

Continuous Purification of Wet-Process Phosphoric Acid via Microchannel

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ABSTRACT: Solvent extraction is an economically efficient method widely used in the purification of wet phosphoric acid. In this study, a microchannel was applied to promote the mixing and purification of phosphoric acid during continuous production. For this aim, solvent extraction was conducted to purify phosphoric acid via methyl isobutyl ketone/tri-butyl phosphate mixtures. Additionally, the Box-Behnken design method was used to survey the solvent extraction process. The effect of various operational parameters such as solvent concentration (45–65 %wt.), temperature (18–28 °C), and organic/aqueous phase ratio (2:1–6:1) at a constant flow rate of 70 mL/L was examined. Experimental results indicated that the microchannel at the residence time of 6.85 min could promote the extraction percentage of and sulfate removal of more than 98% and 60%, respectively, compared to the batch extractor.

KEYWORDS: Solvent extraction; Microchannel; Purification; Wet-process phosphoric acid.

INTRODUCTION

Solvent extraction is a method to separate compounds based on their relative solubility in two different immiscible liquids. In this method, one or more species transfer from feed into the solvent phase. By definition, solvents enriched in solutes are called extract, and those depleted in solutes are called the raffinate [1,2].

Phosphoric acid is an important mineral acid industrially produced by two general routes called

the “thermal and wet process”. In the wet process that is more economical and environmentally friendly, the wet Phosphoric Acid (WPA) is produced by the reaction of rock phosphate with sulfuric acid and by filtration of prepared pulp [3-6].

Owing to the presence of impurities in raw materials, the phosphoric acid obtained from the chemical method contains many inorganic species. Some of these chemical

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elements reduce the quality of its ultimate use in fertilizers or the pharmaceutical and food industries [7-9].

Many researchers have investigated various methods of purification such as crystallization, precipitation, liquid-liquid extraction, and ion exchange [10-13]. Meanwhile, solvent extraction is an efficient, practical, and economical method, which is currently widely used in the industrial process [14-16].

In the selection of the solvent, a wide range of liquids can be used, but the following should be taken into account in their selection: selectivity, distribution coefficient, and solubility. Different studies have focused on appropriate solvents such as esters [17], ketones [18], and alcohols [19] that are efficient, have low toxicity and have high selectivity. Meanwhile, mixtures of solvents are appealing methods to reduce the impurities (metal ions, sulfate ions, chromium, calcium, or anionic and cationic ones) of industrial phosphoric acid were obtained by combining solvent and chemical extraction methods [20, 21].

For example, *Ahmed et al.* considered the use of methyl isobutyl ketone (MIBK) and Tri-Butyl Phosphate (TBP) mixtures to purify the wet-process phosphoric acid. As they observed, the highest purification yield was achieved at 30 °C with a MIBK percentage of 55% [22]. Solvent extraction with tri-n-butyl phosphate and cyclohexanol mixtures via multi-stage counter-current extraction from the industrial WPA in a reciprocating plate extraction column increased yield by over 85% at 40 °C [17].

For several decades, conventional extractors such as continuous mixing reactors, filled columns, and RTL extractors have been used in chemical processing for solvent extraction [23-25]. However, the use of these conventional devices has many disadvantages as they need large space and high energy consumption. Different technologies have been utilized to increase the efficiency of solvent extraction and lower the related processing costs. The microfluidic technique has many unique benefits such as short diffusion distance, simplified process control, high volume/surface ratio, and higher transport (e.g., heat and mass transfer) rates [26-28]. Moreover, low chemical content makes microchannels attractive for reactions involving specific conditions or specific substances [29].

Jafari et al. used a twisted microstructure to evaluate mass transfer between immiscible liquid phases, including water (Cu) + D2EHPA + kerosene [30]. They used three

different twisted micromixers to conduct the experiments and compare results to a plain microchannel. The experimental results indicated that creating more twists in the microchannel was more effective for liquid-liquid mass transfer; however, more energy is required to pump fluids rather than in a plain microchannel.

In another study, *Sahu et al.* studied the extraction of propionic acid from toluene to water in two different glass microchannels with a square cross-section of 150×150 μm [31]. Their experiment was conducted in the slug flow regime, with a Y-junction serpentine micro-channel at the residence time of 3.29s, and 100% extraction efficiency was obtained.

