Residence-Time Measurements in a Single-Drop Cell with Kühni Compartments

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Abstract. Extraction columns with rotating internals, such as the Kühni extractor, are used e.g. for the separation of biogenic liquid-liquid systems. In this type of extractor, compartments with impellers are placed one above the other in the column. One important parameter in column design is the residence time of the drops inside the individual compartments. The residence time, however, is strongly influenced by the compartment geometry and the operating conditions. Therefore, in this work the influence of the impeller diameter and compartment height were investigated for different drop diameters and rotor speeds. The measurements of the drops residence time for toluene (d) + water (c) system were carried out in a single-drop lab-scale cell at AVT - Thermal Process Engineering, RWTH Aachen University. The results for the discrimination of the compartment-geometry influence showed that using a bigger impeller diameter and lower compartment height leads to higher drop residence times. A very pronounced influence on the drops residence time can be found for small drop diameters.

Keywords: Residence time, Single drop, Kühni compartments, Solvent extraction

1. Introduction

Liquid-liquid extraction is a thermal unit operation that will gain in importance due to the future change in chemical feedstock. More platform chemicals will be produced from biomasses that have a lower vapour pressure and higher viscosity than crude-oil products due to comparably high oxygen content. This will, in turn, make distillation for separation less feasible. Nowadays, the safe design of technical liquid-liquid extraction columns requires experiments on pilot-plant scale. Since these experiments are extremely material and time consuming [1], industry and research are very interested in simulating pilot-scale columns to minimize the number of experiments or even to replace them. For this reason, a simulation program called ReDrop (Representative Drops) for the design of extraction columns was developed at AVT - Thermal Process Engineering of RWTH Aachen University. This program has already been successfully applied to pulsed columns and columns with rotating internals [2] with physical extraction and reactive systems. Since ReDrop follows individual drops along their path in an extraction column, all relevant phenomena in the column are taken into account, such as drop sedimentation, breakage and coalescence as well as mass transfer. In liquid-liquid extraction, impurities in the system have a strong effect on the drop behaviour in terms of sedimentation, mass transfer and coalescence. Therefore, the models accounting for these phenomena contain system-specific parameters that are obtained from single-drop experiments with the original liquid-liquid system.

One type of extraction column is the Kühni extractor. In this type of column the energy input is supplied by stirrers that are placed in compartments along the column. One main advantage of this type of column is

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that the compartment geometry may be tuned alongside the column to the local conditions, for example interfacial tension and density difference. Thus, especially for this type of column ReDrop may be advantageous for geometry optimization along the column which can be done a lot quicker and with less substance than on the basis of pilot-plant experiments. A parameter study showed that the drops residence time inside the compartments is by far the most crucial parameter for the accurate description of the column behaviour both in terms of operating limits such as flooding as well as separation performance [2]. Models in literature describing the drops residence time in Kühni compartments, however, show huge deviation from experimental data. Furthermore the experimental data which were the basis for modelling do not allow the discrimination of the compartment geometry, such as impeller diameter or compartment height since these values were not systematically varied during the experiments.

Therefore, the goal of this work was to systematically investigate the influence of these parameters on the residence time of single drops in Kühni compartments. This was done in a specially designed DN60 single-drop cell, with five Kühni compartments with the standard-test system toluene (d) + water (c). The influence of compartment height and impeller diameter was investigated by determining the drops residence time and the residence-time distribution. The rotor speed and the drop diameter were varied for each study.

2. Experimental

2.1. Material System

Distilled toluene (dispersed phase) and bi-distilled water (continuous phase) were used. The physical properties of distilled toluene and bi-distilled water at 20°C are shown in Table 1. 50 mmol/L NaCl were added to condition the bi-distilled water [3]. Both phases were mutually saturated by filling the toluene into the measuring cell, and stirring the system for 20 minutes. After that, the toluene phase was withdrawn from the cell and filled into the storage bottle for the dispersed phase.

