

# Experimental Approaches for Understanding Mixing Performance of a Minireactor

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*Three experimental approaches are presented to determine the mixing characteristics of a new kind of high-efficiency membrane dispersion minireactor in which microfiltration membranes are applied as the dispersion media. Residence time distribution (RTD) curves were measured for determining the macromixing characteristics. A typical Dushman reaction (iodide–iodate) coupled with a neutralization and precipitation reaction of  $\text{BaSO}_4$  were introduced to characterize the micromixing performance of a single-phase mixing process. A dye extraction method was also applied to study the micromixing performance of a two-liquid phase-mixing system. The RTD result showed that the flow performance in the minireactor was almost in the plug flow condition. The micromixing performance was expressed with a segregation index, which could be  $<0.002$ . The single-phase micromixing performance reached the desired level. The result of precipitation of  $\text{BaSO}_4$  showed that the mixing performance had a substantial influence on the particle size and size distribution. It was found that in the single-phase mixing process the mixing performance was mainly influenced by the phase flux and the membrane pore size. With decreasing dispersed fluid flux or the membrane pore size, or with increasing continuous fluid flux, the micromixing performance was enhanced. The dye extraction method can correctly determine the mixing performance of a two-liquid phase-mixing system. Unlike the single-phase mixing process, in the two-liquid phase-mixing process there was a minimum value for the mixing efficiency with the change of the continuous- or dispersed-phase flux, when the membrane pore size was  $>0.9 \mu\text{m}$ . © 2005 American Institute of Chemical Engineers AIChE J, 51: 2923–2929, 2005*

**Keywords:** membrane dispersion, micromixing, macromixing

## Introduction

Mixing, a widely used unit operation process in the chemical industry, has a decisive impact on the overall performance of the processing engineering. Mixing can be classified as micromixing (mixing at the molecular scale), mesomixing, and macromixing. Many methods have been developed over the last few years to describe the mixing performance in different devices. The most popular of broadly considered methods of

measuring macromixing performance is to elucidate the residence time distribution (RTD). Residence times and residence time distributions are important characteristics for all chemical reactors because they provide information about macromixing and the flow behavior in the respective reactors.<sup>1,2</sup> Some authors<sup>3–6</sup> have reviewed the characteristics of micromixing and its available measurement methods. Almost all the developed methods are based on mixing-sensitive chemical conversions, and can be classified into three main schemes: single reaction ( $A + B \rightarrow R$ ), consecutive competing reactions ( $A + B \rightarrow R$ ;  $R + B \rightarrow S$ ), and parallel competing reactions ( $A + B \rightarrow R$ ;  $C + B \rightarrow S$ ). In the latter two schemes, yield and/or selectivity, achieved for specific reaction products, constitute the param-

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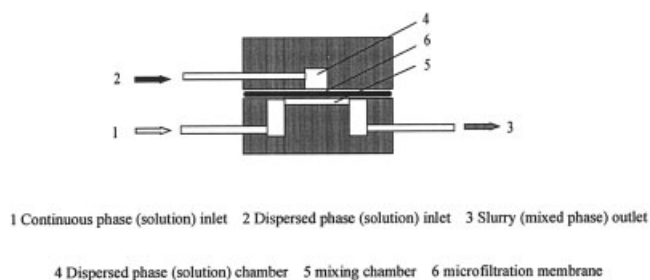


Figure 1. Minireactor.

ters for quantifying the mixing performance, of which the “Villermaux/Dushman method” has gained wide acceptance. The published results show that the micromixing performance can be determined very well when the volume ratio between the two miscible solutions is very large. On the other hand, most of the methods are designed to describe the single-phase mixing process in which one miscible solution is mixed into another solution. For the multiphase-mixing processes only few results can be referenced at the present time. In 2004 a dye extraction method<sup>7</sup> was developed to explain a liquid–liquid mixing process, which is based on the phase transfer of the solvatochromatic dye Nile red from a water/methanol phase to a heptane phase.

