



Intensification of high-phase-ratio extraction via microbubble-agitation in gas-liquid-liquid systems



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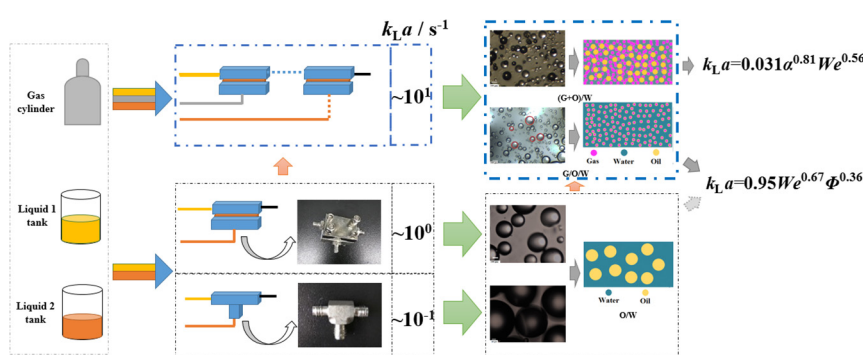
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HIGHLIGHTS

- Gas-liquid-liquid systems with microbubble-agitation were developed.
- Double-membrane-dispersion microextractor was designed.
- Mass transfer characteristics of gas-liquid-liquid systems were analyzed.
- The most efficient mode of microbubble-agitation was recommended.
- Two dimensionless equations were established to predict the intensification effect.

GRAPHICAL ABSTRACT



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ABSTRACT

Microbubble-agitated extraction was conducted with microextractors for process intensification of extraction with high phase ratio. Hexanoic-acid/water/*n*-octanol system with phase ratio of 80 was selected as a model system. Single and double membrane dispersion modules were developed for generating liquid-liquid and gas-liquid-liquid microdispersion systems. 10 different methods, 6 of which containing microbubbles, were designed to realize high-phase-ratio extraction. The effect of dispersion size, structure of gas-liquid-liquid emulsion and amount of microbubbles were systematically investigated. The most efficient and stable mode was recommended, with which Murphree efficiency could reach 90% in 0.5 s and the overall volumetric mass transfer coefficient ranges in 7.88–41.34 s⁻¹, about 40 times greater than liquid-liquid system. The mechanism of the intensification effect by introducing microbubbles was discussed. Two typical gas-liquid-liquid structures were selected to study the effects of microbubbles in adjusting phase ratio and promoting turbulence in continuous phase. Two dimensionless equations were established to correlate mass transfer coefficients respectively, both of which showed good coincidence with experimental data.

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1. Introduction

Liquid-liquid extraction, which is also known as solvent extraction, is a method for separating compounds based on the difference

in their solubility between two immiscible liquids (usually water and organic solvents) (King, 1987). Liquid-liquid solvent extraction plays an important role in chemical industrials for separation as well as enrichment of various target components from complicated solutions (Xie et al., 2014; Wilson et al., 2014). Nowadays, liquid-liquid extraction with high phase ratios is becoming increasingly commonly applied in industry, for example,

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separation of zinc and iron (with aqueous-to-organic phase ratio of about 5) (Sinha et al., 2014), purification of phosphoric acid and sulphuric acid (with phase ratio ranging in 0.2–5) (Agrawal et al., 2008) and removal of heavy metal ions (with aqueous-to-organic phase ratio ranging in 5–50) (Ren et al., 2007). The demands in practical separation processes stimulate to develop new methods and equipment for performing effective liquid–liquid extraction processes with high phase ratio. However, effective intensification for extraction processes with high phase ratio is still difficult in overcoming the disadvantages in low specific surface area and hold-up of the dispersed phase.

The extractors, such as mixer-setters, extraction columns and centrifugal extractors, have been designed primarily according to liquid-liquid systems with low phase ratios (Wang and Chen, 2001). During extraction processes, when the phase ratio is higher, more energy is demanded to be injected into system to improve the mass transfer performance, taking increased stirring intensity in mixing chamber and higher flow rates in extraction column as examples. However, the intensification is still not effective when the phase ratio further increases and accordingly the equipment size significantly increases. For example, the equipment size of mixer-setters and centrifugal extractors increases sharply with the increase in phase ratio, due to the decrease in mass-transfer characteristics and difficulty in phase separation caused by the emulsification of the system (Hosseinzadeh et al., 2014; Zhou et al., 2007). The height of extraction columns increases with the increase in phase ratio, due to bad mass transfer performance and low phase separation rate caused by the severe axial back-mixing or flooding for the high phase ratios (Tang et al., 2004).