For this purpose, in this work solvent extraction of wet phosphoric acid was experimentally studied in two microchannel and batch modes. The mixtures of methyl isobutyl ketone (MIBK) / Tri-Butyl Phosphate (TBP) were selected to study phosphoric acid extraction. Moreover, the Design of Experiments (DOE) was used to study the extraction condition. Operating variables were temperature, the solvent concentration (weight percentage of methyl isobutyl ketone to tributyl phosphate), and the ratio of the organic phase to the aqueous phase. Moreover, the percentage of extraction and sulfate removal amounts were selected as the criteria for measuring the quality of the extraction. In the end, by examining the percentage of extraction and time of extraction, the quality of extraction in the microchannel was compared with that of other extraction equipment.

EXPERIMENTAL SECTION

Materials

In this research, the Phosphoric acid (54% purity) was prepared by Arya Phosphoric Jonub Company, (Iran). The reagents Methyl Isobutyl Ketone and Tributyl Phosphate were purchased from Sigma-Aldrich. All the chemicals were in analytical grade. Furthermore, distilled water was used in the operational procedure. Detailed specifications of chemicals used in this work were shown in Table 1.

Apparatus and procedures

The micro-extractor setup was prepared with Plexiglas T-shaped micromixer and a stainless steel microtube with an internal diameter of 0.8 mm and a length of 500 cm. The temperature of the micro-extractor was regulated

Table 1: Specifications of the Chemicals.

Chemical name	CAS #	source	Purity (wt %) ^a	GC analysis (wt %) ^b
Methyl Isobutyl Ketone	108-10-1	Sigma-Aldrich	≥ 99.0 %	≥ 99.2 %
Tributyl Phosphate	126-73-8	Sigma-Aldrich	≥ 97 %	≥ 97.6 %

a) Obtained by the supplier. B) Experimentally determined by gas chromatography.

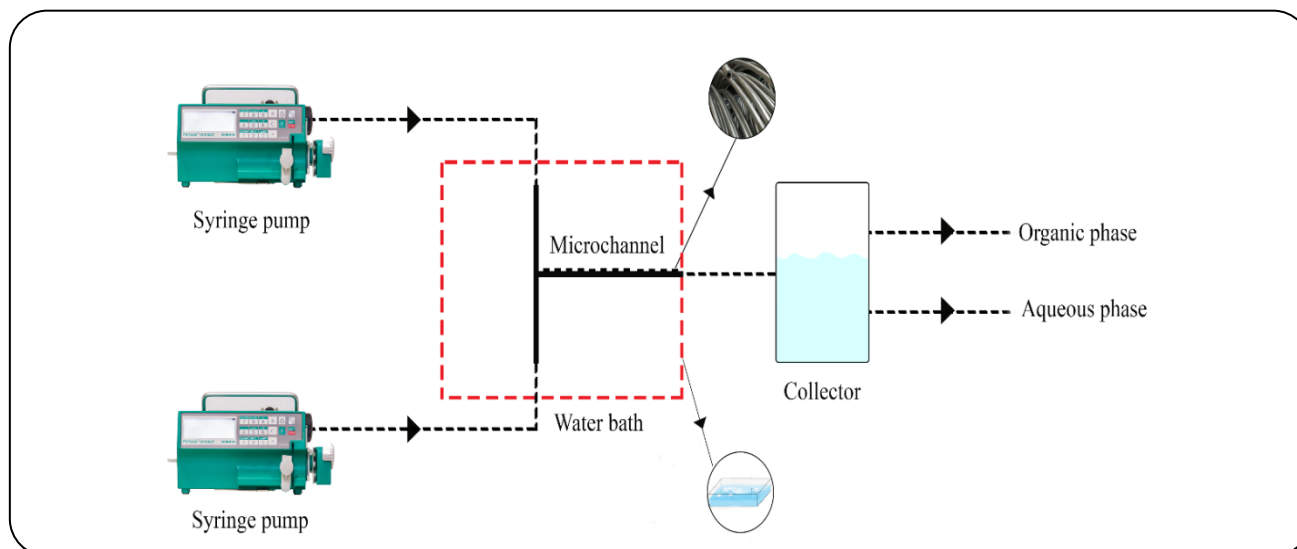


Fig. 1: The micro-extractor setup.

in an incubator (with an uncertainty of ± 0.2 K). Moreover, the experiments were repeated in batch mode. Scheme 1 shows the micro-extractor setup.