Over the past decades, microstructured devices have triggered an explosion of scientific and industrial interest. The rapid development of microchemical systems has led to a considerable variety of microstructured devices, especially for microreaction and micromixing processes. Many researchers focused on the micromixing performances in different microstructured micromixers.<sup>7–11</sup> The micromixing characteristic is considered to be the basis for development of new microchemical systems. Ehrfeld and coworkers<sup>8</sup> studied the micromixing performance in several micromixers with a competing reaction system, and compared it with that in the traditional macroscopic mixing units. Panić et al.<sup>7</sup> provided an improved experimental protocol for the Dushman reaction by controlling the final pH value in the reactors, which supplied more reliable experimental data than those in Ehrfeld et al.<sup>8</sup> They also described the mixing performance of five micromixers whose mixing principle and internal structure geometry were different. Two experimental methods of competing parallel reaction and dye extraction were introduced.

In our laboratory a new kind of microstructured device, designated *membrane dispersion minireactor*, was developed, in which microfiltration membranes are applied as the dispersion media.<sup>12–16</sup> In the reactor, single-phase mixing and multiphase-mixing processes can be successfully performed. When a liquid–liquid mixing process is carried out, the dispersion phase is dispersed as small droplets into the continuous phase within a very short time. Previous works demonstrated that this type of minireactor, like most of the microstructured devices, has the characteristics of high mass transfer efficiency, large capacity, low energy cost, and controllability.<sup>14</sup> The membrane dispersion minireactor has been used in liquid–liquid extraction processes, such as oil deacidification,<sup>15</sup> extraction of citric acid and succinic acid,<sup>14,16</sup> and in the preparation process of nanoparticles such as BaSO<sub>4</sub><sup>12</sup> and TiO<sub>2</sub>.<sup>13</sup> However, the mixing performance of the new device has not been tested. To better

understand the mixing performance of the membrane dispersion minidevice, several experimental approaches have been introduced. The macromixing performance was characterized with RTD curves. The micromixing performance of the single phase was detected by a parallel competing reaction system and the liquid–liquid phase-mixing process was denoted by a dye-extraction method. Moreover, the mixing performance was further tested by the preparation of BaSO<sub>4</sub> nanoparticles.

## Experimental Equipment and Approaches

### Minireactor

An illustration of the membrane dispersion minireactor used in this work is shown in Figure 1. It can be seen that there are three main sheets in the minireactor: two stainless steel sheets and a membrane sheet. The dispersed solution inlet and dispersed solution chamber are in one sheet. The continuous solution inlet, mixing chamber, and slurry outlet are in the other sheet. The three sheets are assembled with bolts. In this minireactor, the dispersed solution (phase) is pressed through a microfiltration membrane into the mixing chamber to mix with another miscible solution or immiscible phase. The geometric size of the mixing chamber was 12 × 4 × 1 mm. The Ni microfiltration membrane with pore sizes of 0.2 and 0.9 μm, and stainless steel membranes with a pore size of 5 μm were applied, respectively.

### Measurement of RTD curves

Residence time distribution curves can provide necessary information for better understanding of the macromixing performance in chemical reactors.<sup>1,2</sup> In this work, a stimulus–response method was applied to determine the residence time, and the experimental setup<sup>17,18</sup> is shown in Figure 2. A solution of methylic orange was selected as the tracer, and the dilute aqueous solution of methylic orange was injected into the reactor with a six-way valve (Waters automated switching valve). Two online ultraviolet (UV) spectrometers (8823A, Beijing Institute of New Technology Application, China) were set at the inlet and outlet to detect the concentration.

### Parallel competing reaction for single-phase mixing

Micromixing was studied by means of a parallel competing reaction: the Dushman reaction between iodide and iodate coupled with a neutralization reaction.<sup>3,4</sup> The reaction formulas are shown as follows

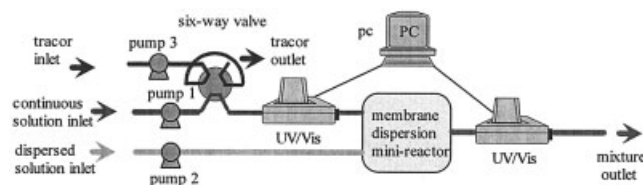
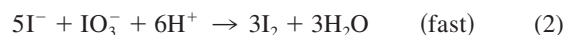
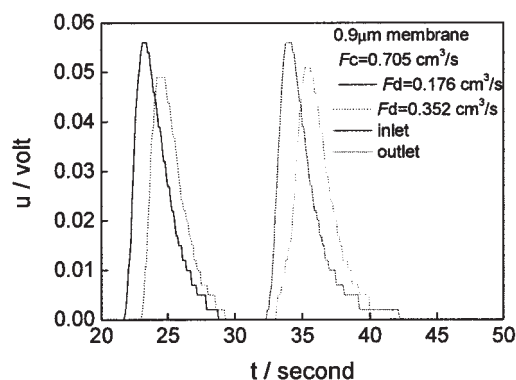


Figure 2. Experimental setup for measurement of RTD curve.