New techniques have been developed for the intensification of the extraction process with high phase ratio, such as emulsification liquid membrane (ELM) extraction (Ferreira et al., 2014; Valenzuela et al., 2005) and predispersed solvent extraction (Luo et al., 2013; Michelsen et al., 1986) methods, whose advantages have been demonstrated in providing larger specific surface area and more efficient mass-transfer processes due to the preparation of emulsified systems. However, the difficulty in destroying the emulsified systems after mass transfer, due to the introduction of surfactants, cannot be ignored, which leads to significant increase in the size of phase separation equipment. Therefore, new techniques for realizing uniform emulsification without surfactant are effective for process intensification of extraction process with high phase ratio.

Since the last decades, microstructured devices have been successfully applied for enhancing heat and mass transfer efficiency and generating uniform reaction environments. (Bothe et al., 2006; Kashid et al., 2011; Maruyama et al., 2004; Sahoo et al., 2007; Tian et al., 2010). In these microstructured devices, two phases contact in microchannels, which leads to high special surface area (in the order of 10,000–50,000 m²/m³ (Jahnisch et al., 2004)), steerable contacting (Dessimoz et al., 2008), short diffusion distance (Ciceri et al., 2014; Yin et al., 2015; Zhang et al., 2014), process enhancement and safe production (Rahimi et al., 2014). As an important kind of microstructured devices, microextractors have recently received more and more attention and show intensification in mass transfer of liquid-liquid systems. Hou et al. (2013) developed a membrane dispersion microextractor to realize La(III) extraction process and found the extraction efficiency is higher when the organic phase is the dispersed phase, when recovery efficiency is above 82%. Kolar et al. (2016) performed solvent extraction of rare earth elements by using a Y-Y microchip, by which the extraction rates are 2–3 times higher than conventional (bulk) extraction processes.

On the other hand, gas-agitated extraction process has also caught some researchers' attention. Su et al. (2009) used an inert gas to agitate extraction process in microextractors, the overall

volumetric mass transfer coefficient of which ranges in 3.8–30.6 s⁻¹, 2 times higher than the system without addition of microbubbles. Huang et al. (2015) proposed a novel bubbling extraction column to intensify solvent extraction with high aqueous-to-organic phase ratio. In the previous study, microextractors containing double membrane modules was developed and demonstrated the process intensification in H₂O₂ extraction from organic to aqueous phase (Tan et al., 2011).

In this paper, gas-agitated extraction was conducted with microextractors for process intensification of extraction with high phase ratio. Hexanoic-acid/water/*n*-octanol system was selected as a model system to evaluate the mass transfer characteristics of different methods. Three different types of extractors as well as 10 different methods (6 of them contain microbubbles and another 4 not) were designed to realize the extraction of the model system and the effect of dispersion size and addition of microbubbles were studied accordingly. The influence of parameters, such as dispersion size, gas-agitation methods and flow rates, were systematically investigated. The mechanism of intensification effect was discussed and mathematical models were established to correlate mass transfer coefficients in different methods.

2. Material and methods

2.1. Apparatus

Sketches of the experimental set-ups for liquid-liquid and gas-liquid-liquid systems are shown in Fig. 1(a) and (b). The two or three fluids flow from the tanks or gas cylinder were dispersed into liquid-liquid or gas-liquid-liquid systems (the dispersion methods are discussed in Sections 3.2 and 3.3). The dispersed liquid-liquid or gas-liquid-liquid system flowed along a channel before flowing into a phase separator and was separated into organic and aqueous phase.

Three different dispersion modules (Fig. 1(c)) were applied:

- (1) Module A was a 2 mm ID Tee connection.
- (2) Module B was a single membrane dispersion module made of stainless steel, whose dispersion medium was a stainless steel microfiltration membrane with 2 μm average pore size and 0.3 mm thickness (Fig. 1(d)). The size of the flow channel was 15 mm (L) × 2 mm (W) × 3 mm (H). The size of the buffer reservoir for dispersed phase was 10 mm (L) × 5 mm (W) × 10 mm (H).
- (3) Module C was a double membrane dispersion module made of two membrane dispersion modules (the same as Module B) in series.

The aqueous and organic solutions were fed into the dispersion module by high precision advection pumps (Beijing Satellite Manufacturing Factory) with measurement ranges of 0–1000 mL/min (aqueous) and 0–10 mL/min (organic) and measurement accuracy of ±1%. A mass flow controller (Beijing Sevenstar Electronics Company), with measurement range of 0–500 SCCM and measurement accuracy of ±1%, was used to ensure a constant gas flux.

In order to maintain the dispersion state and realize adjustment of residence time, stainless steel capillary pipes with 2 mm ID and different lengths were connected to the outlet of the dispersion module.

A phase separator, 20 mm ID × 100 mm height, was used following the capillary pipe to make mass transfer slow down for sampling.

2.2. Materials

Hexanoic-acid/water/*n*-octanol (physical properties listed in Table 1), was selected as the model system for testing the mass

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