<table>
<thead>
<tr>
<th></th>
<th>distilled toluene</th>
<th>bi-distilled water</th>
</tr>
</thead>
<tbody>
<tr>
<td>density [kg/m³]</td>
<td>865.9</td>
<td>998.1</td>
</tr>
<tr>
<td>dynamic viscosity [mPa s]</td>
<td>0.5828</td>
<td>1.009</td>
</tr>
<tr>
<td>interfacial tension [N/m]</td>
<td>0.0340</td>
<td></td>
</tr>
</tbody>
</table>

2.2. Single-Drop Experiments

The measurements of the single-drops residence time were carried out in a lab-scale cell with a diameter of 60 mm (see Fig. 1a). Five Kühni compartments which consist of five 4-blade impellers and six ring stators with an inner diameter of \(D_s = 35\) mm were installed in the cell. Here the bottom stator contains a PTFE bearing for the shaft (\(D_{Sh} = 10\) mm). Both impellers and stators are made from stainless steel (316Ti).

Fig. 1: (a) Single-drop measuring cell with Kühni compartments and (b) Hydrodynamics inside a compartment.

In the measuring cell the individual drops are produced with a defined size through a glass nozzle by using a computer-driven Hamilton syringe. The tip of the glass nozzle is positioned just above the PTFE bearing at the inlet of the bottom compartment. When the drop detaches from the glass nozzle, it starts to rise in the continuous phase from the bottom to the top. A high-speed video camera (Photonfocus DS1-D1024-160-PC-10) is used to record drop sedimentation. By means of a mirror which is installed in a 45° angle to
the viewing axis, both the front and the side view of the cell can be recorded. Of the five compartments only the middle one is recorded, since suction effects in the compartments influence the hydrodynamics in their adjacent compartments. For each stirrer speed reaching from 50 to 200 min⁻¹, drop diameter in the range from 1.56 to 4.01 mm and compartment geometry, the sedimentation of about 100 individual drops was recorded. The videos were then evaluated with a computer program, which was developed at AVT - Thermal Process Engineering, RWTH Aachen University.

Inside a compartment, there are toroidal vortices generated by the stirrer which rotate around the shaft axis in the upper and lower zone (see Fig. 1b). The toroidal vortices strongly influence drop sedimentation. In zone 1 the velocity of the toroidal vortex in the vicinity of the shaft is co-current to drop sedimentation, whereas the toroidal vortex in the vicinity of the compartment wall is counter-current to drop sedimentation. This is opposed to the velocity of the toroidal vortex in zone 3 where in the vicinity of the shaft the velocity is counter-current and in the vicinity of the compartment wall it is co-current to drop sedimentation. The toroidal vortices may, therefore, cause drops being back-mixed from zone 2 to zone 1 or from zone 3 to zone 2. In order to take these phenomena into account and to account for other stirrer geometries, the drops residence times were not only evaluated for the entire compartment but also for each zone, namely the lower zone (1), the impeller zone (2), and the upper zone (3) (see Fig. 1b).

3. Results and Discussion

Experimental results for the influence of compartment geometry, such as the impeller diameter and compartment height, shall be discussed based on the slowing factor \( k_V \) which is used in ReDrop to account for the effect of the compartment geometry on the drops sedimentation behavior. \( k_V \) is defined as:

\[
k_V = \frac{v_{\text{char}}}{v_{\infty}} = \frac{H_C}{\tau_m} \frac{v}{v_{\infty}}
\]

where \( v_{\text{char}} \) is the characteristic velocity of an individual drop inside the compartment which can be determined from the compartment height \( H_C \) divided by mean residence time \( \tau_m \). \( v_{\infty} \) is the drops terminal velocity, that is reached in an indefinitely extended continuous phase and was determined in a cell without internals. In addition, to quantify the compartment-geometry influence on the width of residence time distribution variances \( s^2 \) of the drops residence times were determined and are discussed in the results:

3.1. Influence of Impeller Diameter on Drops Residence Time

In this investigation the measurements with the impeller diameter of \( D_R = 30 \text{ mm} \) were performed and compared to the measurement results of Friebe [5] who performed experiments with an impeller diameter of \( D_R = 40 \text{ mm} \). Compartments with a height of 35 mm and stators with an inner diameter of 35 mm which corresponds to a free cross-sectional area of 30% were used.