**Figure 3. Residence time distribution curves (0.9  $\mu\text{m}$  membrane).**



Many researchers have studied the kinetics of these reactions.<sup>19</sup> The fractional yield of  $\text{I}_2$  and  $\text{I}_3^-$  were measured by spectrophotometry (UV-vis recording spectrophotometer, UV-8345, Hewlett-Packard) at 353 and 286 nm. A segregation index,  $X_S$ , is defined to explicitly quantify the micromixing quality,<sup>4</sup> which is defined as

$$X_S = \frac{Y}{Y_{ST}} = \left( \frac{n_{\text{I}_2} + n_{\text{I}_3^-}}{n_{\text{H}^+,0}} \right) \left( \frac{6n_{\text{IO}_3^-,0} + n_{\text{H}_2\text{BO}_3^-,0}}{3n_{\text{IO}_3^-,0}} \right) \\ = \frac{(C_{\text{I}_2} + C_{\text{I}_3^-})V}{n_{\text{H}^+,0}} \left( \frac{6C_{\text{IO}_3^-,0} + C_{\text{H}_2\text{BO}_3^-,0}}{3C_{\text{IO}_3^-,0}} \right) \quad (4)$$

In Eq. 4,  $Y$  is the ratio of acid mole number consumed by reaction 2 divided by the total acid mole number injected;  $Y_{ST}$  is the value of  $Y$  in total segregation case when the micromixing process is infinitely slow. So the value of  $X_S$  lies between 0 and 1; for perfect micromixing,  $X_S = 0$ , and in a totally segregated medium,  $X_S = 1$ .

The experiment setup is shown in Figure 1. In the single-phase mixing process, two reactant solutions, diluted sulfuric acid solution and a solution containing the other reactants, were prepared. The acid solution was pumped into the dispersed-phase chamber, and the other solution was pumped directly into the mixing chamber. The flow rates were controlled by two metering pumps. The triiodide complex formed in the minireactor was detected by sampling at the outlet.

### Nanoparticle preparation

The precipitation of barium sulfate has been investigated by experimental method to determine the influence of the mixing performance in the reactors.<sup>20-23</sup> Precipitation experiments with barium sulfate show some influence of micromixing intensity on the particle size and of macromixing on particle morphology.<sup>23</sup>

Two reactant solutions of sodium sulfate and barium chloride were pumped into the minireactor with two metering pumps, and the two miscible solutions were micromixed in the reactor with sodium sulfate solution as the continuous fluid. Because the reaction system was at supersaturation, nanopar-

ticles of barium sulfate were synthesized. The particles were separated by centrifugation. After being washed five times with distilled water and once more with absolute ethyl alcohol, and then dried in an oven at 80°C for 12 h, the barium sulfate nanoparticles were obtained. The morphology of the nanoparticles was characterized by SEM (JEM-6301F, JEOL, Tokyo, Japan) and TEM (JEM200CX, JEOL) images.

### Dye-extraction experiment for liquid-liquid mixing

The micromixing performance of a liquid-liquid mixing process in the new minireactor was measured with a dye-extraction method.<sup>7</sup> The solvatochromatic dye Nile red was selected as the indicator, water/methanol as the aqueous phase, and  $n$ -heptane as the oil phase. Nile red was extracted from the aqueous phase to the oil phase. In the membrane dispersion minireactor, the dispersed phase was dispersed as microdroplets into the continuous phase through the microfiltration membrane, which provided a substantial interface area for mass transport in the two-liquid mixing process. The two phases were pumped into the minireactor from the continuous-phase inlet and dispersed-phase inlet, respectively. The mixed oil-in-water or water-in-oil suspension flowed out from the outlet. After quick phase separation, the Nile red concentration in the organic phase was measured with a spectrophotometer (UV-vis recording spectrophotometer, UV-8345, HP) at 489 nm and the amount of the extracted dye was calculated.

