



Improving performance of liquid–liquid extraction with temperature for mass transfer resistance in both phases



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ABSTRACT

The influence of temperature on hydrodynamic and mass transfer of liquid–liquid extraction process was investigated with single drop experiments in a jacketed pilot column and with respect to variations in physical properties. The applied temperatures were within the conventional range of 15–40 °C. In order to include the influence of mass transfer direction, the chemical system of 4-methylpentan-2-one–acetic acid–water was chosen which exhibits mass transfer resistance against solute transfer in its both phases. Results revealed the significant impact of temperature on the rate of mass transfer with an average enhancement of 46.1% and 36.1% for dispersed to continuous phase and vice versa directions, respectively. The extraction efficiency is the most effective term in this regard, due to enhancement of solute diffusivity in phases. The data were nicely reproduced using a correlation of dimensionless numbers to obtain the effective diffusivity for substitution instead of molecular diffusivity in Newman's equation. The model predicts the overall mass transfer coefficient at different temperatures.

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1. Introduction

Mass transfer to and from drops is the subject concerned in different unit operations especially in liquid–liquid extraction. This process has typical applications in separation of different liquid structure materials, metal extraction, waste water treatment and in petroleum industries. However, expensive and time-consuming experimental investigations constrain researchers with pilot plants and laboratory-scale apparatuses. In this regard and owing to real operations where dispersion of one phase into another one is needed; study on the behavior of single drops is essential. It is since the behavior of single drops determines the performance of extraction columns with swarm of drops, regardless of drop breakage and coalescence phenomena which have to be considered in liquid–liquid extraction operations.

Heretofore, the influence of some parameters on mass transfer coefficient, such as the presence of contaminants [1], aqueous phase pH [2], the presence of salts [3,4] and nanoparticles [5] has been studied. However, effect of temperature for this routine process has not been much investigated, perhaps due to preferred conventionally operating at ambient temperature. Indeed, direct heat application followed by energy

recovery is beneficial in view of process intensification with no extra reagent addition or using special devices. Temperature plays an important role to alter process performance, as long as components of degradation/reaction and/or significant evaporation are not relevant. Recently, effect of temperature on the batch liquid–liquid extraction of phenol from aqueous solutions has been investigated by Palma et al. [6]. Also, in our recent works, the effect of temperature was investigated for the chemical systems of toluene–acetic acid–water [7] and cumene–isobutyric acid–water [8]. Both of these chemical systems exhibit mass transfer resistance in just one phase. Accordingly, an extended investigation in this context was necessary based on a system with the feasibility of mass transfer resistance in both phases. The equilibrium solute distribution and mutual solubility of phases which are usually altered by temperature have therefore to be considered.

In this work, the recommended chemical system of 4-methylpentan-2-one [methyl isobutyl ketone (MIBK)]–acetic acid–water which has been previously used in a number of investigations [9–13] was used. The more important properties of this system are the medium interfacial tension and the existence of mass transfer resistance in both phases. Corresponding equilibrium data at different temperatures have been recently reported [14]. The hydrodynamic and mass transfer of single drops were studied at different temperatures. Both mass transfer directions of dispersed to continuous phase ($d \rightarrow c$) and vice versa ($c \rightarrow d$) were examined. The results of this study help to introduce

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Nomenclature and units

a	the correlation parameter (–)
C	solute concentration (wt%)
c	viscometer constant (–)
d	drop diameter (mm)
D	diffusivity ($\text{m}^2 \text{s}^{-1}$)
E	extraction fraction (–)
$E\ddot{o}$	Eötvös dimensionless number ($g\Delta\rho d^2/\gamma$) (–)
H	dimensionless group defined by Grace et al. (–)
k	viscometer constant, local mass transfer coefficient ($\mu\text{m s}^{-1}$)
L	dimensionless group defined for Newman's equation ($k_c d/2mD_d$) (–)
K_{od}	overall mass transfer coefficient ($\mu\text{m s}^{-1}$)
m	solute distribution coefficient (–)
M	Morton dimensionless number ($g\mu_c^4\Delta\rho/\rho_c^2\gamma^3$) (–)
N_{PG}	inverse of Morton dimensionless number (–)
Pe	Peclet number ($Re Sc$) (–)
R^2	coefficient of determination (–)
Re	drop Reynolds number ($\rho u_t d/\mu$) (–)
Sh	Sherwood number
Sc	Schmidt number based ($\mu/\rho D$)
T	temperature ($^{\circ}\text{C}$)
t	drops contact time and efflux time in viscometer (s)
u_t	terminal velocity (m s^{-1})
We	drop Weber number ($\rho_c u_t^2 d/\gamma$)

Greek symbols

β	eigen-value (–)
\mathfrak{R}	enhancement factor in diffusivity (–)
γ	interfacial tension (mN m^{-1})
μ	viscosity ($\text{kg m}^{-1} \text{s}^{-1}$)
ρ	density (kg m^{-3})
Δ	difference

Subscripts and superscripts

c	continuous phase, critical size
d	dispersed phase
f	final value
i	initial value
m	molecular
oe	overall effective
od	overall dispersed value
t	terminal
*	equilibrium

most proper temperatures in order to achieve higher efficiencies. A reliable strategy was employed for modeling the experimental data.

