Doctoral Thesis

# **Study on a** Homogenization Effect **and a Scale-Up of Batch Rotor-Stator Mixers**

(バッチ式ローター・ステーター型ミキサーの均質化効果と スケールアップに関する研究)

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# **Introduction**

# Main purpose of this study

Rotor-stator mixers are used in chemical, biochemical, food-processing, cosmetic, and pharmaceutical industries to improve process efficiency and product quality. Despite widespread use of these mixers, evaluation methods and scale-up criteria for this type of mixers are still unknown.

The purpose of this study is to propose a theoretical evaluation method of the homogenization effect for different stator configurations of internally and externally circulated batch rotor-stator mixers. Further, another aim of this study is to derive the scale-up factor in relation to the mixing time and to confirm the validity of the scale-up procedure proposed in this thesis.

# Outline and scope of this study

This thesis is divided into five chapters as shown in Figure 1-1. The Scheme image of this study is shown in **Figure I-2**. The outlines of each chapter are as follows:

Chapter 1 provides an explanation of the background of this study and the current knowledge about general rotor-stator mixers. The objectives of this study are described in this chapter. Further, problems related to the actual operation of rotor-stator mixers and necessities of a solution to these problems are discussed.

In chapter 2, a scale-up factor for the mean drop diameter in externally circulated batch rotor-stator mixers is proposed. The homogenization index *(HI)* is defined as the scale-up factor obtained

from the local energy dissipation rate of turbulence and the circulation number. The local energy dissipation rate is calculated using the volume of the homogenization region, and the local power consumption is obtained from the measured net power consumption and the flow rate. The mean drop diameter using a certain time interval for different configurations of mixers and different operating conditions is also measured in order to evaluate the validity of the scale-up index using *H.I.* Experimental results show that *HI.* could well account for the mean drop diameter under different rotor speed conditions (13-27 m/s), with a different gap width between the rotor and the stator (0.15-0.25 mm) and different sizes of mixers and production volumes (rotor diameters were 30 and 57 mm; production volumes were 1.5 and 9 L). These results indicate that *H.I*. can be used for predicting the mean drop diameter. The results also suggest that the scale-up criteria for the mean drop diameter in relation to the mixing time is based on *HI.,* and not necessarily on geometrical similarities, same rotor tip speed, or gap width in the case of similar mixer configurations and the experimental production volume range (1 to 10 L) used for the model product, which is similar to typical dairy foods.

**In chapter** 3, a theoretical evaluation method of the homogenization effect for different stator configurations of internally circulated batch rotor-stator mixers is proposed. The homogenization effect has been evaluated indirectly using a homogenization coefficient  $(C_h)$  based on the measured power number, estimated flow number, and calculated shear frequency derived from the number of

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rotor blades and stator holes. As a result of the  $C_h$  evaluation during operation with water, it is inferred that the homogenization effect is considerably high when the stator hole diameter is small and the number of stator holes is large. The results also suggest that the clearance between the rotor and the stator has less influence on the homogenization effect than the hole diameter and the number of holes. In the case of operation with the model product, the same trend as that for the operation with water is obtained. On the basis of this fact, it has been considered that the performance estimation using  $C_h$  has a high degree of adequacy. The total energy dissipation rate that is contained in  $C<sub>h</sub>$  can well account for the mean drop diameter in relation to the mixing time. The results demonstrate that the homogenization effect for different configurations of rotor-stator mixers can be compared and evaluated using the calculated  $C<sub>h</sub>$  of the water mixing operation without actual product trials. This information can be useful for determining the optimum mixer design and the adequate mixer selection.

In **chapter 4,** the scale-up factor for the mean drop diameter in internally circulated batch rotor-stator mixers is proposed. The total energy dissipation rate  $(\varepsilon_t)$  is used as the scale-up factor for the mean drop diameter.  $\varepsilon_t$  is calculated from the power number, flow number, rotational speed, and the number of rotor blades and stator holes. Because it is difficult to directly measure the circulation flow rate of an internally circulated mixer, the flow rate of a production-scale mixer is estimated by measuring the time required for complete mixing-termed "completely mixed time" and the power number of

the production- and pilot-scale mixers and the flow number of the pilot-scale mixer. The experimental results indicate that  $\varepsilon_t$  can account for the mean drop diameter. Theoretical verification suggests that  $\varepsilon_t$ includes information about differences in the mixer configuration (rotor diameter, stator hole diameter, stator wall thickness, stator opening ratio, and gap width) and manufacturing conditions (mixing time:  $t_m$ ; rotational speed: N; and total product volume: V). The results show that the mean drop diameter decreases in proportion to  $t_m$ ,  $N^4$ , and  $V^{-1}$ . Then, the mean drop diameter for the production-scale mixer is estimated from a model product experiment with the pilot-scale mixer. These results also indicate that the scale-up criteria for the mean drop diameter can be determined on the basis of  $\varepsilon_t$  in terms of the mixing time and not necessarily in terms of the geometric similarities between mixer configurations, constant rotor tip speed, or constant clearance between the rotor and the stator.