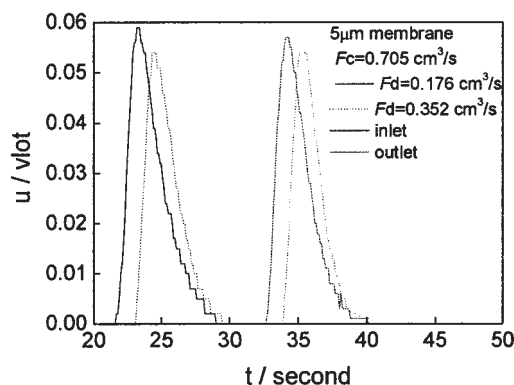
## Results and Discussion

### Macromixing performance

Figures 3 and 4 show the stimulus-response curves obtained under various dispersion-phase fluxes. The response curves at the outlet are very similar to those stimulus curves at the inlet, indicating that the fluid passed through the minireactor in an ideal plug-flow mode. The results mean that the residence time is the function of just two phase flow rates. The axial mixing coefficients are very small, and uneven flow could almost be ignored.

### Micromixing performance of the single-phase mixing process

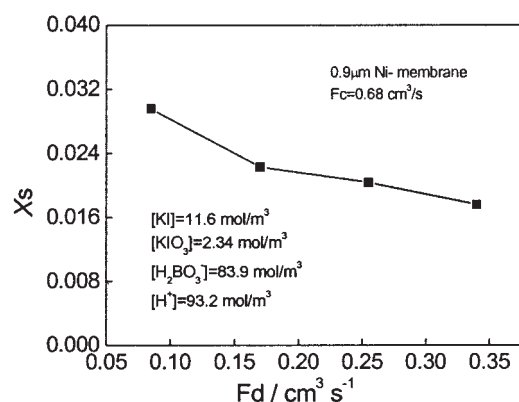
Equation 4 indicates that  $X_S$  is a function of transfer rate of  $\text{H}^+$ .  $X_S$  will increase if the transfer rate of  $\text{H}^+$  decreases, which



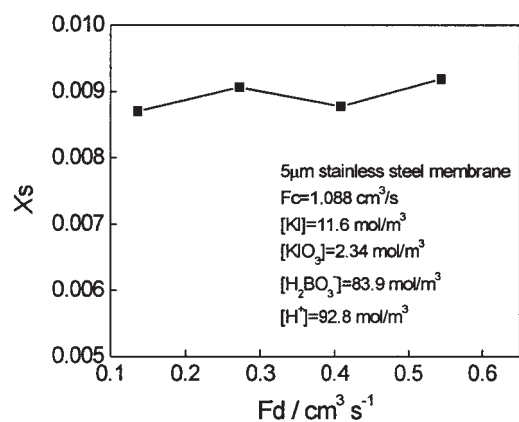
**Figure 4. Residence time distribution curves (5  $\mu\text{m}$  membrane).**

means more segregated medium to be reached. The effect of the dispersed fluid flux on the segregation index  $X_s$  is shown in Figure 5.  $X_s$  decreased with an increase of the dispersed fluid flux. With the increase of the dispersed fluid flux, the turbulence intensity and the mass transfer efficiency of  $H^+$  were improved. Then, the segregation index  $X_s$  decreases, and the mixing performance becomes enhanced. The mixing performance in the minireactor is determined not only by micromixing, but also by meso- or macromixing. When the flux ratio is small, the mixing performance is mainly determined by micromixing, although with increasing flux ratio the role of meso- or macromixing to the reactor's performance becomes increasingly more important. Usually the flux ratio is  $<0.1$  for determining micromixing performance in normal stirring reactors. However, the flux ratio in this work ranged from 0.1 to 10.0, which is always applied in real industry processes. Therefore the obtained data in this work are much more reliable and comparable for designing and scaling-up the equipment.