2. Experimental

2.1. Chemicals and physical properties

Acetic acid and MIBK were purchased from Merck and Aldrich with mass purities of 100% and more than 99.5%, respectively. De-ionized water with electrical conductivity of $0.06 \mu\text{S cm}^{-1}$ was used for continuous phase. Solute analysis was performed with titration method using 0.1 M NaOH (Merck) solutions.

The range of physical properties for the continuous and dispersed phases with respect to temperature variation and for both the mass transfer directions are given in Table 1. The average of initial and final concentration of solute (acetic acid) in MIBK drops for $d \rightarrow c$ and $c \rightarrow d$ mass transfer directions were 12.0 g L^{-1} ($\sim 1.48 \text{ wt}\%$) and 7.4 g L^{-1} ($\sim 0.91 \text{ wt}\%$), respectively. So, MIBK density, viscosity and interfacial tension properties were measured with these average solute concentrations. Also, for $c \rightarrow d$ mass transfer direction, concentration of acetic acid in water was 25.0 g L^{-1} ($\sim 2.5 \text{ wt}\%$). It has to be noted that mutual saturation of phases (without solute) was provided prior to experiments.

Densities were measured using an Anton Paar digital vibrating u-tube density-meter (model DMA 4500) equipped with automatic viscosity correction. The uncertainty of density measurements for this self-adjustable temperature density-meter was $\pm 0.05 \text{ kg m}^{-3}$. The temperature in the cell was adjusted to $\pm 0.01 \text{ }^{\circ}\text{C}$ with a solid-state thermostat. Measurement of viscosity was performed for both phases at different temperatures. An Ubbelohde viscometer with an uncertainty of $\pm 2 \times 10^{-3} \text{ kg m}^{-1} \text{ s}^{-1}$ was used in this regard. According to Poiseuille law, the viscosity equation is

$$\mu = \rho \left(kt - \frac{t}{c} \right) \quad (1)$$

where μ , ρ and t are dynamic viscosity, density, and efflux time, and k and c are the viscometer constants, respectively. The constant k and c parameters were obtained by measurements on standard liquids. As presented in Fig. 1, within the used temperature range of $15\text{--}40 \text{ }^{\circ}\text{C}$, viscosity of organic and aqueous phases decreases about 28.6% and 42.2%, respectively, for both mass transfer direction cases. To measure interfacial tension, drop volume method [15,16] was used at different temperatures. As shown in Fig. 2, interfacial tension decreases moderately with temperature.

The modified Wilke–Chang correlation, reported by Reddy and Doraiswamy [17] was used for predicting molecular diffusivity (D) in phases, considering the relevant physical properties for each mass transfer direction (Table 1). As illustrated in Fig. 3, a significant enhancement in molecular diffusivity of acetic acid in MIBK and water ($\sim 52\%$ and $\sim 88\%$, respectively) is relevant within the used temperature range for both directions. These variations are pertained to direct dependency of diffusivity coefficient to absolute temperature and inversely to the solvent viscosity.

Table 1

Physical properties of the chemical system with respect to temperature variation for both mass transfer directions.

Property	$d \rightarrow c$		$c \rightarrow d$	
	Dispersed phase	Continuous phase	Dispersed phase	Continuous phase
ρ (kg m^{-3})	790.8–814.1	992.1–999.1	788.2–811.5	995.2–1003.1
μ ($\text{kg m}^{-1} \text{s}^{-1}$)	$(0.499\text{--}0.693) \times 10^{-3}$	$(0.653\text{--}1.140) \times 10^{-3}$	$(0.471\text{--}0.666) \times 10^{-3}$	$(0.711\text{--}1.218) \times 10^{-3}$
D ($\text{m}^2 \text{s}^{-1}$)	$(1.66\text{--}2.51) \times 10^{-9}$	$(0.98\text{--}1.87) \times 10^{-9}$	$(1.73\text{--}2.66) \times 10^{-9}$	$(0.92\text{--}1.72) \times 10^{-9}$
γ (mN m^{-1})	8.27–8.67		8.26–8.64	

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