In all the experiments, a mixture of water and phosphoric acid with a ratio of 4:6 was selected as feed. As mentioned above, a composition mixture of MIBK+TBP was introduced as an extraction solvent. Two streams of the solvent mixture and acid were fed into a T-mixer by using a syringe pump (SONDOS TEB™ 350 with an uncertainty of $\pm 3\%$) and mixed in the passed through the microtube. Based on the feed rate of 70 mL/h, the residence time for all tests was 2.15 min. The output could settle for 15 minutes to reach equilibrium, and aqueous and organic solvents were differentiated into two phases. The samples were carefully separated, weighed, and collected for analysis using the titration method. The titration was carried out with an acid-base reaction and an 827 pH detector (Metrohm pH Lab with an uncertainty of ± 0.003 .) was used to determine the pH of the solution.

The percentage of extraction was determined with phosphoric acid concentrations in the organic phase toward feed solutions.

$$E\% = \frac{H_3PO_4 \text{ in the organic phase}}{H_3PO_4 \text{ in the initial solution}} \quad (1)$$

The concentration of sulfate ion was determined by measuring light absorbance using a spectrophotometer (DR 5000, Hach, Jenway, USA) at 420 nm with an accuracy of $\pm 0.5\%$.

The percentage of sulfate removal was calculated using the following equation:

$$SO_4^{2-} = \frac{[SO_4^{2-}]_1 - [SO_4^{2-}]_0}{[SO_4^{2-}]_0} \quad (2)$$

$[SO_4^{2-}]_0$ is the sulfate value at the initial feed, and $[SO_4^{2-}]_1$ is the COD sulfate value at the aqueous phase. The sulfate ions were measured based on the standard method [30].

It should be pointed out that the selection of the solvents was considered due to immiscibility with aqueous, toxicity, and flammability. The extraction of phosphoric acid with MIBK can be carried out in a room

Table 2: Input variables and their levels.

Parameters	Value
Temperature (°C)	18, 23, 28
Organic solvent ratio (wt. %)	45, 55, 65
Organic to aqueous phase ratio (O/A)	2, 4, 6

temperature, while extraction with TBP should be carried out at a higher temperature regarding its relatively high viscosity.

In all experiments, all the samplings were measured after 5 min to reach a steady condition. Moreover, each experiment was repeated three times.

Design of experiments

Two classes of response surface designs are the central composite design and the Box-Behnken design [32, 33]. In central composite design a ball where all of the corner points lay on the surface of the ball, but in the Box-Behnken design, the ball is located inside the box defined by a wireframe that is composed of the edges of the box. Box-Behnken design doesn't need as many center points because points on the outside are closer to the middle. So, the Box-Behnken might feel a little safer since the points are not as extreme as all of the factors. Also, the number of observations for the Box-Behnken design is lower than the central composite design. Among all other Response Surface Methodology (RSM) designs, Box-Behnken experimental designs need fewer runs (15 runs for three variables).

The experimental design method was carried out by Minitab software via Box-Behnken design. The selection of methyl isobutyl ketone and tributyl phosphate as solvents was based on previous works [34, 35].

In the following, the mixture of water+phosphoric acid and MIBK+TBP were named and the aqueous phase and organic phase were respectively. The variables were temperature, the solvent concentration (weight percentage of methyl isobutyl ketone to tributyl phosphate), and the ratio of the organic phase to the aqueous phase. Furthermore, significant results were the percentage of extraction and sulfate removal amounts. Table 2 presents input variables as well as their dimensions and levels.

RESULTS AND DISCUSSION

The parameters that affect the performance of the microchannel for solvent extraction are temperature,

solvent concentration, and the ratio of the organic phase to the aqueous phase. All these parameters affect the performance such as the percentage of extraction and sulfate removal amounts which these results are summarized in Table 3.

Effect of temperature

Three levels of 18, 23, and 28°C were considered for the temperature variable. As Fig. 2 shows, the temperature has no significant effect on the extraction percentage; however, increasing temperature decreases the H_3PO_4 distribution coefficient. Furthermore, as Figure 3 shows, sulfate removal can increase and then decrease with temperature increase.