Figure 6 shows the effect of the continuous fluid flux on the segregation index. As the continuous fluid flux increases, the segregation index decreases, which indicates that the mixing performance becomes enhanced. The segregation index could be  $<0.002$ , which means an almost perfect mixing state. Increasing continuous fluid flux results in a decreasing residence time, which does not confer any benefit to the mixing, but

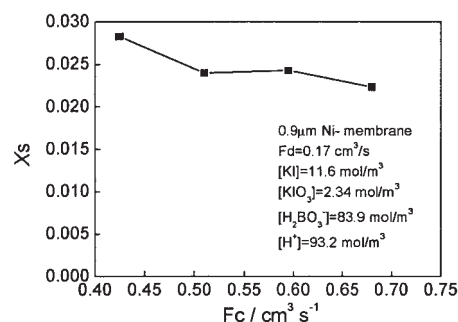


(a)

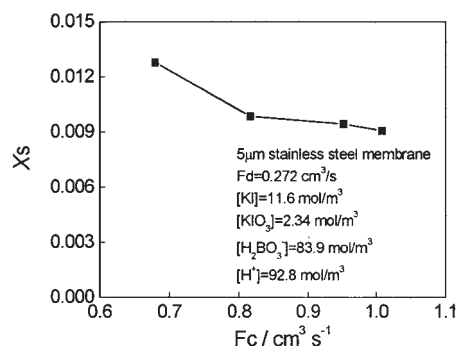


(b)

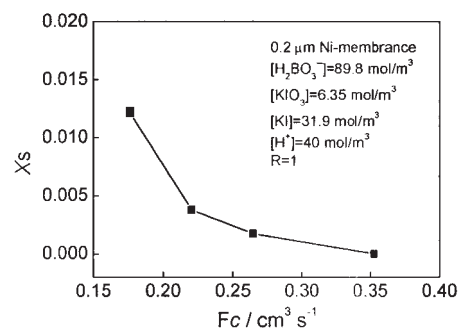
Figure 5. Effect of dispersed fluid flux on segregation index.



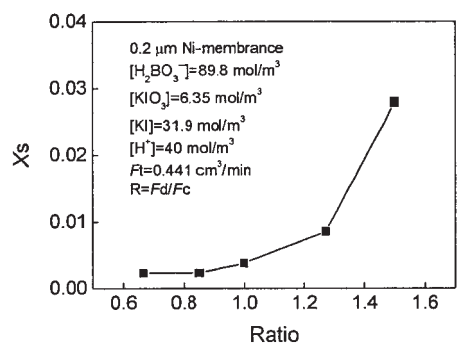
(a)



(b)



(c)



(d)

Figure 6. Effect of continuous fluid flux on segregation index.



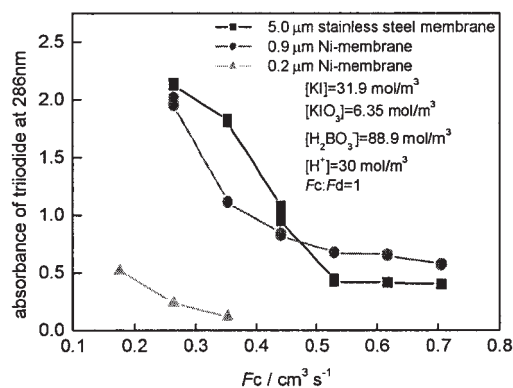


Figure 7. Effect of membrane pore size on micromixing.

rather results in decreasing flux ratio and increasing turbulence intensity, two phenomena that are of substantial benefit to micromixing. Meanwhile an increase in continuous fluid flux could reduce the mixing size, and the mixing process would be predominantly governed by the micromixing characteristic. If the micromixing characteristic is robust, the mixing performance of the minireactor will be closer to the perfect mixing state.

On the other hand, it can be seen that the segregation index is in the range of 0.010–0.032, shown in Figures 5 and 6. Thus, it is possible to conclude that the micromixing performance of the minireactor is much better than that of normal reactors, and the micromixing performance can be improved through increasing the continuous fluid flux and the dispersed fluid flux.

