THE EFFECT OF SCALE ON LIQUID-LIQUID DISPERSION IN IN-LINE SILVERSON ROTOR–STATOR MIXERS

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Abstract. The effect of scale and processing conditions on power draw and drop size distributions in three in-line Silverson rotor-stator mixers was investigated with the aim to determine the most appropriate scaling up parameter. The largest mixer was a factory scale device, whilst the smallest was a laboratory scale mixer. All the mixers were geometrically similar and fitted with double rotors and standard double emulsor stators. Emulsification of 1 wt.% silicone oils with viscosities from 9.4 to 339 mPa·s in water containing surfactant were employed to examine the effect of rotor speed, flow rate, dispersed phase viscosity, and scale on drop size distributions, for both single and multiple pass modes of operation. It has been found that for all three scales, power draw can be correlated as the sum of two power terms, with the proportionality constants, $PoZ$ and $k_1$, practically scale independent. Sauter mean drop size appears to correlate better with tip speed than energy dissipation rate.

Keywords: rotor-stator mixer, scale-up, tip speed, energy dissipation rate, emulsification, liquid-liquid dispersion

1. INTRODUCTION

Mixing of two or more immiscible liquids to form a stable emulsion is an important processing step in the manufacture of many products such as shampoos, deodorants, salad dressings, creams, bitumen and pharmaceuticals, commonly carried out in in-line high shear rotor-stator mixers. In-line rotor-stator mixers are attractive as they can combine multiple process operations, and they may be used in continuous processing in a single pass (SP) mode or batch processing in a multiple pass (MP) mode.

Despite the widespread application of in-line rotor-stator mixers, the current understanding of the performance of rotor-stator devices is still rather limited. Frequently, the development of new emulsion-based products is based on experience, and process parameters are typically selected by trial and error at increasing scales.

This approach results in high development costs, lost time to market and considerable material wastage due to the numerous trials required at ascending scales from the laboratory to the plant. To accurately scale-up liquid-liquid dispersion processes in rotor-stator mixers it is important to understand the effect of process and formulation parameters on droplet size to predict and control the characteristic properties of multiphase products from the laboratory scale through to the manufacturing scale.

Commonly used scale-up parameters for dispersed liquid-liquid systems in geometrically similar devices include energy dissipation rate per unit mass:
\[ \varepsilon = \frac{P}{(\rho \nu V_T)} \]  

(1)

Or the rotor tip speed:

\[ U_T = \pi ND \]  

(2)

In the case of in-line (continuous) mixers the effect of flow rate and the mode of operation (single or multiple passes) can be accounted for by correlating mean drop size with energy density [1]:

\[ E_v = \varepsilon \]  

(3)

In summary, average drop size (Sauter mean diameter) can be correlated with:

\[ d_{32} \propto \varepsilon^b \]  

(4)

\[ d_{32} \propto U_T^b \]  

(5)

\[ d_{32} \propto E_v^b \]  

(6)

However, it is not clear which of the above is suitable for in-line rotor-stator mixers.

Power consumption of in-line rotor-stator mixers in turbulent flow is the sum of the power required to rotate the rotor, power resulting from the flow, and losses \( P_L \) which are typically neglected [2, 3]:

\[ P = P_0 + \rho_c N^3 D^5 + k_i M N^2 D^2 + P_L \]  

(7)

Eq. (7) has been validated for a pilot plant [4] and small scale [5] Silverson mixer.

In this work the effect of power draw and average drop size/drop size distributions of three geometrically similar in-line Silverson rotor-stator mixers has been investigated. This study significantly builds on previous work [5] by investigating a factory scale device and the effect of single and multiple passes. At each scale the effect of rotor speed, flow rate and dispersed phase viscosity on mean drop size has been investigated.

