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# Linking continuous and recycle emulsification kinetics for in-line mixers



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#### ABSTRACT

In-line high-shear mixers can be used for continuous or batch dispersion operations depending on how the pipework is arranged. In our previous work (Carrillo De Hert and Rodgers, 2017a) we performed a transient mass balance to establish the link in-between these two arrangements; however this model was limited to the estimation of the mode of dispersed phases yielding simple monomodal drop size distributions. In this investigation we expanded the previous model to account for the shape of the whole drop size distribution. The new model was tested by performing experiments under different processing conditions and using two highly viscous dispersed phases which yield bimodal drop size distributions. The results for the continuous arrangement experiments were fit using two log-normal functions and the results for the recycle arrangement by implementing the lognormal function in the previously published mass balance. The new model was capable of predicting the  $\bar{d}_{32}$  for different emulsification times with a mean absolute error of 12.32%. The model presented here was developed for liquid blends, however the same approach could be used for milling or de-agglomeration operations.

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#### 1. Introduction

In-line rotor-stators (RS) are one of the most promising equipments that have been implemented to intensify dispersion processes. Their increase in popularity has been due to their capacity to generate highly-localised strong energy dissipation regions. In-line RS are installed in the pipework and two different types of operation arrangements are possible depending on the direction of the outflow of the RS. (1) If the outflow is directed to a secondary vessel or process, the RS is operated in a continuous fashion; evidently the material can be processed n number of times to cause further size reduction, "multi-pass processing" occurs when the material is processed *n* > 1 times. (2) On the other hand, if the outflow is directed to the feeding tank it is operated in a recycle arrangement and processing time and volume becomes important processing parameters. The schematic representation of both configurations is shown in Fig. 1.

#### 1.1. Previous study: continuous arrangement

In one of our previous studies (Carrillo De Hert and Rodgers, 2017a), we installed a pump at the feed of the RS to control the flow rate  $\dot{Q}$  and studied the emulsification kinetics using a 10 cSt silicon oil (SiOil). The 10 cSt SiOil yielded monomodal drop size distributions (DSD). The characteristic measure of central tendency used was the mode  $Mo_n$  of the DSD, where the sub-index refers to the *n*th pass. For a RS working at given stirring speed N, the multi-pass data for the continuous arrangement was well correlated by

$$Mo_n = A_1 \left(\frac{n}{\dot{Q}}\right)^{-\frac{1}{5}} N^{-\frac{6}{5}}$$
 for  $n = 1, 2, ..., n-1, n$  (1)

where  $(n/\dot{Q})$  is proportional to the residence time of the material inside the RS. We attribute the effect of mean residence time to the internal recirculation or fluid re-

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#### Nomenclature

Latin cumbolc

	Lutin Syn	IDOIS
	Ż	flow rate (m <sup>3</sup> s <sup>-1</sup> )
	$\bar{d}_G$	logarithmic mean of the log-normal distribution (m)
	$\bar{d}_{G,n,x}$	logarithmic mean of the log-normal distribu-
	-,,.	tion for the nth pass for: $x=L$ large and $x=s$
		small drops (m)
	Īres T	mean residence time in the tank (s)
	Ai	ith fit constant (–)
	B;	ith fit constant (–)
	d <sub>i</sub>	diameter of the ith drop class (m)
	$f_{unx}(d_i)$	frequency of the ith drop size after n passes for
	J 0, 11, 12 ( 17	x=L large, $x=s$ small or $x=T$ total distribution (-)
	f <sub>v.Rec</sub> (d <sub>i</sub> )	frequency of the ith drop size for the recycle
	<b>J</b> <sup>0</sup> ,100(1)	arrangement (–)
	$F_{v}(d_{i})$	cumulative distribution of the ith drop size (–)
	Mon	mode of the distribution for <i>n</i> passes (m)
	Mo <sub>n,x</sub>	mode of the distribution after <i>n</i> passes for: $x = L$
		large or $x = s$ small drops (m)
	Mo <sub>Rec</sub>	mode of the distribution in the recycle arrange-
		ment (m)
	Ν	stirring speed (s <sup>-1</sup> )
	n	number of passes (–)
	R <sup>2</sup>	coefficient of determination (–)
	SG	logarithmic standard deviation of the log-
		normal distribution (m)
	t	time (s)
	$V_T$	volume of the vessel (m <sup>3</sup> )
	Greek symbols	
	$\mu_d$	viscosity of the dispersed phase (Pas)
	$\phi_n$	volume fraction of the material for <i>n</i> -passes (-)
	$\phi_{s,n}$	relative volume fraction of the small drops after
		n-passes (–)
Dimensionless numbers		
	Ро	power number PN $^{-3}$ D $^{-5} ho^{-1}$
Abbreviations		
	DSD	drop size distribution
	RS	rotor-stator
	SiOil	silicon oil
	51011	