![Fig. 2: Comparison of the slowing factors (left) and variances of the drops residence times (right) as a function of drop diameter and stirrer speed for different impeller diameters.](image)

Fig. 2 depicts the influence of different impeller diameters on the slowing factors \( k_V \) and the variance of the drops residence times for all investigated drop diameters \( d \) and rotor speeds \( N \). The slowing factor significantly decreases with the drop diameter and rotor speed. Comparing \( k_V \) for the two impeller diameters
shows that a bigger impeller leads to lower slowing factors for stirrer speeds of 100 min\(^{-1}\) and higher. This is because a smaller impeller leads to a lower intensity of the toroidal vortices inside the compartment which allows the drops to pass the compartment more easily. A pronounced influence can be found for drops with diameters of \(d = 1.56\) and 1.93 mm. Due to their lower sedimentation velocity and inertia they are affected more heavily by a change in hydrodynamics inside the compartment caused by different impeller diameters.

For both impeller diameters, a slight increase of the residence-time variances with the rotor speed can be observed for big drops, while for small drops \((d = 1.56\) mm\) this increase is a lot stronger. The variances of the drops residence times for \(D_R = 40\) mm is significantly larger than for \(D_R = 30\) mm, which becomes very evident for \(d = 1.56\) mm at rotor speeds \(N > 50\) min\(^{-1}\). This is because at high rotor speeds small drops move more randomly and are also back-mixed more often between the individual compartment zones.

In general, back-mixing increases with the rotor speed and decreases with the drop diameter (see Table 2). Since drops may be back-mixed more than once, back-mixing values higher than 100% are possible. Small drops entering one zone are back-mixed to the lower zone more easily due to their lower sedimentation velocity and inertia. High rotor speeds increase the drops probability to get hit by an impeller blade, and they are, in turn, slowed down and dragged back down into the lower zone 1. A bigger impeller \((D_R = 40\) mm\) leads to a comparably higher amount of back-mixing. For \(D_R = 30\) mm, drops bigger than \(d = 2.56\) mm were not back-mixed, even not at \(N = 200\) min\(^{-1}\), while for \(D_R = 40\) mm these drops still get back-mixed. A plausible explanation for the effect of \(D_R\) onto back-mixing is that drops can pass smaller impellers more easily without being hit by an impeller blade that would slow them down leading to a higher radial acceleration into the wall region where the lower toroidal vortex would drag them back into the lower zone 1. In the upper zone 3 back-mixing is caused by the bigger impeller generating a stronger toroidal vortex that increases the drops probability to get dragged back down into the impeller zone.

### Table 2: Percentage of back-mixing for different impeller diameters.

<table>
<thead>
<tr>
<th>drop diameter (d) [mm]</th>
<th>(D_R = 30) mm</th>
<th></th>
<th></th>
<th></th>
<th>(D_R = 40) mm</th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>zone 2 to zone 1</td>
<td>zone 3 to zone 2</td>
<td>zone 2 to zone 1</td>
<td>zone 3 to zone 2</td>
<td>zone 2 to zone 1</td>
<td>zone 3 to zone 2</td>
<td>zone 2 to zone 1</td>
<td>zone 3 to zone 2</td>
</tr>
<tr>
<td>150 min(^{-1})</td>
<td>200 min(^{-1})</td>
<td>150 min(^{-1})</td>
<td>200 min(^{-1})</td>
<td>150 min(^{-1})</td>
<td>200 min(^{-1})</td>
<td>150 min(^{-1})</td>
<td>200 min(^{-1})</td>
<td></td>
</tr>
<tr>
<td>1.56</td>
<td>1.37%</td>
<td>12.82%</td>
<td>5.48%</td>
<td>26.92%</td>
<td>18.31%</td>
<td>116.22%</td>
<td>15.49%</td>
<td>55.41%</td>
</tr>
<tr>
<td>1.93</td>
<td>0%</td>
<td>9.52%</td>
<td>0%</td>
<td>5.952%</td>
<td>11.36%</td>
<td>101.25%</td>
<td>18.18%</td>
<td>141.05%</td>
</tr>
<tr>
<td>2.56</td>
<td>0%</td>
<td>0%</td>
<td>0%</td>
<td>0%</td>
<td>1.06%</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

