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Hydrodynamic Studies in Miniature Rotating Disc Contactor

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Abstract

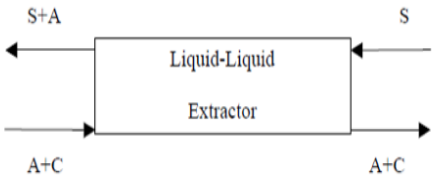
Objective: To employ the use of rotating disc contactor to extract leftover traces of Uranium and Plutonium from used fuel cells.
Methods/Statistical analysis: The hydrodynamic variables were studied under no mass transfer condition between 0.01N Nitric Acid (aqueous phase) and a solution of 30% Tri-Butyl Phosphate and 70% Dodecane (organic phase) in a miniature rotating disc contactor. There were two rounds, first with aqueous as continuous phase (organic as dispersed phase) and aqueous as dispersed phase (organic as continuous phase).The drop size analysis was carried out using Image J software and Microtrac S3500.
Findings: The empirical behavior of different hydrodynamic variables such as hold up, characteristic velocity, flooding and drop size, were observed by varying the rotor speed and inlet flow rate of the continuous phase. Consistency of empirical results were checked with standard literature correlations. Finally, discrepancy in the theoretical and experimental values were identified and explained.
Improvements: This project leaves an opportunity for development of a unified correlation to obtain the value of hold up in no mass transfer condition for different rotor speeds.

***Keywords*:** Rotating disc contactor, hydrodynamic variable, liquid-liquid extraction process, aqueous phase, organic phase.

1. Introduction

The process of liquid-liquid extraction separates a liquid feed of two or more components (solute dissolved in carrier) with a second liquid phase, called the solvent, which is immiscible (or only partly miscible) with the carrier and completely miscible solute components of the liquid feed. This process may be carried out in a number of stages either in cross current or countercurrent cascades [1].Extensive research is being conducted on hydrodynamics and mass transfer behaviour in RDC to improve its performance [2].A simple liquid-liquid extraction consists of liquid feed of two components, a solute (A) dissolved in a carrier (C), which is to be separated by using pure solvent (S) as depicted in Figure 1. Clearly, S must be immiscible or only partly miscible with C and completely miscible with A.

Figure 1. *A counter current liquid-liquid extraction process*



All components must meet certain specifications for successful extraction process. The main criteria for the extractant are high selectivity. The interfacial tension, density and viscosity are further important parameters[3].In liquid-liquid extraction process one of the phases is dispersed in another in the form of droplets. Hence study of hydrodynamic parameters such as hold up, characteristic velocity, flooding and drop size become important. Introduction of an additional component (extractant) makes liquid-liquid extraction process complex. Hence, liquid-liquid extraction process is used when other separation methods such as distillation or absorption are uneconomical, or maybe impossible. The rotating disc contactor (RDC), as one of the major extraction columns, has been widely used in petroleum refining and chemical industries on account of its high throughput, low investment, easy operation and maintenance [4].

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2.Hydrodynamic Design of RDC

The mass transfer between the flowing liquid phases in an extraction column depends, among other factors, on the contact interfacial area between continuous and dispersed phases. The interfacial area available for mass transfer in a counter-current extraction tower depends upon the volume fraction or holdup, of the dispersed phase, as well as on the mean droplet size. It is therefore important, at the design stage, to be able to predict the dispersed liquid holdup for a given system, column geometry and set of operating conditions [5].

A. Hold Up

Hold up is defined as the volume fraction of the dispersed phase to the total volume of the mixing section of the column. It is denoted by symbol *x* or ϕ. It can be calculated using following correlation given by [6]:

$$\phi = \Pi\Phi\Psi\Gamma \tag{1}$$

where,

$$\Pi = 0.19 + \left[\frac{\epsilon}{g}\left(\frac{\rho_c}{g\gamma}\right)^{0.25}\right]^{0.67} \tag{2}$$

with,

$$\epsilon = \frac{4P}{\pi d_C^2 H \rho_c} \tag{3}$$

where P can be calculated using:

$$N_P = C_1 Re_R^{C_2} \tag{4}$$

with the values of C_1 and C_2 given as:

C1 = 23.1 and C2 = -0.568 for Re < $6.74x10^4$
C1 = 0.244 and C2 = -0.155 for Re > $6.74x10^4$

$$\Phi = \left[V_d\left(\frac{\rho_c}{g\gamma}\right)^{0.25}\right]^{0.69} exp\left[7.13(V_c)\left(\frac{\rho_c}{g\gamma}\right)^{0.25}\right] \tag{5}$$

$$\Psi = \left(\frac{\Delta\rho}{\rho_c}\right)^{-0.65}\left(\frac{\mu_d}{\mu_w}\right)^{0.14} \tag{6}$$

$$\Gamma = \left(\frac{d_R}{H}\right)^{0.62}\left(\frac{d_S}{d_C}\right)^{-0.26}\left[H\left(\frac{g\rho_c}{\gamma}\right)^{0.5}\right]^{-0.1} \tag{7}$$

Static hold is defined as the volume fraction of dispersed phase which remains motionless by settling over and under the stator and rotor surface. For calculation of static hold up, suggested the following correlation:

$$\phi = 2.4x10^{-10}\left(\frac{\mu_d}{\mu_c}\right)\left(\frac{\Delta\rho}{\rho_c}\right)\left(\frac{\gamma\rho_d d_{32}}{\mu_d^2}\right)^{1.85} n^{-0.45} + 0.018 \tag{8}$$

B. Drop Size

The surface-mean diameter, d_{vs}or d_{32} (Sauter mean diameter) is most appropriate for mass transfer calculations as it gives the interfacial surface area equal to that for entire population (of drops) for the same mass of drops. The expression for d_{vs}or d_{32} is given by [7]:

$$d_{32} = d_{vs} = \frac{\sum_N n_i d_i^3}{\sum_N n_i d_i^2} \tag{9}$$

The drop size prediction equations are given as [8]:

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for$Re_D \leq 50\,000$

$$\frac{d_{32}}{D_R} = C_3 \left(\frac{ND_R^2\rho_c}{\mu_c}\right)^{-1.12} \left(\frac{\mu_c}{\gamma\rho_c D_R}\right)^{-1.38} \left(\frac{\Delta\rho}{\rho_c}\right)^{-0.24} \left(\frac{D_R^2\rho_c g}{\gamma}\right)^{0.05} \left(\frac{H}{D_R}\right)^{0.42} \quad (10)$$

and for $Re_D \geq 50\,000$

$$\frac{d_{32}}{D_R} = C_4 \left(\frac{ND_R^2\rho_c}{\mu_c}\right)^{-0.55} \exp\left[-0.23\frac{N^2 D_R}{g}\right] \left(\frac{\mu_c}{\gamma\rho_c D_R}\right)^{-1.30} \left(\frac{\rho_d}{\rho_c}\right)^{0.75} \left(\frac{D_R^2\rho_c g}{\gamma}\right)^{-0.30} \left(\frac{H}{D_R}\right)^{0.28} \quad (11)$$

C. Characteristic Velocity

The drops move in the direction of acting buoyancy forces and it accelerates until it reaches the velocity at which the buoyancy forces are equal to the drag forces. This steady velocity is called the single drop terminal velocity. In the region of small drop size and small drop Reynolds number, the dependence is linear and corresponds to Strokes' Law [9]:

For Re < 10,

$$V_T = \left(\frac{d^2 g\Delta\rho}{18\mu_c}\right) \quad (12)$$

and for Re > 10,

$$V_T = 0.249d\left(\frac{g^2\Delta\rho^2}{\rho_c\mu_c}\right)^{1/3} \quad (13)$$

Following equation for characteristic velocity has been suggested by [10]:

$$V_k = 0.077\left(\frac{\gamma\Delta\rho g}{\rho_c^2}\right)^{0.25} \left(\frac{g_c}{D_R N^2}\right) \left(\frac{D_s}{D_R}\right)^{2.1} \left(\frac{H}{D_R}\right)^{0.9} \left(\frac{D_R}{D_c}\right)^{2.4} \left\{\left(\frac{\Delta\rho}{\rho_c}\right)^{0.6} \left(\frac{\gamma^3\rho_c}{\mu_c^4 g}\right)^{0.25}\right\}^{0.5} \quad (14)$$