Li et al. reported similar results for the relation between the distribution coefficient and the temperature in the range of 20-60°C [19]. Moreover, the experimental results obtained by *Amin et al.* revealed that for several solvents in the temperature range (5-60 °C), the temperature did not have a significant effect on the extraction percentage [35].

Effect of Solvent Concentration

As Fig. 2 shows, with increasing the percentage of the MIBK from 45 to 55 wt.%, the extraction percentage was reduced. With further increase of MIBK to 65 wt.%, the extraction percentage was gradually decreased. Furthermore, solvent concentration has a different effect on sulfate removal so the sulfate removal increased as the amount of MIBK increased from 45 to 55 wt.%. Moreover, as Fig. 3 depicts, increasing the amount of MIBK to 65 wt.% led to a decrease in sulfate removal.

Effect of the organic to aqueous phase ratio

One of the most important parameters affecting the extraction percentage was the organic to aqueous phase ratio. As Fig. 2 shows, the effect of the organic to aqueous phase ratio on the percentage of extraction, within a residence time of 6 min, was investigated at three levels, including 2:1, 4:1, and 6:1 ratios. The results indicate that

Table 3: Design of experiments using Box-Behnken design and the obtained experimental results.

Run No.	Organic to aqueous phase ratio	Organic solvent ratio	Temperature	E %	SO ²⁺ 4%
1	4	55	23	56.35	34.11
2	4	45	18	43.37	23.18
3	2	65	23	28.55	28.76
4	6	45	23	79.82	43.59
5	6	55	18	74.70	31.10
6	2	55	28	31.22	30.49
7	4	45	28	77.47	37.81
8	2	55	18	33.73	25.86
9	4	55	23	27.39	26.32
10	2	45	23	35.72	44.20
11	4	55	23	62.03	38.51
12	6	65	23	78.26	28.06
13	4	65	18	51.13	28.71
14	4	65	28	42.22	26.67
15	6	55	28	68.87	31.00