* no measurement results

### 3.2. Influence of the Compartment Height on Drops Residence Time

In order to discriminate the influence of the compartment height \(H_C\) the experimental results with the standard compartment height of \(H_C = 35\) mm [5] were compared to results obtained from measurements with a compartment height of \(H_C = 43.5\) mm. All other geometric values were kept constant for both compartment heights, meaning that impellers with a diameter of 40 mm and stators with a free cross-sectional area of 30% were installed in the cell. The slowing factor and the variances of drops residence times for the two different compartment heights are plotted for different drop diameters \(d\) and stirrer speeds \(N\) in Fig. 3. The compartment height strongly influences the drop residence time for small drops. It can be clearly seen that for almost all measuring points \(k_V\) is higher for \(H_C = 43.5\) mm than for \(H_C = 35\) mm. The slowing factor of 1.93 mm drops at \(N = 200\) min\(^{-1}\), e.g., is roughly 100% higher for \(H_C = 43.5\) mm than for \(H_C = 35\) mm. Varying the compartment height results in a change in the hydrodynamics in the compartment, especially the intensity and shape of the toroidal vortices in the lower and upper zone. As the results, drops can pass the compartment more easily as compared to the lower compartment.

For both compartment heights the variances of the drops residence times increase with the stirrer speed and decrease with drop diameter. The lower compartment shows larger variances of the drops residence times than the higher one, which is very evident for \(d = 1.56\) mm and \(N = 200\) min\(^{-1}\). With higher rotor speeds and decreasing drop size, drop motion becomes more random and back-mixing is promoted as shown in Table 3. A strong effect of the compartment height can be mainly found for small drops and high rotor speeds. Furthermore, the results indicate that the lower compartment promotes more back-mixing between
the zones than the higher compartment. For the higher compartment, drops bigger than 2.56 mm did not back-mix, while for the lower compartment a small percentage of these drops were still back-mixed.

![Graph showing comparison of slowing factor and variance of drops residence time as a function of drop diameter and stirrer speed for different compartment heights.]

**Table 3:** Percentage of back-mixing for different compartment heights.

<table>
<thead>
<tr>
<th>drop diameter $d$ [mm]</th>
<th>$D_R = 30$ mm</th>
<th>$D_R = 40$ mm</th>
</tr>
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<tbody>
<tr>
<td></td>
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</tr>
<tr>
<td>1.93</td>
<td>11.26%</td>
<td>101.25%</td>
</tr>
<tr>
<td>2.56</td>
<td>1.06%</td>
<td>- *</td>
</tr>
</tbody>
</table>

* no measurement results

4. **Conclusion**

The impeller diameter and compartment height of a Kühni compartment strongly influence the drops residence time. Drops in the compartment with a smaller impeller diameter spend less time, which leads to higher slowing factors. Also the variance of the drops residence times is larger when using an impeller with a bigger diameter. Thus, when using a smaller impeller in order to reduce the energy input for drop breakage, also the drops residence time and back-mixing of the drops in the compartment is reduced. It was shown that a lower compartment height leads to higher drop residence times. Especially the residence times of small drops are strongly influenced by the compartment height. It was found that the variance of the drop residence times is reduced when increasing the compartment height. Furthermore, back-mixing of the drops from one zone to the next lower zone increases with the impeller diameter and decreases with the compartment height. On the basis of the experimental findings obtained in this work the influence of the impeller diameter and compartment height on the drop movement will be taken into account by an appropriate model to describe the drops residence times in Kühni columns within the ReDrop simulation tool.

5. **Acknowledgement**

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6. **References**


