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# Investigation of extraction fraction in confined impinging jet reactors for tri-butyl-phosphate extracting butyric acid process☆

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ABSTRACT

The extraction fraction $E$ and overall volumetric mass transfer coefficient $k_La$ of TBP extracting butyric acid process in confined impinging jet reactors (CIJR) with two jets were investigated. The main variables tested were the concentration of tri-butyl-phosphate (TBP) and butyric acid, the impinging velocity $V$, the impinging velocity ratio of two phases $V_{org}/V_{aq}$, the nozzle inner diameter $d_i$ and the distance $L$ between the jet axes and the top wall of the impinging chamber. The results showed that $E$ and $k_La$ increase with an increase of the impinging velocity $V$, the concentration of TBP $C_{org}$, and the impinging velocity ratio $V_{org}/V_{aq}$. However, $E$ and $k_La$ decrease with an increase of the inner diameter $d_i$ from 1 to 2 mm, the concentration of butyric acid $C_{aq}$ from 0.5% (v/v) to 2% (v/v). The factor $L$ ranging from 3 to 11 mm has a negligible effect on $E$ and $k_La$. A correlation on these variables and $k_La$ was proposed based on the experimental data. These results indicated good mass transfer performance of CIJR in the extraction operation.


## 1. Introduction

Treatment for dilute solution of organic carboxylic acid is an important part of the mass transfer and separation processes. It is very easy for the organic carboxylic acid with Lewis bases to form complexing reactions. Thus, many researchers [1–6] made plenty of studies about the treatment for dilute solution of organic carboxylic acid, such as acetic acid, oxalic acid, citric acid, and sebacic acid, by using the method of complexing. During such a process, the solute that needs to be separated in the solution comes in contact with the complexing agent firstly, and the complexes are formed after the reaction, and then transferred into the extraction agent to complete the separation. Among the previous researches, most of the complexing agents used are phosphorus oxide and amine extractants, including tributyl phosphate (TBP), trialkyl phosphine oxide (TPO) and trioctylamine. For there are alkali Lewis bases in these extractants, it is easier for them to undergo complexing reactions with organic carboxylic acid. Compared with the traditional extraction method, the complexing extraction can achieve higher extent of extraction and bigger reaction rate at low solute concentration. Besides, the complexing method has higher extraction efficiency and selectivity for dilute organic carboxylic acid solution. In addition, the used complexing agents can be refreshed by stripping the combined organic carboxylic acid with alkali agents, such as NaOH, which will make full use of the complexing agents by recycling.

The impinging jet techniques, first proposed by Elperin [7], make two or more high speed jets collide in a narrow zone. At the collision, the axial velocity of the jets will be converted into the fluctuating velocity, and then a collision zone characterized by very severe turbulence is formed. Because of high turbulence, the materials in the jets are brought into close contact. The shear force generated by interphase collision makes the droplets break up, leading to an increase of contacting area and enhanced surface refreshing. At the same time, the shear force also decreases the phase mass transfer resistance. For these advantages, impinging jets can strengthen the phase heat and mass transfer, and reduce the volume of the producing devices. There are many applications and researches [8] about the impinging jets in various fields, such as drying of solid particles, absorption and desorption of gas, combustion of coal and gas, phosphate burning, and preparation of ultrafine particles/dust. Kleingeld *et al.* [9] investigated the gas absorption capacity of the high strength impinging jet reactor using the sodium hydroxide–carbon dioxide system, showing that the efficiency of gas absorption was better than the traditional method. Huai *et al.* [10] studied the effects of the inlet air temperature, mass flow-rate ratio, initial moisture content and air velocity of the impinging stream on the drying process. Wu *et al.* [11] prepared ultrafine barium sulfate powder with an

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impinging jet reactor, and the powder showed an ellipsoidal shape with an average diameter of 100 nm.

In recent years, although impinging jets have been applied to the wastewater treatment and dilute acid extraction by some researchers, the number of related researches is limited. Tamir [12] designed a liquid–liquid impinging jet extraction device first and found that its mass transfer coefficient was much greater than the traditional devices. Dehkordi [13,14] designed a new impinging jet extraction device based on the former researches, and tested it using a weak extractant with moderately slow chemical kinetics to extract cooper ions. The experimental results indicated that the extraction and stripping rates of copper in impinging jet reactors are 7 to 8 times bigger than the traditional CSTR per unit volume of reactors. Saien *et al.* [15–18] made a detailed research about the influences of jet momentum, nozzle diameter and the inter-nozzle distance for the impinging jets on the extraction fraction $E$ by the standard test system of toluene–acetone–water recommended by the European Federation of Chemical Engineering (EFCE). The results showed that the overall volumetric mass transfer coefficient $k_La$ was 3–5 times bigger than that in the study made by Dehkordi [13]. The values of $k_La$ and $E$ were very sensitive to pH, and decreased with an increase of pH in the range of 5.5 to 8. Low amounts (maximum $10^{-4}$ mol·L$^{-1}$) of NaCl in aqueous phase had a significant improvement for the toluene–acetone–water system, $k_La$ and $E$ increase about 35.6% and 22.7%, respectively. The extraction fraction $E$ of the low interfacial tension chemical system is higher than that of the high interfacial tension chemical system under similar conditions. Compared with conventional contactors, $k_La$ of the two impinging-jet contacting device (TIJCD) had a much better performance. Qi *et al.* [19] investigated the extraction fraction of the submerged circulative impinging stream reactor for acetic acid extraction, and the result proved the excellent performance of the impinging jets. Wang *et al.* [20] tested the extraction of phenol wastewater by TBP extractant in the submerged circulative impinging stream reactor (SCISR), and achieved a 20% promotion of the extraction fraction than the traditional reactor. Zhang *et al.* [21] tested the extraction effect for $Cr^{6+}$ using the kerosene solution of TBP in the impinging jet reactor and found a 20% increase in $E$ compared with the traditional method.