D. Flooding

Flooding phenomenon usually happens in extraction column where a small portion of dispersed phase comes out in the continuous phase outlet of the column. Ideally extractors are designed such that they operate near flooding to maximize productivity [9]. In practice, extractors are designed to operate at 40 to 60 percent of the predicted flooding point due to uncertainties in design and process impurity, which allows future capacity increase.

3. Experiments and Results

A. Equipment description

The RDC equipment used in the experiment consists of 25 rotating disc welded to the central vertical rotating shaft as shown in Figure 2. The rotating disc is positioned in between the stator discs so that one rotor disc and two stator discs forms one compartment. See the table1 for detailed geometry of miniature rotating disc contactor. Details are summarized in Table 1.

Table 1. RDC Dimensions

Rotor Diameter	0.053m
Stator Diameter	0.063m
Column Diameter	0.1054m
Compartment Height	0.025m
Number of compartments	25

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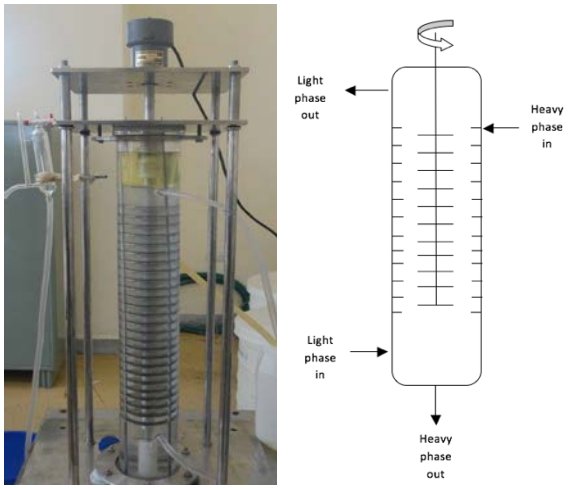
Figure 2. Miniature RDC and Schematic RDC



B. Physical properties of Aqueous and Organic Phase

The physical properties of 0.01N Nitric Acid (aqueous phase) and 30% Tri Butyl Phosphate-70%Dodecane solution are listed in Table 2.

Table 2. Physical properties

Phases	Density (kg/m^3)	Viscosity (kg/m.s)	Interfacial Tension (N/m)
0.01N Nitric Acid	997.413	0.001025	0.01038
30% Tri Butyl Phosphate-70%Dodecane	813.07	0.0016	

C. Hold Up Calculation

The continuous phase was filled in the column first and set at the desired flow rate while at the same time rotating disc contactor was started and maintained at the desired speed. The dispersed phase was introduced from the top (or bottom) of the column. The interface position was maintained just below the continuous phase inlet by adjusting the interface regulator. All the experiments were carried out for more than four residence time to reach steady state operation. At the end of the experiments the holdup of the column was measured by the usual displacement method. In the displacement method, the aqueous and organic phase inlets were closed simultaneously and the dispersed phase hold up in the column was allowed to settle at the interface. The original interface position during the experiments was noted down and the additional collected hold up over the interface was measured for hold up determination. For the measurement of hold up, the aqueous phase consisted of 0.01N nitric acid and organic phase contained 30% TBP in dodecane.

