flow of the mixture of oil and water were split and recombined at every stage in YM-1 and YM-2, the size distribution of droplets is thought to be affected by the total number of stages. Therefore, we performed the emulsification at 10% of oil concentration by changing the total number of stages from 1 to 11 using YM-2 made of aluminum. YM-2 was used in this series of experiments, as the velocity of liquids in each stage of this mixer is constant, and therefore the effects of the total number of stages (if there is any) could be easily distinguished. Fig. 6 shows the effect of the total number of stages, *N*, on the number density distribution of size of droplets under the total flow rate of 12 l/h. An emulsion with a bimodal size distribution of droplets having peaks at 2 and 60 μm was obtained when a single stage mixer was used. As the number of stages were increased, the droplets of ca. 60 μm size disappeared, and then smaller droplets having sizes around 2 and 17 μm increased. The size distribution of droplets in emulsions produced using five mixing stages was almost identical to that of emulsions produced using 11 stages, suggesting that five stages are enough to obtain a stable emulsion under this condition. These results show that the two immiscible liquids were split and recombined in the mixer and the distribu-

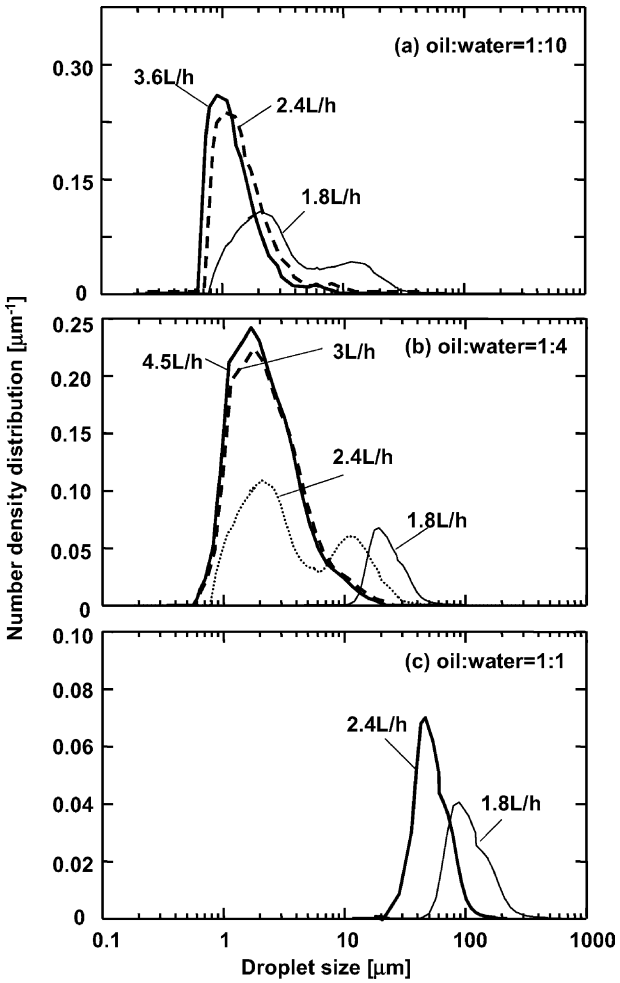


Fig. 5. Effect of the total flow rate and oil concentration on the distribution of droplet produced by YM-1.

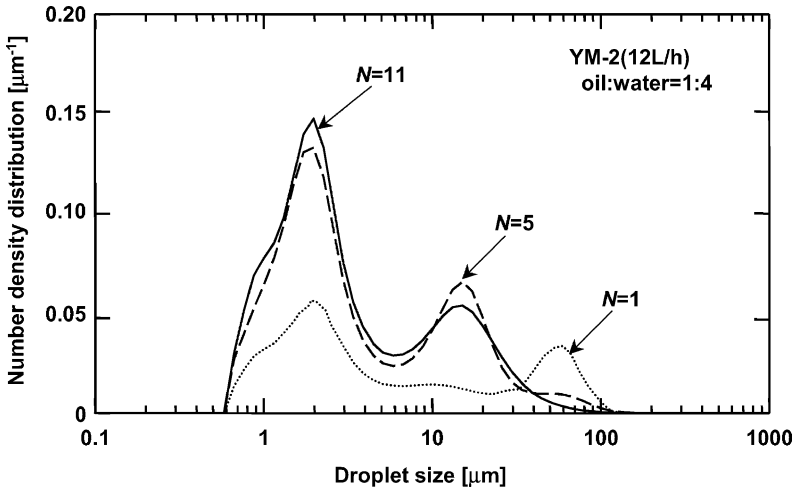


Fig. 6. Effect of the number of stage on the size distribution of droplet produced at 12 l/h and 20% of oil concentration by YM-2.

tion could be changed just by changing the total number of stages.