Butyric acid ($CH_3CH_2CH_2COOH$) which is easy to be dissolved in water is an important organic solvent, and it has been used to produce butyrate, medicine, spices and flavorings. Butyric acid is easy to be recognized even at a very low concentration because it has an unpleasant smell like the spoilage cream. The aim of this study is to investigate experimentally the extraction fraction $E$ and the overall volumetric mass transfer coefficient $k_La$ for the extraction of butyric acid using TBP and kerosene as the organic phase. The effects of operating conditions such as the concentration of TBP and butyric acid, the impinging velocity and the impinging velocity ratio of two phases on extraction fraction were studied. And a correlation about these variables and the overall volumetric mass transfer coefficient $k_La$ was proposed.

## 2. Experimental Section

### 2.1. Experimental setup

The experimental flowchart is shown in Figs. 1 and 2. The impinging chamber is actually a 70 mm high hollow cylinder of id 7 mm.

At first, the butyric acid solution of 1% (v/v) and TBP kerosene solution of 10% (v/v) were prepared in two separate Plexiglas tanks. Then, the solution would be pumped into the impinging chamber, and the velocity of the impinging jet was adjusted through the ball valves. After two streams collide in the impinging zone with the expected impinging velocity, the solution mixture exits from the lower outlet. During the testing, samples were taken when the flow was stabilized and sealed by the plastic wrap to allow the solution to stratify and clarify. The upper layer was the organic extraction phase, and the lower layer was the raffinate.

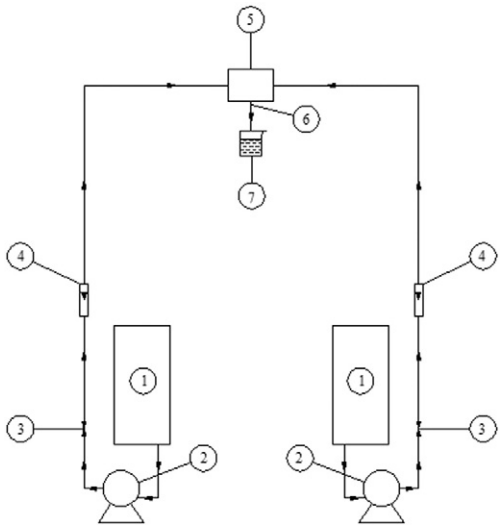


**Fig. 1.** Experimental flowchart. 1. tank; 2. solution pumps; 3. valve; 4. flowmeters; 5. impinging zone; 6. solution export and 7. sample cup.

### 2.2. Chemicals

The chemicals used in this study were deionized water, aviation kerosene, tri-butyl-phosphate, butyric acid, NaOH standard solution (0.01 mol·L$^{-1}$), and HCl standard solution (0.01 mol·L$^{-1}$). All reagents are from local markets and of AR grade.

### 2.3. Physico-chemical properties

According to the density of the pure reagents, the density of the solutions used in the experiment were calculated and shown in Table 1.

A number of experiments were conducted to determine the distribution coefficient of butyric acid. Six initial concentrations of butyric acid solution were 0.5%, 1%, 2%, 3%, 5%, and 10% (v/v), and the initial concentration of TBP was 10% (v/v). The equilibrium concentration of butyric acid in the aqueous phase was analyzed by acid–base titration, while that in the organic phase was calculated by mass-balance. The distribution relationship between the aqueous phase and organic phase of butyric acid is plotted in Fig. 3, and the slope of the line is the distribution coefficient.

When the concentration of the butyric acid $C_{aq}$ is less than 1% (v/v), the fitted formula is

$$y = 2.5978x, \quad r^2 = 0.997. \tag{1}$$

The distribution coefficient is 2.5978 in the interval.

On the other hand, the slope of the line is relatively small when $C_{aq}$ is more than 1% (v/v), and the formula is

$$y = 1.3279x + 0.0303, \quad r^2 = 0.997. \tag{2}$$

The distribution coefficient is 1.3279 in the interval.

### 2.4. Measurements

Five milliliters of raffinate was sampled with a pipette when samples were stratified. The concentration of butyric acid in the samples was analyzed by acid–base titration. Then, the extraction fraction $E$ and overall