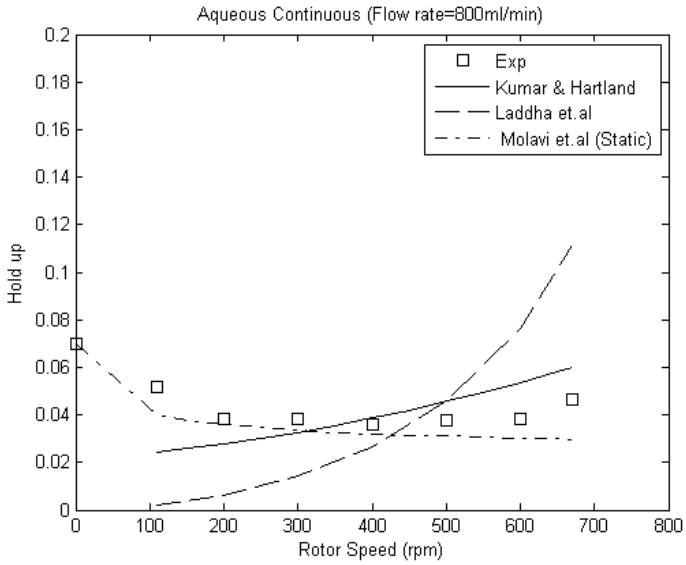
I.Aqueous phase as continuous phase

Experiments with aqueous phase (0.01 N Nitric Acid) as continuous phase and organic phase (30% TBP 70% *n*-dodecane) as dispersed phase were performed under no mass transfer condition at different rotor speeds. The flow rates of aqueous phase (continuous phase) and organic phase (dispersed phase) were respectively maintained at 800ml/min and 200ml/min. The rotor speed was varied from zero rev/min to speed at which flooding was observed. A sufficient time of 3-4 residence time was given for system to reach steady state operation and at the end of the experiment hold up was measured by displacement technique. The experimental hold up data was compared with the prediction from various literature correlations. The hold up correlations reported by [11], [10],[6] were used for the comparison with the experimental data. Among the various correlations used the reported the average static hold up only. The comparison between the experimental and predicted hold up using the literature correlation is shown in Figure 3.The results obtained from [10],[6] suggest the correlation predict increase in hold up with increasing rotor speed. This correlation takes into account the factor of decrease in drop size with increasing rotor speed hence increasing the volume fraction of dispersed phase in the mixing column.

The prediction from [11] correlation agrees well with the experimental data at low rotor speeds. This suggests that at zero rotor speed almost all the hold up comes from static hold up. Increasing the rotor speed decreases the amount

of static hold up and increases the amount of dynamic hold up. Also the result suggests that dynamic hold up account for exceedingly increasing amount of fraction of total hold up with increasing rotor speed. Hence more deviation from [11] prediction is observed as shown in Figure 3.

Figure 3. Aqueous Continuous (Flow rate=800ml/min)



At the middle region from 200 to 600 rpm the hold up remains almost constant in the region where the increase in hold up (by decrease in drop size) is countered by decrease in hold up (by decrease in static hold up). Further increase in rotor speed the static hold becomes almost constant with increasing rotor speed but the drop size keeps on decreasing. This results in an increase in total hold up.

II.Organic phase as continuous phase

Experiments with organic phase (30% TBP 70% *n*-dodecane) as continuous phase and aqueous phase (0.01 N Nitric Acid) as dispersed phase were performed under no mass transfer condition at different rotor speeds for different flow rates. The flow rates of organic phase (continuous phase) and aqueous phase (dispersed phase) were respectively maintained first at 800ml/min and 200ml/min and then at 600ml/min and 200ml/min. The rotor speed was varied from zero rev/min to speed at which flooding was observed. A sufficient time of 3-4 residence time was given for system to reach steady state operation and at the end of the experiment hold up was measured by displacement technique. The experimental hold up data was compared with the prediction from various literature correlations. The hold up correlations reported by [11],[6] were used for the comparison with the experimental data. The comparison between the experimental and predicted hold up using the literature correlation is shown in Figure 4 and Figure 5.As stated earlier, the results obtained from [6] suggest the correlation predict increase in hold up with increasing rotor speed. This correlation takes into account the factor of decrease in drop size with increasing rotor speed hence increasing the volume fraction of dispersed phase in the mixing column.