Figure 7 shows the effect of the membrane pore size on the mixing performance, which was substantially influenced by the membrane pore size. When the pore size was 0.9 or 5  $\mu\text{m}$ , the mixing performance under the same experimental conditions was similar. When the pore size was 0.2  $\mu\text{m}$ , the mixing performance was enhanced to an even greater degree. Obviously the membrane pore size will influence the mixing size. With a decrease of the pore size the mixing size decreases, and thus a better mixing state could be attained. The curves in Figure 7 also show that the micromixing performance can be further improved by increasing the continuous fluid flux.

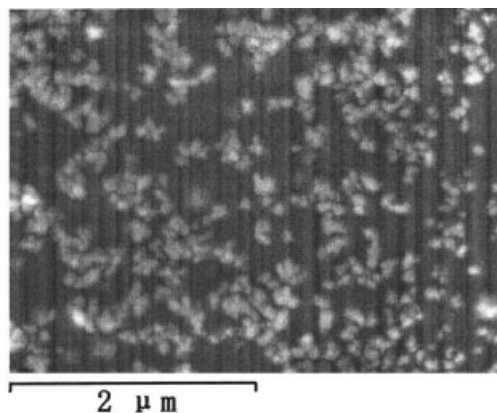


Figure 8. SEM images of  $\text{BaSO}_4$  particles.

$\text{BaCl}_2$ : 100  $\text{mol/m}^3$ , 0.37  $\text{cm}^3/\text{s}$  dispersed fluid;  $\text{Na}_2\text{SO}_4$ : 400  $\text{mol/m}^3$ , 0.40  $\text{cm}^3/\text{s}$  continuous fluid; 0.2  $\mu\text{m}$  membrane.

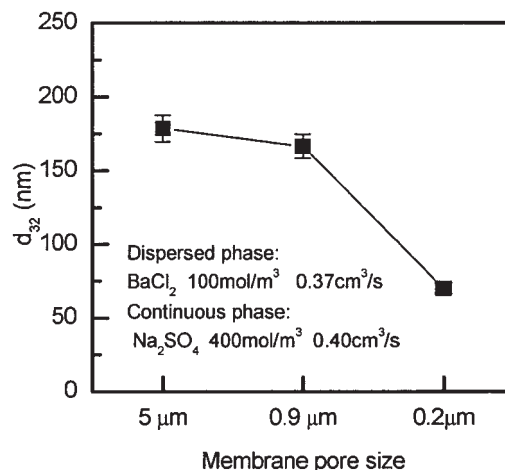


Figure 9. Influence of membrane pore size on average size of  $\text{BaSO}_4$  nanoparticles.

### Precipitation of nanoparticles

Figure 8 is a SEM microphotograph of  $\text{BaSO}_4$  nanoparticles prepared by mixing 100  $\text{mol/m}^3$   $\text{BaCl}_2$  solution with 400  $\text{mol/m}^3$   $\text{Na}_2\text{SO}_4$  solution. When the continuous fluid flux and the dispersed fluid flux were 0.40 and 0.37  $\text{cm}^3/\text{s}$ , respectively, the particles prepared in the membrane dispersion reactor were in the sub-200-nm range, which is much smaller than the size of those (range: 1–10  $\mu\text{m}$ ) precipitated in the normal Taylor–Couette reactor or other classic stirred tank reactors.<sup>20–23</sup> The size of the particles is affected by the micromixing performance.<sup>23</sup> The better performance of micromixing results in smaller particles. Thus compared against other reactors, the new minireactor has a potentially higher micromixing performance.

In our previous work,<sup>12</sup> the relationship between the operation conditions and the average particle size was investigated. The particle size decreases with increasing continuous fluid flux. An increase of the continuous fluid flux results in both increasing supersaturation and improvement of the micromixing, both of which favor precipitation of nanosized particles.

The influence of the membrane pore size on the average

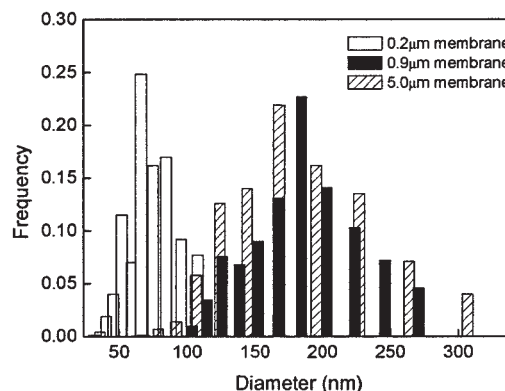


Figure 10. Influence of membrane pore size on size-distribution of  $\text{BaSO}_4$  nanoparticles.