3.4. Comparison of the emulsification abilities of YM-1, YM-2, and IMM mixers

Next, we compared the emulsification abilities of YM-1, YM-2, and IMM mixers. Fig. 7 shows the number density distributions of droplet size of emulsions produced at high flow rates using aluminum YM-1, YM-2 and IMM mixers. The oil concentration was fixed to 20 wt.% in this series of experiments. A narrow size distribution having a peak at around 2 μm was obtained by using YM-1 at a total flow rate of 4.5 l/h. This distribution was almost equivalent to that obtained by using the IMM mixer at a total flow rate of 1.5 l/h. It should be noted that the channels in YM-1 are much larger than those in the IMM mixer, and the flow rate was also ca. 3 times larger than that in the IMM mixer. On the other hand, the size distribution of droplets shifted to a larger size region when the emulsion was produced at a total flow rate of 12 l/h using YM-2. Since the number of elements at the outlet of YM-2 was 36, this means that the area of outlet cross-section was 36 times larger than that in YM-1. Considering the flow rates and the outlet cross-sections of YM-1 and YM-2, the apparent fluid velocity of the outlet flow in YM-2 is ca. one-tenth of that in YM-1. This is thought to be the reason why YM-2 gives a larger distribution. We could not perform experiments at higher flow rates using YM-2 because of the capacity of the pump. These results suggest that the velocity in the mixing zone as well as the number of stages is a crucial factor to obtain a uniform emulsion.

Thus, a soap free emulsion with droplets of similar sizes could be produced by both YM-1 and IMM mixers irrespective of their different structures. To clarify the reason, we compared the flows in both micromixers from the viewpoint of device structure. The hydraulic equivalent diameter of a

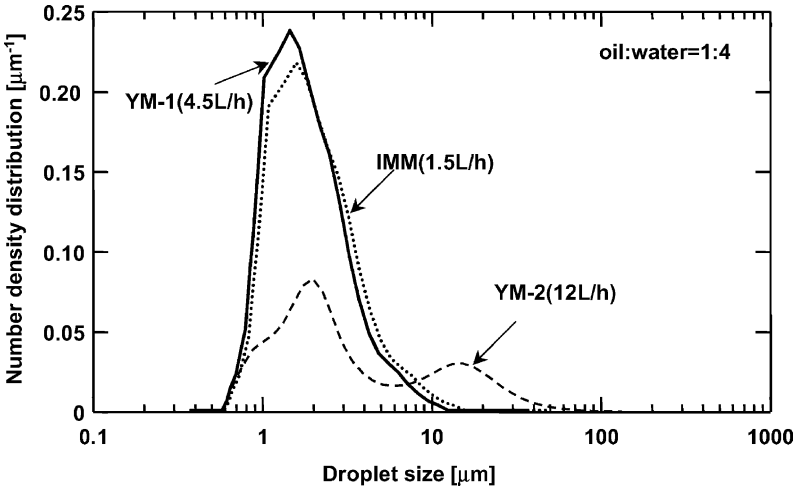


Fig. 7. Comparison of the size distribution of droplet produced at 20% of oil concentration among three micromixers.

single mixing element of YM-1 is 400 μm. The structure of all of the mixing elements are identical, but the number of mixing elements decreases with the increase in stage number, and becomes one at the top stage. Namely, the flow velocity increases with the decrease in the total cross-sectional area. Since the hydraulic equivalent diameter of the IMM mixer is 40 μm, the cross-sectional area of the channels inYM-1 is significantly larger than those in the IMM mixer. The structure of each element in YM-1 is equivalent to two repeated units of splitting and recombination in static mixers. Furthermore, the number of elements decreased one by one at every stage in YM-1. According to the model of static mixer, the cross-sectional area of the oil segment at the *N*th stage from the bottom, S_N, can be estimated by the following equation:

$$S_N = A\left[\frac{Q_o}{Q_o + Q_w}\right]\left(\frac{N/11}{2^{N-1}}\right) \quad (1)$$

where Q_o and Q_w are the flow rates of oil and water, respectively, and A is the total cross-sectional area of the bottom stage. From Eq. (1) we can estimate the stage number in YM-1, N, at which the cross-sectional area of an oil segment becomes equivalent to that of the IMM single mixer. The cross-sectional area of an oil segment in IMM, S_{IMM}, is calculated as follows:

$$S_{IMM} = A_{IMM}\left[\frac{Q_o}{Q_o + Q_w}\right] \quad (2)$$

where A_{IMM} is a total cross-section area of a microelement in IMM. By combining Eqs. (1) and (2), we can obtain the ratio of cross-sectional areas of oil segments in the *N*th stage from the bottom in YM-1 to that in the IMM mixer as follows:

$$\frac{S_N}{S_{IMM}} = \left(\frac{A}{A_{IMM}}\right)\left(\frac{N/11}{2^{N-1}}\right) = 166.6\left(\frac{N/11}{2^{N-1}}\right) \quad (3)$$