entrainment inside the RS, this has been reported by authors such as Özcan-Taskin et al. (2011) and Mortensen et al. (2017) using particle image velocimetry and by Xu et al. (2014) using



laser Doppler anemometry. Therefore the lower  $\dot{Q}$  the more times the emulsions re-enters the region where drop breakup occurs.

We did not do experiments at different scales, however there is strong evidence that the characteristic volume of the RS is its swept volume (Hall et al., 2013). Hall et al. (2011) did not find a correlation in-between the coarse emulsion (n = 0)and the one at the outlet of the RS, therefore in the continuous arrangement all the information of the coarse emulsion is lost after one pass is completed.

In a second study (Carrillo De Hert and Rodgers, 2017b), we extended Eq. (1) to account for the effect of the viscosity of the dispersed phase  $\mu_d$  by doing experiments using SiOils in the 10 cSt–30,000 cSt range. We found that SiOils thicker the 350 produced bimodal DSD. The mode of the large  $Mo_{n,L}$  and the mode of the small  $Mo_{n,s}$  drops were used to characterise the drop sizes of the emulsion, for the same geometry used in (Carrillo De Hert and Rodgers, 2017a) the following correlations were obtained for the SiOil in the 10 cSt–2760 cSt range

$$Mo_{n,L} = 1.18 \times 10^5 \mu_d^{0.365} N^{-1.05} \left(\frac{n}{\dot{Q}}\right)^{-\frac{1}{5}}$$
 (2)

$$Mo_{n,s} = 1.69 \times 10^3 \mu_d^{-0.365} N^{-1.05}$$
<sup>(3)</sup>

Furthermore we correlated the DSDs using a simple mixing rule

$$f_{\nu,n,T}(d_i) = (1 - \phi_{s,n}) f_{\nu,n,L}(d_i) + \phi_{s,n} f_{\nu,n,s}(d_i)$$
(4)

where  $f_{v,n,T}(d_i)$  is the total drop size distribution;  $f_{v,n,L}(d_i)$ and  $f_{v,n,s}(d_i)$  are the drop size distribution of the large and small drops respectively; and  $\phi_{s,n}$  is the volume fraction of the small drops for the *n*th pass through the RS. We further used Generalised Gamma probability density functions for  $f_{v,n,L}(d_i)$  and  $f_{v,n,s}(d_i)$  and the power-law function below for  $\phi_{s,n}$ 

$$\phi_{\mathrm{s},n} = C_{\phi,0} \mu_d^{C_{\phi,\mu_d}} N^{C_{\phi,N}} \left(\frac{n}{\dot{Q}}\right)^{C_{\phi,\tilde{t}}}$$
(5)

#### 1.2. Previous study: recycle arrangement

In the recycle arrangement (see Fig. 1b) a coarse emulsion (t = 0) is pumped through the RS and back into the its feeding vessel. For t > 0 it is expected to have a mixture of material that has passed n times, the fraction of each material  $\phi_n$  is a function of time t, volume of the vessel  $V_T$  and of  $\dot{Q}$ . As can be expected, the DSD distribution of the coarse emulsion is important in this arrangement until its complete consumption.



Fig. 1 - Schematic representation of (a) the continuous arrangement and (b) the recycle arrangement.

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