Dispersed phase:  $\text{BaCl}_2$ , 100  $\text{mol/m}^3$ , 0.37  $\text{cm}^3/\text{s}$ ; continuous phase:  $\text{Na}_2\text{SO}_4$ , 400  $\text{mol/m}^3$ , 0.40  $\text{cm}^3/\text{s}$ .

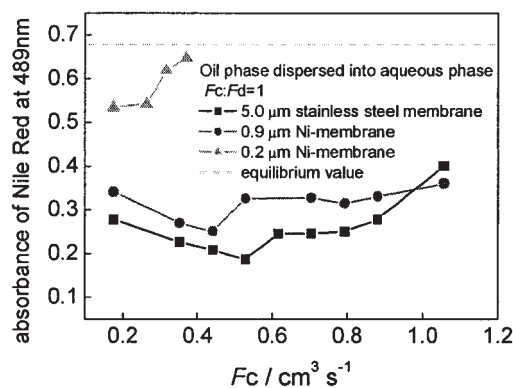


Figure 11. Absorbance at 489 nm with organic phase as the dispersed phase.

sizes and particle size distribution of barium sulfate is shown in Figure 9<sup>12</sup> and Figure 10. It can be seen that the average particle sizes decrease and the particle size distributions become narrower with decreasing membrane pore size. As the membrane pore size decreases, the micromixing performance is vastly improved, resulting in improved morphology and size distribution of the nanoparticles.

#### Micromixing performance of liquid–liquid mixing process

The processes of multiphase mixing are always encountered in unit operation and chemical reaction engineering. To understand the mixing performance of multiphase-mixing processes, a liquid–liquid mixing process in the minireactor was carried out. After phase separation, the absorbance of the organic phase was measured. The results with the oil phase used as the dispersed phase are plotted in Figures 11 and 12. The influences of the membrane pore size are also shown in these figures. When the membrane pore size was 0.2  $\mu\text{m}$ , the mixing efficiency of the two-liquid phase-mixing process was similar to that of the single-phase mixing process, and was increased with increasing flow fluxes. However, when the membrane pore size was 0.9 or 5  $\mu\text{m}$ , unlike the single-phase mixing process, the mixing efficiency was initially decreased with an increase in the continuous-phase flux. After a minimum value was reached, it was increased. The results are similar to the

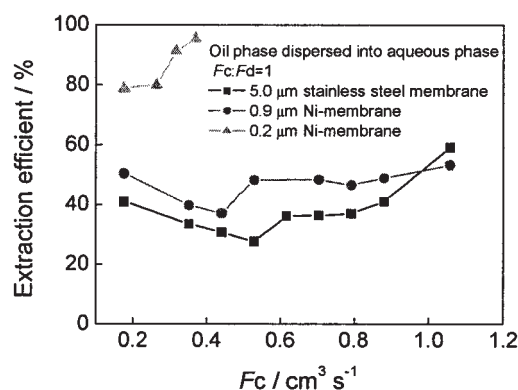


Figure 12. Extraction efficiency with organic phase as the dispersed phase.

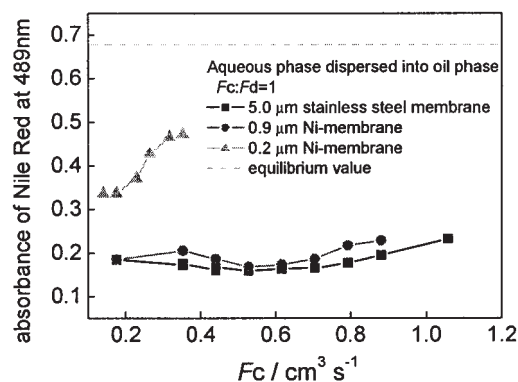


Figure 13. Absorbance at 489 nm with aqueous phase as the dispersed phase.