Eq. (3) tells us that when $N = 4.81$, S_N/S_{IMM} becomes unity. This means that YM-1 equipped with 4 or 5 stages can produce an emulsion equivalent to that obtained using the IMM mixer from the viewpoint of segment size. The velocity of liquid is an important factor as mentioned above. Comparing the average velocity at each stage in YM-1 with that in the IMM mixer, the velocity at the 5th stage of YM-1 was almost the same as that of the IMM mixer. Judging from the result that the size distribution did not change beyond a certain flow rate, the velocity at the 5th stage was enough to attain to an ultimate size distribution. Thus, YM-1 has a potential to make a fine emulsion irrespective of its large microchannel. Moreover, the structure of YM-1 has other advantages such as follows: the degree of mixing of the fluid can be easily controlled by changing the number of mixing element sheets with a fixed hydraulic equivalent diameter, and this mixer can be operated at high flow rates because of its relatively little pressure drop.

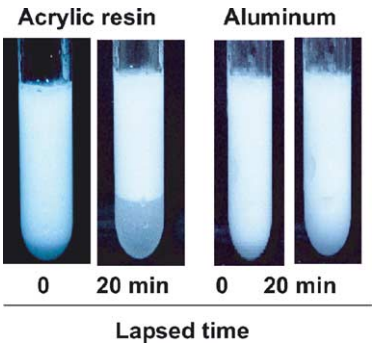


Fig. 8. Effect of wall material type on the stability of emulsion.

3.5. Presumed mechanism for the formation of a soap free emulsion using a micromixer

It was shown that a soap free emulsion could be made by YM-1, but it is questionable how the emulsion can stabilize without a surfactant. To clarify the reason, we examined the effects of wall materials on emulsion formation. We made two types of YM-1, one made of aluminum and the other, acrylic resin, and produced soap free oil–water emulsions at feed rates of 0.6 l/h of oil and 3.6 l/h of water. Fig. 8 shows the photographs of the emulsions obtained using both mixers, just after emulsion formation and after a lapse of 20 min, respectively. The emulsions produced by the mixer made of aluminum were stable after 20 min from formation. On the contrary, as for the emulsion produced by the mixer made of acrylic resin, creaming initiated just after formation and half of the volume was separated into two phases after 20 min. The phase separation of an unstable emulsion is controlled by three factors: creaming, aggregation, and union. Creaming is the rising (or sinking) of droplets induced by the density difference of the continuous phase and the dispersed phase, aggregation occurs when the oil droplets tend to form a community, and union indicates the fusing of several small droplets to a big single droplet. Fig. 9 compares the microphotographs of droplets in the emulsion produced by both mixers. The droplets made from the aluminum mixer were fine and highly dispersed. On the contrary, the emulsion made from the acrylic resin mixer contained large droplets, which was formed by aggregation/unionization. From these results, it was found that the wall material strongly influenced on the stability of the droplets.

Since the production conditions were same except of the wall material for both mixers, it is assumed that the stability of emulsion was affected by the interactions between device wall and fluids. To examine quantitatively the effect of such interactions, the ζ-potential values of emulsions made using the aluminum YM-1 mixer were measured at several conditions as listed in Table 2. The ζ-potential values of the emulsion made with and without a surfactant using the IMM mixer are also listed for comparison. The absolute value of ζ-potential became large as the flow rate of oil

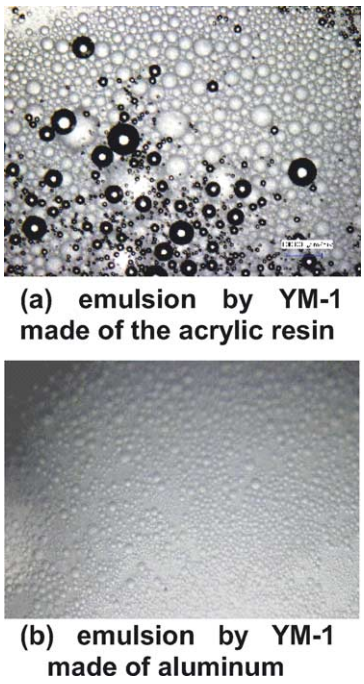


Fig. 9. Comparison of droplet size produced by aluminum mixer with that by acrylic resin mixer.