The prediction from [11] correlation agrees well with the experimental data at low rotor speeds. This suggests that at zero rotor speed almost all the hold up comes from static hold up. Increasing the rotor speed decreases the amount of static hold up and increases the amount of dynamic hold up. Also the results suggest that dynamic hold up account for exceedingly increasing amount of fraction of total hold up with increasing rotor speed. Hence more deviation from [11] prediction is observed as shown in Figure 4 and 5.At the middle region from 200 to 400 rpm the holdup remains almost constant in the region where the increase in hold up (by decrease in drop size) is countered by decrease in hold up (by decrease in static hold up). Further increase in rotor speed the static hold becomes almost constant with increasing rotor speed but the drop size keeps on decreasing. This results in an increase in total hold up.

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Figure 4.Organic Continuous (Flow rate=800ml/min)

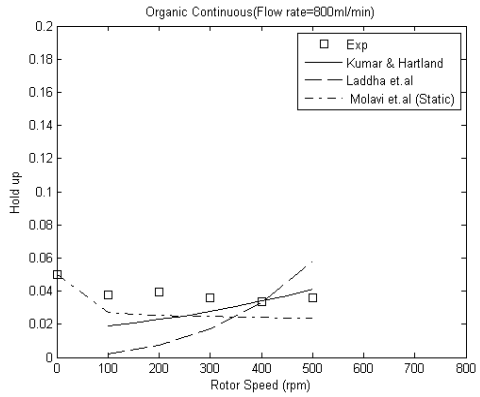
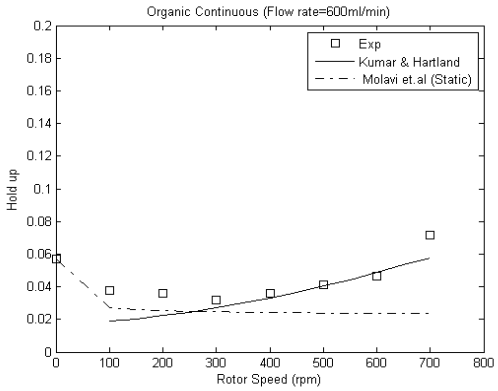


Figure 5.Organic Continuous (Flow rate=600ml/min)



D. Drop Size Measurement

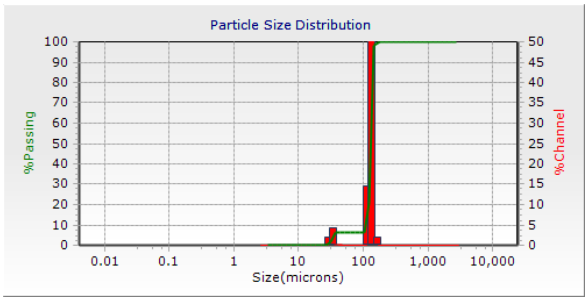
I.Image J analysis technique

Particle size measurement can be done with image analysis of sample image using various techniques. Image J is open source software available for various image processing operations. This software can be used to analyze the photograph of the sample to determine the average drop size of the system.

II.Laser diffraction analysis

The S3500 uses the phenomenon of scattered light from multiple laser beams projected through a stream of particles. The amount and direction of light scattered by the particles is measured by an optical detector array and then analyzed by using the Microtrac Software (FLEX 11). Particulate samples may be delivered in a wet or dry state, depending on the characteristics of the sample and the sample delivery equipment configured with the system. The mean diameter calculated using Microtrac was reported to be 131.3 microns as shown in the Figure 6. The results obtained from image analysis using Image J found to deviate from the experimental data. Better results for image analysis can be obtained by refining the quality of sample image by using high-speed camera with high frame rate.

Figure 6.Result obtained from Microtrac S3500 drop size analysis



E. Characteristic Velocity

The value of characteristic velocity decreases with increasing rotor speed as shown in Figure 7 and Figure 8.It was also observed that characteristic velocity does not depend on the inlet flow rate of either the continuous or dispersed phase. The characteristic velocity decreases with increasing rotor speed due to decrease in drop size as predicted[6]. Moreover, the same relation goes for the terminal velocity of drops.