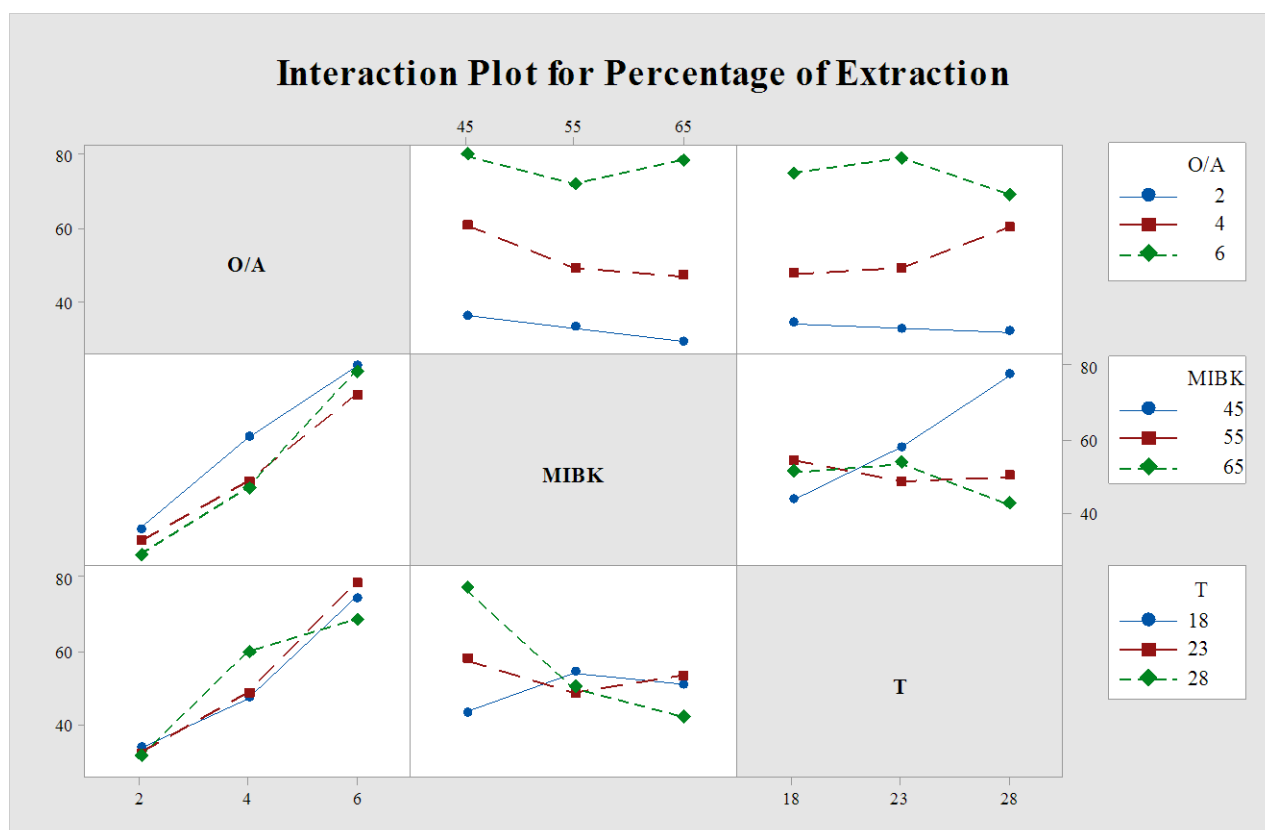
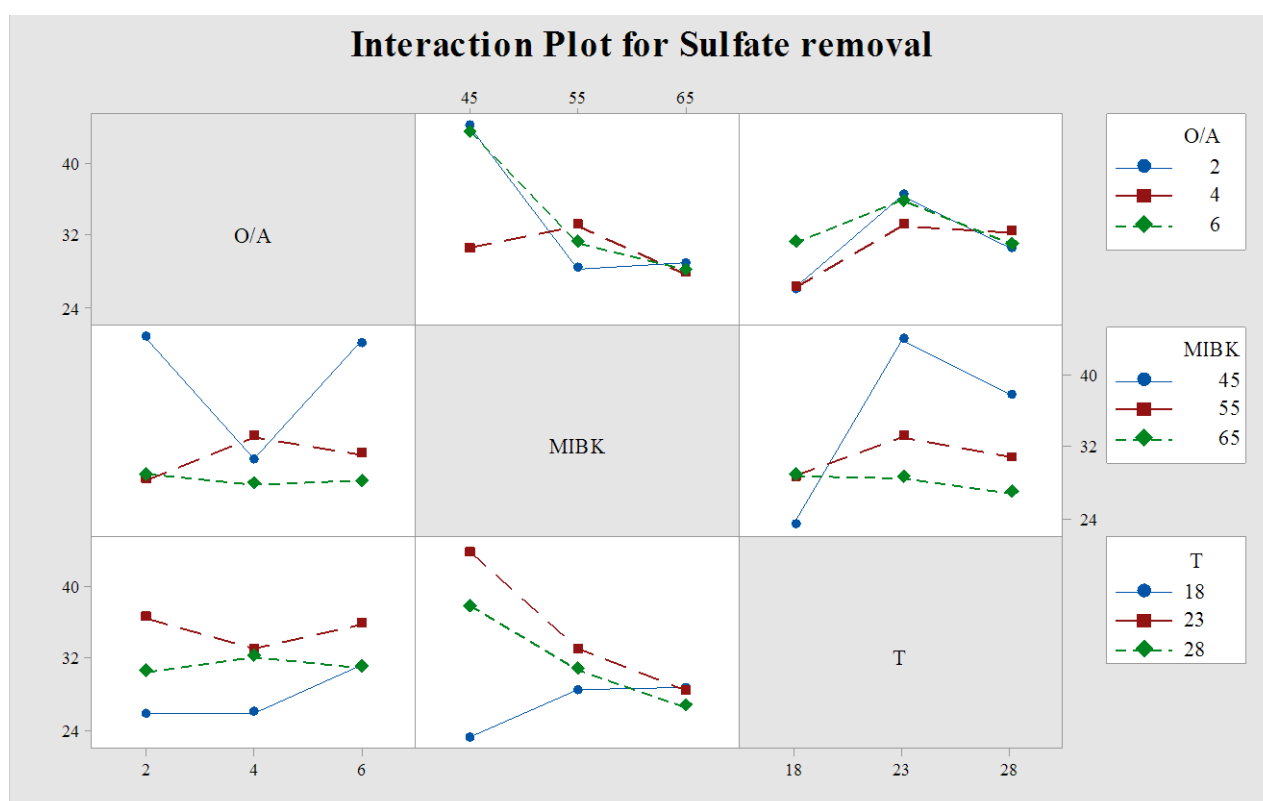


Fig. 2: Interaction plot of the extraction percentage.