2. MATERIALS AND METHODS

2.1 Equipment

![Figure 1. Experimental rig for investigation of emulsification in in-line Silverson rotor-stator mixers for single pass (SP) and multiple pass (MP) modes](image)
The experimental rig shown in Fig. 1 consisting of two mixing vessels and an in-line Silverson rotor-stator mixer enables investigation of single pass (SP) and multiple pass (MP) emulsification. Coarse emulsions were prepared in an 800 L mixing tank for the single pass experiments and a 60 L tank for the multiple pass experiments using high shear dissolver disks and were pumped to the mixer with flow rate measured by Coriolis flow meter.

The in-line Silverson rotor-stator mixers investigated are shown in Fig. 2, fitted with double rotors enclosed between double Silverson emulsor screens. In all mixers the rotor-stator gap width was 0.25 mm and the size of the stator holes was 1.59 mm.

![Silverson mixer](image)

**Figure 2.** Details of the (a) Laboratory scale, (b) Pilot plant scale and (c) Industrial scale in-line Silverson rotor-stator mixers investigated

### 2.2. Materials

In all three mixers, emulsification of 1 wt.% silicone oils (Dow Corning 200 fluid) with viscosities of 9.4 and 339 mPa·s in water were investigated, and all emulsions were stabilised by 0.5 wt.% of sodium laureth sulphate (SLES, Texapon N701, Cognis UK Ltd.) surfactant.

### 2.3. Emulsification

Silicone oil was added to a solution of SLES in the mixing vessels at 25°C and agitated for 5 minutes to produce a dispersion of approximately 45-50 µm drops. For the single pass runs, drop size distributions (DSDs) were measured at a range of rotor speeds shown in Fig. 2, at the mixer inlet and outlet using a Mastersizer 2000 particle size analyser (Malvern Instruments, UK). The effect of number of passes on DSD was investigated at selected rotor speeds at a constant flow rate.

### 2.4. Power draw

Power draw at each scale was measured by calorimetry and the losses due to friction in the bearings were determined using a bladeless rotor [4]. Power was calculated from an energy balance across the Silverson mixing head under steady state temperature conditions:

\[ P = MC_\rho(\Delta \theta) \]  

(8)
3. RESULTS AND DISCUSSION

3.1 Power Draw

Fig. 3a shows dimensionless power draw as a function of dimensionless flow rate measured at three different scales. 

![Figure 3a](image-url)

**Figure 3a.** Dimensionless power draw as a function of dimensionless flow rate and (b) Predicted power draw from Eq. (7) versus measured power draw for all scales.

Power constants in Eq. (7) obtained for each scale are summarised in Table 1. $P_{0Z}$ (~0.24) is practically scale independent for geometrically similar systems, whilst $k_1$ varies with scale between 7.5 and 11.8. Fig. 3b illustrates that Eq. (7) can be applied to accurately predict power draw at different scales.

Table 1. Power draw constants for three scales of mixer determined by calorimetry

<table>
<thead>
<tr>
<th>Power constant</th>
<th>088/150</th>
<th>150/250</th>
<th>450/600</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zero flow power constant, $P_{0Z}$</td>
<td>0.254</td>
<td>0.229</td>
<td>0.231</td>
</tr>
<tr>
<td>Flow power constant, $k_1$</td>
<td>9.59</td>
<td>7.46</td>
<td>11.80</td>
</tr>
</tbody>
</table>

3.2 Single Pass Emulsification

![Figure 4](image-url)

**Figure 4.** Drop size as a function of (a) energy dissipation rate and (b) tip speed for emulsions of 1 wt.% 9.4 and 339 mPa-s silicone oils at three scales for a single pass at $t_R = 0.45$ s.
Sauter mean diameters of both oils at all three scales as a function of energy dissipation rate and tip speed at a constant average residence time of 0.45 s are summarised in Fig 4. Fig. 4a shows that Sauter mean diameters at three scales are scattered around straight lines of slopes of -0.33 for high viscosity oil and -0.39 for low viscosity oil, with coefficients of determination ($R^2$) of 0.899 and 0.931, respectively. The exponents on $\varepsilon$ are relatively close to -0.4, indicating that turbulent inertial forces determine the maximum stable drop size.