F. Flooding

Flooding was observed for the both systems at different values of inlet flow rate. Decreasing the continuous phase flow rate, flooding occurs at a higher rotor speed. It was also observed that flooding for the system with aqueous as continuous phase occurs at higher rotor speed than that for the same inlet flow rate conditions for the system with organic as continuous phase. The terminal velocity of dispersed aqueous drops is lowered due to the higher viscosity of organic continuous phase, so flooding condition reaches at lower rotor speed. As the flow rate for the continuous phase is reduced keeping the dispersed phase inlet flow rate same, the dispersed phase can now

travel easily due to less resistance offered from the continuous phase flowing in the counter direction. Increasing the rotor speed produces small drops, with less terminal velocity, hence flooding occurs.

Figure 7.Aqueous Continuous

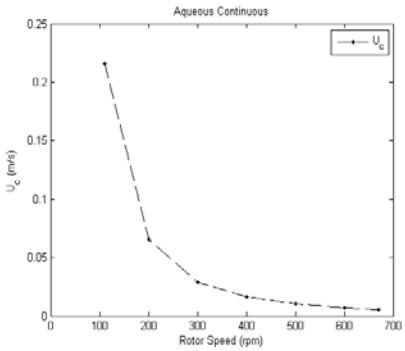
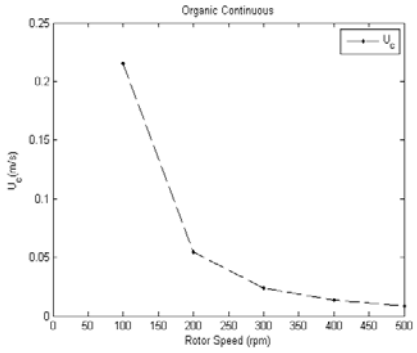


Figure 8.Organic Continuous



4. Conclusion

Hydrodynamic study is one of the important considerations for designing a column. Hydrodynamic variables such as hold up, drop size, flooding and characteristic velocity, were studied and experimentally measured. Different equationswere implemented to find the consistency of the correlations with the experimental data.

- At low rotor speed (0rpm to 400rpm) equation (8) and for high rotor speed (> 400rpm) equation (1-7) are in good agreement with the experimental data. Also it was concluded that at low rotor speed, significantly large fraction of hold up comes from static hold up.
- Flooding was observed at low rotor speed for high inlet flow rate of continuous phase and by decreasing the inlet flow rate of continuous phase, flooding shifted to higher rotor speeds. Also it was concluded that for system with aqueous as continuous phase, flooding occurs at higher rotor speed than that for system with organic as continuous phase.
- Effect on characteristic velocity with increasing rotor speed was studied using equation (14) and it was concluded that with increasing rotor speed the characteristic velocity decreases due to decrease in drop size.
- Drop size measurement for water-dodecane system, with sodium dodecyl sulphate as stabilizer, was carried out by different techniques and comparisons were drawn. The image analysis results by Image J and manual analysis were found to deviate from the experimental Microtrac S3500 particle size analyzer result.

NOMENCLATURE

C_n = Coefficients
d = drop diameter
d_{32} = Sauter mean diameter
D_c, d_C, D_C = Column diameter
d_{max}, d_m = maximum drop diameter
D_r, d_R, D_R= Disc diameter
D_s, d_S, D_S = Stator ring opening
f_v = volume fraction
g_c , g= Acceleration due to gravity
H = Compartment height
k = Film mass transfer coefficient
n = Number of compartment
N = rotor speed
N_{ODP} = Number of transfer units
P_R = Power per compartment
R = Flow ratio
v = Drop Volume
V = superficial phase velocity
V_k = Characteristic Velocity

V_{slip} = Slip Velocity
x = Dispersed phase hold-up
Z_c= Compartment Height

Greek Letters:

β, δ = Drop-size distribution parameter
γ = Surface tension
Δρ = Density difference
ε_m = Power per unit mass
μ_c = Viscosity of continuous phase
ρ_c = Density of continuous phase
σ = Interfacial tension
φ = Dispersed phase hold-up

Groups:

Fr = Froude
Np = power number = $P/N^3 d_R^5 \rho_c$
Re = Reynolds

Subscripts:

c = continuous phase
d = dispersed phase
f = flooding
i= fraction of drops of size d_i

5. Acknowledgment

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