

Determination of single droplet sizes, velocities and concentrations with image analysis for reactive extraction of copper



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HIGHLIGHTS

- Single organic droplet copper extraction was measured using funnel and imaging.
- Droplet inner concentrations were determined from analysis of video frames.
- Results include concentration profiles and average concentrations in droplet.
- Funnel measurement leads to higher mass transfer due to indirect measurement.
- Dynamic droplet analysis is enabled using velocity, shape and concentration.

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ABSTRACT

The proposed image analysis method allows the measurement of organic phase droplet sizes, velocities, and copper concentrations in single droplet column copper extraction using hydroxyoxime complexation. The method uses image acquisition sequences from video, detection of moving droplets, binarization of background subtracted images, and noise reduction from images. The image analysis method enabled characterizing the shape of droplets, by determining the droplet minor and major axis lengths. The method can detect droplet concentration directly inside the column wherever the droplet is visible. Image based method was validated against reference samples which were analyzed using spectrophotometry. The traditional concentration measurement using the spectrophotometric analysis of column outlet sample collection was performed for comparison purposes. The direct image analysis showed smaller variation in mass transfer results because of longer and non-uniform residence times when using sample collection. However, separately collected sample analysis together with the image analysis enables determination of the copper mass transfer during all the three steps of column experiment. Image analysis can also be used to reveal concentration profiles inside the droplet. This method is not limited to extractants, but it can be applied to systems where a suitable color change is present depending on camera sensor technology.

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1. Introduction

Mass transfer is an important phenomenon affecting the design of liquid-liquid contactor units utilizing reactive extraction. To properly design the units, it is important to understand and quantitatively evaluate the effect of different mass-transfer phenomena in the whole process. These phenomena are solute transfer from bulk to the interface, interfacial reaction, and transport from interface to bulk. The mass transfer in solvent extraction depends on, among other variables, droplet sizes, velocities, and concentrations.

When the size, velocity, and inner concentration of a single droplet are determined, mass transfer into the droplet is defined.

The presence of suitable reagents enhances mass transfer between the continuous and droplet phases in the reactive extraction. Especially in industrial hydrometallurgical processes, metal extraction with complex forming extractants is in common use. Also substantial application areas of reactive extraction can be found among environmental, petrochemical, chemical, and biochemical applications (Bart and Stevens, 2004).

To experimentally investigate combined interfacial kinetics and mass transfer, different experimental methods are available (Hanna and Noble, 1985). Among these methods, single droplet measurements are widely applied in mass transfer experiments

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Nomenclature

A	absorbance, [-]
A, B, C, D, E, F	parameters of quadratic formula
c	concentration, [mol/L, mmol/L]
d	diameter [mm]
E	droplet aspect ratio [-]
g	earth gravitational acceleration [9.81 ms ⁻²]
I	light intensity, [-]
L	optical path length, [mm]
l	length or distance, [mm]
n	amount of copper, [mol, mmol]
p	pixel value, [-]
u	droplet velocity, [mm/s]
V	volume, [mL]
\dot{V}	droplet phase feed flow rate, [mL/min]
X	conversion ($X = 1 - c/c_0$), [-]

Greek alphabet

$\Delta\rho$	density difference ($\rho_c - \rho_d$), [kg/m ³]
Δc	concentration difference, [mmol/L]
ρ	density, [kg/m ³]
γ	interfacial tension, [mN/m]
ε	molar absorptivity, [L/(mmol mm)]
μ	dynamic viscosity [Pa s]

Subscripts, indices

0	initial value
aq	aqueous phase
bg	background
BOT	column bottom part
c	continuous phase
ch	chord length
cr	critical value
d	droplet or droplet phase
e	equivalent
i, j	pixel location indices
major, minor	major and minor axis of a droplet image
org	organic phase
p	value for pixel
Rise	rise
Sample	analysis from sample
t	terminal velocity
TOP	column top part

Dimensionless numbers

E_o	Eötvös number, $E_o = g\Delta\rho d^2/\gamma$
Mo	Morton number, $Mo = g\mu_c^4\Delta\rho/(\rho_c^2\gamma^3)$

of liquid-liquid systems to determine the mass transfer coefficients, interfacial kinetics and extraction efficiencies (for example, Whewell et al., 1975; Henschke and Pfennig, 1999; Kumar and Hartland, 1999; Biswas et al., 1996, 1997; Wegener et al., 2009).