was increased, and was comparable with that of the emulsion made with a surfactant. It was found that the ζ-potential value could be used to estimate the stability of emulsions made by YM-1. On the other hand, the ζ-potential value of the emulsion made by acrylic rein mixer was nearly zero even at high flow rates. This suggests that at high velocities, an electric double layer is formed at the oil surface by the friction between aluminum wall and oil, resulting in the stabilization of the droplet. From this observation, we can presume that a soap free emulsion is formed at a short contact time in a microchannel through the mechanism as follows: Although the friction between wall and fluid is well recognized for a macrotube, there is almost no effect on the bulk properties of the fluid because the volume of fluid affected by wall friction is very small. On the other hand, in a microchannel, mixing is achieved through the contact of small oil and water segments. Under this situation, the total volume of oil segments charged by wall friction is significantly large, therefore, the oil segments reflect the bulk property of oil. These small charged oil segments encounter small water segments in the microchannel and a stable emulsion is easily formed. Thus, it was shown that the mixing of microsegments enhanced not only the mixing efficiency but also the interfacial properties between oil and water, both of which lead to the efficient formation of emulsions.

Table 2
ζ-potential of the emulsion

Mixer	Oil (l/h)	Water (l/h)	Surfactant	ζ-potential (mV)
IMM	0.03	0.6	SDS	−59.5
IMM	0.12	1.2	None	−58.6
YM-1	0.09	3.6	None	−20.3
YM-1	0.36	3.6	None	−74.8

3.6. Application of micromixer for solvent extraction

Since micromixing realizes a large interfacial area, liquid–liquid extraction could be performed efficiently [11,12]. As shown above, a soap free emulsion can be produced by YM-1 made of metal. Since the size of the oil droplets is extremely small and therefore the interfacial area between oil and water is extremely high, effective extraction between the oil and water phases can be expected just by passing the liquids through the mixer. To examine the performances of the micromixers for the rapid and continuous extractions of phenol in dodecane by water, experiments were conducted using YM-1 made of aluminum and the IMM mixer. Fig. 10 shows the change in the phenol concentration in aqueous phase at the outlet of mixer with the total flow rate during the extraction of phenol in 3.0×10^{-2} M phenol/dodecane. The phenol concentration in aqueous phase increased up to a constant concentration of the equilibrium state with the increase in the flow rate. Comparing the performance of both mixers, the flow rate at which the phenol concentration reached equilibrium for YM-1 was larger than that for the IMM mixer. The flow rate required to attain equilibrium was 2.4 l/h for YM-1 and was much higher than the value 0.3 l/h for the IMM mixer. A high flow rate was required for YM-1 to form sufficiently small droplets, as shown in Fig. 7. Small droplets are necessary to obtain a sufficiently large interfacial area. The increase in the interfacial area brought about the increase in the flux of mass transfer, resulting in the equilibrium extraction. The contact time to reach a maximum extraction yield was ca. 0.3 s for YM-1. Thus, it is shown that YM-1 can be applied for rapid extraction and has a high potential to conduct extraction continuously at extremely

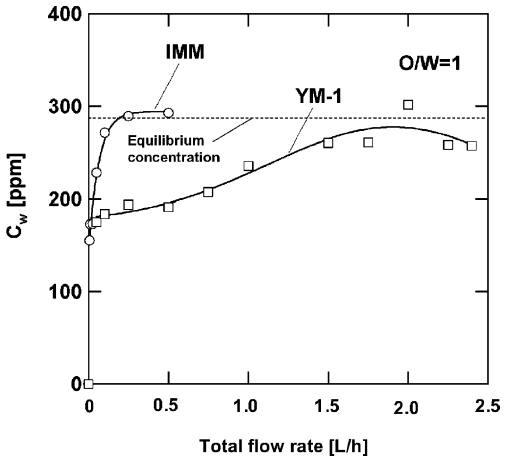


Fig. 10. Effect of total flow rate on the extraction yield of phenol with both micromixers.

short contact times (several milliseconds). In addition, YM-1 can also respond flexibly to various extraction conditions just by simply changing the number of its mixing stages.

4. Conclusion

Two types of micromixers were fabricated by micromachining. Both mixers consisted of 11 sheets of mixing elements. The continuous and simultaneous splitting and recombination of fluids proceeds in each mixing element. To examine their mixing performances, we tried to produce a soap free emulsion from oil and water with several flow rate ratios. It was shown that both micromixers showed high performances for the production of soap free emulsions at short contact times. The distribution of oil droplet size shifted toward the smaller size region with the increase in the total flow rate and the decrease in the oil concentration. The maximum production rates of emulsion achieved in the experiments were 5 l/h for YM-1 and 20 l/h for YM-2, respectively. By examining the effects of device wall material, flow rate, and oil concentration on the size of droplets and the stability of the resulting emulsion, it was clarified that the emulsion was formed by the efficient mixing of small segments of the fluids and the interaction between metal wall and oil. It is expected that the proposed mixers can be used for industrial purposes, such as large-scale extraction.

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