



Extraction of dye from aqueous solution in rotating packed bed



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HIGHLIGHTS

- Liquid–liquid extraction in rotating packed bed for removal of methyl red.
- Determination of mass transfer coefficient that is not yet available in literature.
- Maximum efficiency and volumetric mass transfer coefficient was ~ 0.98 and 0.2 1/s.
- Volumetric mass transfer coefficient in RPB much higher than conventional ones.

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ABSTRACT

The influence of centrifugal acceleration on mass transfer rates in liquid–liquid extraction was investigated experimentally in rotating packed bed (RPB) contactor. The extraction of methyl red using xylene was studied in the equipment. The effect of rotational speed (300–900 rpm), flow rate of the aqueous ($4.17\text{--}20.8 \times 10^{-6} \text{ m}^3/\text{s}$), and organic phase ($0.83\text{--}2.5 \times 10^{-6} \text{ m}^3/\text{s}$) on the mass transfer performance was examined. The maximum stage efficiency attained was ~ 0.98 at aqueous to organic flow rate ratio of 10. The results suggest that contactor volume required to carry out a given separation can be reduced by an order of magnitude with RPB in comparison to conventional extractors.

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1. Introduction

Dyes are widely used in various industries such as textile, leather tanning, paper, plastics, food, cosmetic, printing etc. for the coloration of their related products [1]. Most of these dyes and their metabolites are toxic and potentially carcinogenic in nature [2]. Further, the presence of these colored compounds in wastewater is aesthetically displeasing. They also affect photosynthesis by inhibiting penetration of sunlight into water bodies [3]. Liquid–liquid extraction has been extensively investigated for removal and recovery of textile dyes from wastewater [4–9]. This technique describes a method of separating the solute present in a solution by adding another immiscible liquid in which the solute is transferred preferentially.

Liquid–liquid extraction has traditionally been accomplished in contactors such as mixer settlers [10–12], spray [13–15], packed [15–17] and mechanically agitated columns [18–20]. Considerable effort has been directed to improve upon the design of these

conventional contactors for enhancing the mass transfer efficiency. Hollow fiber contactor wherein the phases were contacted across micro-porous hollow fibers was studied for extraction of succinic acid [21], pesticide [22], ibuprofen [23] among others. Several contactors have been devised to replace terrestrial gravity governing the flow hydrodynamics in these conventional contactors by centrifugal acceleration. The advantages of operating under centrifugal force field include handling systems with low density difference, rapid attainment of steady state due to low holdup and higher throughput [24]. In impinging stream contactor [25], the phases flow outward between a rotating and a stationary circular disk. Annular centrifugal extractors were developed based on the principle of Taylor–Couette flow. Mixing of the streams occur between two coaxial rotating cylinders [26–28]. In rotating spray column [29], the drops were dispersed under centrifugal acceleration into the continuous phase.

The range of the volumetric mass transfer coefficient in the above stated contactors is listed in Table 1. Increase of the magnitude of the volumetric mass transfer coefficient would decrease the contactor volume required for a given separation, and consequently also the capital and operating cost. In recent years, rotating packed bed (RPB) has emerged as a promising alternative for intensifying

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Notations

A	cross section area of contactor (m ²)
C	concentration (mol/m ³)
E _{oc}	stage efficiency
h	axial height of packed bed (m)
K _{La}	overall volumetric mass transfer coefficient (1/s)
(K _{La}) _m	overall volumetric mass transfer coefficient for perfect mixing condition (1/s)
m	distribution ratio
Q	flow rate (m ³ /s)
r	radial distance (m)
V	velocity (m/s)
∇	volume (m ³)

Subscripts

o	organic phase
a	aqueous phase

Superscripts

i	inlet
e	exit

Greek symbols

ω	rotational speed (rpm)
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mass transfer in a wide variety of applications. In this equipment, the phases are contacted between two co-axially rotating cylindrical disks filled with packing materials. The liquid is accelerated under the action of centrifugal force and splits into fine droplets, threads, and thin films in the rotating packing. This results in significant intensification of micro-mixing and mass transfer. Though RPB has been extensively investigated for gas–liquid systems [30–35], only a few studies have reported on mass transfer between two liquid phases. These include polymerization [36], bromination of butyl rubber [37] and biodiesel production [38]. The results of these studies indicated significant improvement in performance.