mass-transfer performances of a membrane dispersion extractor<sup>24</sup> and the results in Panić et al.<sup>7</sup> and Benz et al.<sup>25</sup> In the two-liquid phase-mixing process the mixing efficiency is a function of the mixing condition, mixing size (droplet size), and overall mass-transfer coefficients. When the membrane pore size is smaller, the mixing size decreases and the mass-transfer coefficients increase with the continuous-phase flux. The mixing becomes more uniform, and thus the mixing efficiency is higher. However, when the membrane pore size is larger, the mixing size becomes larger. Therefore, the mixing conditions deteriorate and mixing efficiency decreases. As the continuous-phase flux increases, both the mixing size and the residence time will decrease, although the mass-transfer coefficient will increase. At a lower flux value the mixing condition becomes worse, and the mixing efficiency is predominantly controlled by the residence time. When the flux was  $>0.6 \text{ cm}^3/\text{s}$ , the mixing condition became more uniform and the overall mass-transfer coefficient was higher. Thus, the mixing efficiency was increased with the continuous-phase flux.

Figures 13 and 14 show the experimental results of the two-liquid phase-mixing process when the aqueous phase was used as the dispersed phase. The results in Figures 13 and 14 are similar to those in Figures 11 and 12. The mixing efficiency for using the aqueous phase as the dispersed phase was lower than that for using the organic phase as the dispersed phase. The mixing size (droplet size) is affected not only by the membrane pore size, but also by the system's physical prop-

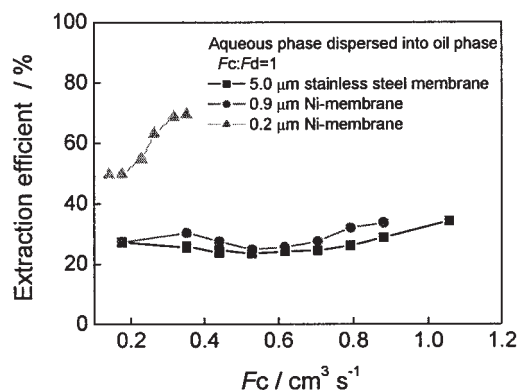


Figure 14. Extraction efficiency with aqueous phase as the dispersed phase.

erties and the wetting ability of the membrane to the two phases. When the dispersed phase was changed from the oil phase to the aqueous phase, the droplet size became larger as a result of the stronger wetting ability of the membranes to the aqueous phase. This is the possible reason for the deterioration of the mixing performance.

## Conclusions

The mixing performances of a novel kind of membrane dispersion minireactor have been investigated with various experimental approaches. The RTD result showed that the flow performance in the minireactor was almost in the plug-flow condition. The single-phase micromixing performance measured with the Dushman reaction (iodide-iodate) in the minireactor attained the desired condition, and the segregation index could be  $<0.002$ . The micromixing performance improved with the increasing of the continuous fluid flux and dispersed fluid flux. Moreover, the micromixing performance was apparently enhanced by decreasing the membrane pore size. The result of precipitation of  $\text{BaSO}_4$  showed that the mixing performance had a substantial influence on the particle size. The membrane pore size is a major contributing factor to micromixing performance. With increasing membrane pore size, the micromixing performance became better, and the  $\text{BaSO}_4$  particle size apparently decreased. It was found that the dye-extraction method can be applied to correctly determine the mixing performance of a two-liquid phase-mixing system. Like the single-phase mixing process, the mixing performance of the two-liquid phase system was influenced by the membrane pore size and the two-phase flow rate. When the pore size was  $>0.9 \mu\text{m}$ , however, there was a minimum value for the mixing efficiency with the changes of the continuous- or dispersed-phase flux.

## Acknowledgments

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## Notation

- $d_{32}$  = mean diameter of particles,  $d_{32} = \sum d^3 / \sum d^2$ , nm  
 $F_c$  = flux of continuous solution (phase),  $\text{cm}^3/\text{s}$   
 $F_d$  = flux of dispersed solution (phase),  $\text{cm}^3/\text{s}$   
 $F_t$  = total flux of dispersed solution (phase) and continuous solution (phase),  $\text{cm}^3/\text{s}$   
 $R$  = flux ratio of dispersed phase and continuous phase  
 $X_S$  = segregation index  
 $u$  = electric voltage information of absorbance of methyl orange, volt

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