The dependency of Sauter mean diameters on tip speed shown in Fig. 4b is similar to the dependency on energy dissipation rate, with gradients of $b = -0.98$ and $b = -1.13$ for the high and low viscosity oil, respectively. For tip speed however, the fit of the experimental data described by the determination coefficients is nearly perfect ($R^2 \approx 1$), which indicates that tip speed can be treated as a better correlating parameter than energy dissipation rate for geometrically similar in-line high shear mixers.

The effects of the scale, dispersed phase viscosity and rotor tip speed on drop size distributions are summarised in Fig. 5. For the lower viscosity oil, the DSDs are practically log-normal (Fig. 5a), while the high viscosity oil DSDs are strongly skewed towards smaller drops (Fig. 5b). At the lower tip speed, the volume of oil in smaller drops is reduced as expected, and a tail of small drops is formed for both low and high oil viscosities. In general, the DSDs are very similar for all scales, indicating that small scale experiments can give representative mean drop size values and equivalent DSDs compared to the large scale.

Figure 5. Drop size distributions at tip speeds of 10 and 20 m s$^{-1}$ for emulsions of 1 wt.% (a) 9.4 mPa·s and (b) 339 mPa·s silicone oil at three scales for a single pass at $t_R = 0.45$ s

The 3.3 Multiple Pass Emulsification

Fig. 6 shows mean drop size after 40 mixer passes as a function of energy density and tip speed. Fig. 6 shows that Sauter mean diameters in multiple pass systems do not correlate well with energy density, which is particularly true at the largest scale, despite the fact that energy density has been reported as a good correlating parameter for energy intensive continuous processing devices such as colloid mills and high pressure homogenisers [1]. However the poor correlation in Fig. 6a indicates that energy density is not suitable for high shear mixers, since the exponent on $\varepsilon$ and $t_R$ should not be equal.

On the other hand, tip speed as a function of Sauter mean diameter provides a good fit, with a $R^2$ regression value of 0.949. Hence for both single and multiple passes, tip speed appears to be useful as a scaling parameter for emulsification in rotor-stator mixers.

In Fig. 6, the number of passes at each scale is 40, however the total residence time ($t_R$) is greater at the largest scale. Fig. 6b indicates that at a constant tip speed, drop sizes for the 450/600 large scale mixer are only slightly smaller than the other scales despite $t_R$ being 15 times greater, implying that the effect of $t_R$ on $d_{32}$ is marginal.
Figure 6. Drop size as a function of (a) energy density and (b) tip speed for emulsions of 1 wt.% 9.4 mPa·s silicone oil at three scales for multiple passes (40 passes).

4. CONCLUSIONS

Measurement of the power draw constants for three in-line rotor-stator mixers indicate that $P_{OZ}$ appears to be roughly scale independent while $k_1$ is similar at different scales. Correlation of mean drop size data from the laboratory scale to the factory scale suggests that tip speed is a better correlating parameter than energy dissipation rate for single and multiple passes. Multiple pass data indicates that the effect of mean residence time on the drop size distributions is marginal.

5. NOMENCLATURE

| $b$ | Exponent (-) |
| $C_P$ | Specific heat capacity (J kg$^{-1}$ K$^{-1}$) |
| $D$ | Outer rotor diameter (m) |
| $d_{32}$ | Sauter mean diameter (m) |
| $E_v$ | Energy density (J m$^{-3}$) |
| $k_1$ | Flow power constant (-) |
| $M$ | Mass flow rate (kg s$^{-1}$) |
| $N$ | Rotor speed (s$^{-1}$) |
| $P$ | Power (W) |

Dimensionless numbers

| $P_0$ | Dimensionless power draw $P/(\rho_c N^3 D^5)$ |
| $N_Q$ | Dimensionless flow rate $Q/(\rho_c N D^3)$ |

6. REFERENCES