The above study suggests the possibility of reducing the required contactor volume for carrying out mass transfer between the solvent and feed stream. Previous studies [4–9] on decolorization of dye solution by liquid–liquid extraction have mainly focused on determining the optimum experimental conditions required for maximizing extraction, and were carried out in batch mode of operation. The advantages of RPB suggest the possibility for reducing the extractor volume for continuous decolorization of dye solution in this contactor. However, there appears to be no data available in the literature to evaluate the performance of this contactor for liquid–liquid extraction process. In this study, solvent extraction of methyl red from textile wastewater using xylene was investigated in RPB. Muthuraman and Teng [9] had performed batch studies for this system. The results indicated that this solvent is effective for

extraction of methyl red from its aqueous solution. In this work, the influence of process parameters such as rotor speed, aqueous phase and organic phase flow rate on stage efficiency and overall volumetric mass transfer coefficient has been presented.

2. Experimental**2.1. Materials**

Methyl red, xylene and hydrochloric acid were obtained from Merck. Aqueous solution of methyl red was prepared by dissolving the solute in double distilled water. The pH of the solution was adjusted to the desired value using hydrochloric acid (0.1 N) with the help of pH meter (EUTECH pH Tutor).

A schematic diagram of the experimental setup used for continuous mass transfer experiments is shown in Fig. 1. The rotor used was a pair of stainless steel circular disks of 160 mm diameter fastened coaxially. The axial distance between the two disks was 20 mm. The space between the disks was packed with a stack of 10 stainless steel wire mesh (10 mesh size). The two disks were connected through a shaft to an AC motor and rotated around the horizontal axis. The rotating packed bed was housed inside a stationary cylindrical casing of diameter 180 mm and axial length 50 mm.

Dye solution and xylene were stored in separate reservoirs. The two phases were mixed online prior to entering the RPB and sprayed onto the inner periphery of the packing from a stationary distributor. The diameter of the distributor was 34 mm and consisted of 24 openings of 1 mm diameter. The dispersion flowed towards the outer periphery of the packing under the action of the centrifugal force. It exited the equipment through an opening in the bottom of the casing wall. The concentration of dye in the raffinate phase after phase separation was analyzed at 523 nm wavelength using UV–vis spectrophotometer (PerkinElmer Lambda 25). The reported data was based on the average of three experimental runs. The maximum deviation of the percentage removal was less than 2.5% (standard deviation = ~1.6).

2.2. Mathematical modeling

The mass balance of the solute over a differential volume in the rotor assuming plug flow of the phases is given by

$$Q_a dC_a = -K_{La}(C_a - C_a^*) dV \quad (1)$$

where the differential volume dV is

$$dV = 2\pi r dr h \quad (2)$$

The term Q_a is the aqueous phase flow rate, C_a and C_a^* is the bulk concentration of dye in the aqueous phase, and that in equilibrium with the organic phase respectively at a radius r from the rotational

Table 1
Comparison of overall volumetric mass transfer coefficient with other contactors.

Ref.	Extractor	Chemical system	$Q_o(\text{m}^3/\text{s}) \times 10^6$; $Q_a(\text{m}^3/\text{s}) \times 10^6$	$K_{La}(1/\text{s})$
[12]	Mixer–settler	Water–Acetone–Toluene	0.88; 0.78	0.0015–0.005
[15]	Spray column	Water–Acetone–Toluene	16–130; 20–130	0.0005–0.008
[15]	Packed bed column	Water–Acetone–Toluene	16–130; 8–65	0.0005–0.0055
[20]	Kühni column	Water–Acetone–Toluene	3.9–8.9; 3.9–8.9	0.005–0.0125
[23]	Hollow fiber	Water–Ibuprofen–Octanol	2.7–7.1; 3.6–7.9	0.0045–0.042
[25]	Impinging jet	Water–Succinic acid–Butanol	1.83–5.0; 1.83–5.0	0.077–0.25
[28]	Annular centrifuge	Water–Benzyl alcohol–White mineral oil	0.33; 0.33	0.002–0.0127
[29]	Rotating spray column	Water–Cr (VI)–Aliquat 336 diluted in kerosene	2.3–3.9; 2.5–6.8	0.06–0.12
Present study	Rotating packed bed	Water–Methyl red–Xylene	0.83–2.1; 4.16–20.83	0.015–0.205 (Eq. (10))

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