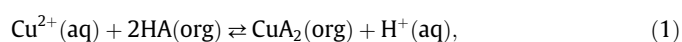
In single droplet systems, a droplet is rising or settling in an ambient continuous liquid. Droplets are collected from a funnel at the column outlet and concentrations are analyzed. Reaction kinetics and mass transfer rates can be determined from this data.

Already in the 1950s, Licht and Conway (1950) and Licht and Pansing (1953) verified that the mass transfer in single droplet extraction is divided into three stages: mass transfer during droplet formation, mass transfer in free rising/settling, and mass transfer during droplet coalescence. Experimental arrangements should be made so that contribution of each phenomena to the extraction process can be determined. It is commonly agreed, that the contribution of droplet formation time to the mass transfer can be substantial, and the related error should be taken into account in the formation of mass transfer correlations (Wegener et al., 2014; Liang and Slater, 1990; Licht and Conway, 1950; Licht and Pansing, 1953). By contrast, the effect of droplet coalescence in the column outlet collector is assumed to be negligible, which has not been clearly shown.

Traditional single droplet experiments do not provide any information on the conditions inside the droplet during its rise. For example, mass transfer leads to concentration changes inside the droplet and at the interface. These changes can generate interfacial tension gradients which in turn lead to the Marangoni convection (Wegener et al., 2009, 2014). The effect of Marangoni convection cannot be directly observed in pure concentration measurements. Because of this, it would be beneficial also to be able to follow droplet velocities and concentration profiles within the droplet, at the interface and in the near vicinity of the droplet in the ambient phase. Flow pattern and concentration front visualization inside a droplet using decolorization with pH indicator have been made by Schulze (2007) and Pawelski et al. (2005) but the concentration profiles have not been measured. Decolorization, however, has

been used to reveal Marangoni convection. Mörters and Bart (2000) and Baumann and Mühlfriedel (2002) have determined indirectly concentration profiles near the phase boundary using a laser induced fluorescence to track tracer concentrations. Baumann and Mühlfriedel measured time-dependent average tracer concentration profiles on the flat interface between two immiscible liquids. Mörters and Bart (2000), using D2EHPA system, determined time-dependent tracer concentration profiles inside a droplet to investigate diffusion inside and outside droplets in reactive extraction. Measured tracer concentration profiles were used as a basis to determine organic complex diffusion coefficient. The determination the effect of continuous phase flow on the droplet internal circulation was not successful due to experimental arrangements. In further studies by Mörters and Bart (2003), the measured concentration profiles were a basis for a Stefan-Maxwell based diffusion model for the mass transfer. Diffusion model was applied to zinc-D2EHPA single droplet experiments but the model was not able to describe experimental results satisfactorily and this is probably due to convective effect not included in the model.

In this work, the problems in single droplet extraction experiments are approached with a direct nonintrusive measurement system where the droplet velocity, droplet diameter, and concentration inside the droplet are determined by using digital imaging and subsequent image analysis. In this research, copper extraction from the aqueous solution to the organic solvent using Acorga M5640 extractant is analyzed. The interfacial reaction,



where the reactant HA (Acorga M5640) exchanges Cu-ions from the aqueous phase and the Cu-complex CuA_2 can be followed directly and visually due to color change.

In experiments, concentrations, droplet velocities, and diameters are determined as averages from several droplets to minimize the effect of experimental variability. This direct droplet concentration analysis allows exact determination of mass transfer rates during the three stages in the single droplet experiment. In tradi-

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