Table 4: The effect of various flow rates on the extraction percentage and the sulfate removal.

Run No.	Flow rate mL/h	E %	SO ²⁻ 4 %
1	15	100	43.12
2	22	99.4	32.58
3	30	98.61	38.75
4	40	89.22	18.5
5	50	84.52	36.88
6	70	79.82	43.59
7	90	73.35	21.44

**Fig. 3: Interaction plot of the sulfate removal.**

with increasing the organic to aqueous phase ratio, the extraction percentage significantly increases; this trend is similar to the sulfate removal as indicated in Fig. 3. *Li et al.* concluded the phosphoric acid purification with the organic to aqueous phase ratio of 1 to 5 and obtained the extraction percentage from 51% to 80.2% [19]. In this regard, previous papers report similar effects of the organic to aqueous phase ratio toward the extraction percentage [35].

Effect of flow rate

Table 4 summarizes the effect of various flow rates, showing different residence times on the extraction percentage and the sulfate removal. The experiment was conducted at the temperature of 23°C, with a solvent concentration of 45 wt.% and an organic to aqueous phase ratio of 6. The results indicate that the extraction percentage increases from 73% to 100% when the flow rate decreases from 90 to 15 mL/h. It can be observed that the percentage

Table 5: Comparison of the test in batch mode and microchannel.

Extractor	t	%E	%SO ₄ ²⁻	Operating Condition
Batch	50 min	78.26	32.96	T=23°C, MIBK=45, O/A=6
Microchannel	6.85min	99.4	32.58	T=23°C, MIBK=45, O/A=6

Table 6: Comparison of the obtained extraction percentage in different extractors.

Extractor	Solvent	Time	O/A	E %
Batch (22)	Methyl isobutyl ketone	20 min	3.5	50
	tri-butyl phosphate			
Continuous (36)	Cyclo hexanol + Tributyl phosphate	60 min	4	85.08
Continuous (19)	Cyclo hexanol + kerosene	120 min	5	90.02
Present work	Methyl isobutyl ketone+ tri-butyl phosphat	6.85 min	6	99.4

of extraction significantly increases with the decrease in flow rate from 90 to 30 mL/h. With the continuation of flow rate decreasing to 15 mL/h, the percentage of extraction increases slightly. However, the removal of sulfate ions does not have a similar trend.

Comparing purification in batch reactor and microchannel

In this part of the work, the purification process in a batch reactor and a microchannel has been performed under the best conditions via a flow rate of 22 mL/h. Table 5 summarized the experimental results of both modes in the same operating condition. According to the table, the best percentage of extraction and sulfate removal are obtained for the microchannel which indicates the superiority of the microchannel compared to the batch reactor

Finally, to demonstrate the superiority of this study, several works have been compared. Although various extractors cannot be compared directly, comparing the residence time order and the extraction percentage, we can easily judge them. Table 6 lists the results of this comparison, in terms of extraction percentage, residence time, and operational conditions.

CONCLUSION

In this work, a microchannel was successfully used as a novel extractor to purify phosphoric acid under various operational conditions. Three factors (temperature, the ratio of organic solvents, and the ratio of organic phase to aqueous) were selected to evaluate extraction efficiency

and eliminate sulfate ions. According to the experimental results, the highest extraction efficiency and sulfate ion removal accessible by the model and experiment was 99.4% and 44.20%, respectively. According to the experimental results, the ratio of organic to aqueous phase ratio has a significant and positive effect on the extraction percentage and the sulfate removal. Furthermore, the use of a microchannel undoubtedly contributed to obtaining this significant result in less than 6.85 min reaction time. Based on the results, the use of a microchannel can reduce the extraction time to about one-third compared to conventional extractors by the increase of mixing. Moreover, many of the impurities in phosphoric acid (Mg, Cr, Fe, Ca, ...) can be eliminated by the method presented in this work. The other operating conditions, type, and size of the microchannel are can be investigated in future work.

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