**Chapter 5** presents a summary of the results and/or conclusions of this study and a discussion of the future work.

# **List of author's published papers**

- (1) Kamiya, T., M. Kaminoyama, K. Nishi and R. Misumi; "Scale-Up Factor for Mean Drop Diameter in Batch Rotor-Stator Mixers," J. *Chern. Eng. Japan,* 43, 4, 326-332, (2010)
- (2) Kamiya, T., H. Sasaki, Y. Toyama, K. Hanyu, M. Kaminoyama, K. Nishi and R. Misumi; "Evaluating Method of Homogenization Effect for Different Stator Configurations of Internally Circulated Batch Rotor-Stator Mixers," J. *Chern. Eng. Japan,* 43,4, 355-362, (2010)
- (3) Kamiya, T., T. Sugawara, H. Sasaki, T. Tomita, K. Hanyu, M. Kaminoyama, K. Nishi and R. Misumi; "Scale-Up Factor for Mean Drop Diameter in Internally Circulated Batch Rotor-Stator Mixers," J. *Chern. Eng. Japan,* submitted (2010)

# **List of author's orally presented papers**

- (1) Guest lecture at Aalborg University (Invited from Professor Lasse Rosendahl) Kamiya, T., "Particle Size Reduction and Emulsification in High Shear Vacuum Mixers," November 7th 2008, at Aalborg University, Department of Energy Technology (Denmark).
- (2) Kamiya, T., M. Kaminoyama, K. Nishi and R. Misumi; "Scale-Up Factor for Mean Drop Diameter in Batch Rotor-Stator Mixers," SCEJ Regional Meeting, Yonezawa, Japan (2009)

# **Patent**

(1) Kamiya, T., T. Asou, T. Tomita and H. Echizen, "Production Method of liquid enteral formula" (in Japanese), Japanese Patent Disclosure No. 2007-046227

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Fig. I-I Flow scheme of this study



**Fig. 1-2** Scheme image of this study

# Chapter 1 Background and motivation of this study

# 1.1 Background of this study

### 1.1.1 Characteristics of rotor-stator mixers

Rotor-stator mixers are used in chemical, biochemical, food-processing, cosmetic, and pharmaceutical industries to improve process efficiency and product quality. A distinguishing feature of rotor-stator mixers is that a high-speed rotor (a driven mixing element) operates in close proximity to a stator (a fixed mixing element). Typical rotor tip speeds range from 10 to 50 m/s. These mixing units generate a high shear rate that ranges from 20,000 to 100,000  $s^{-1}$ (Atiemo-Obeng and Calabrese, 2004). Therefore, these devices are also called high-shear mixers. The local energy dissipation may be three orders of magnitude greater than that in a conventional mechanically agitated vessel. High speed, high shear, and high power are the main characteristics of rotor-stator mixers.

They are employed in many process operations that involve homogenization, dispersion, emulsification, grinding, dissolving, chemical reaction, and cell disruption.

### 1.1.2 Summary of current knowledge about rotor-stator mixers

Despite widespread use, the current understanding of rotor-stator mixer devices is not definitive. Over the past few decades, several studies on rotor-stator mixers have been conducted.

With regard to the maximum stable drop size, Davies (1985, 1987) reported that the drop size decreases with an increase in the

energy dissipation rate per unit mass. This phenomenon has been confrrmed in different types of emulsification devices (such as static mixers, turbine impellers, colloid mills, and fine valve homogenizers).

Davies (1985, 1987) also indicated that a turbulent fluctuation velocity, and not the shear rate, is responsible for drop breakup.

Calabrese *et al.* (2000, 2002) have reported further details of the drop breakup mechanism in a rotor-stator mixer. From their investigations, it was found that a shear gap between the rotor and the stator was not a major factor responsible for the drop breakup. It is more likely that breakup occurs in the stator slots or in the jet emanation from the stator slot. However, a narrow gap in the stator slot contributes to a high level of turbulent kinetic energy in it. Barailler *et al.* (2006) have also reported the importance of shear gap.

Maa and Hsu (1996) compared the equilibrium drop size between different sizes and configurations of rotor-stator sets (four-blade rotors with 12 rectangular slots and two-blade rotors with 8 slant slots). They also compared the trend in drop size reduction of the different sets.

Bourne and Garcia-Rosas (1986) studied the micro-mixing performance of different rotor-stator configurations in the same mixing device (six types of rotors and two types of stators). They concluded that there is an optimum combination of rotor-stator sets; however, the stators had a negligible effect on both the predicted energy dissipation and the measured product distributions in the mixing systems.

Urban (2006) reported the drop size for different types of

emulsification devices (such as rotor-stator systems, disc systems, and high-pressure systems). Although he compared the mean drop diameter experimentally, he did not discuss the theoretical effects of differences in mixer configurations.

A study on the energy dissipation rate of a rotor-stator mixer was conducted by Utomo *et al.* (2008). From their results of complete three-dimensional computational fluid dynamics simulations, it was found that the highest energy dissipation rate occurred around the leading and trailing edges of the stator slots. They also indicated that a scale-up procedure should be based on the constant energy dissipation rate per unit mass and geometrical similarities, rather than the constant tip speed and constant gap width as proposed by other researchers (Bourne and Studer, 1992; Atiemo-Obeng and Calabrese, 2004).

In addition to the above-mentioned factors, another scale-up factor termed shear number derived from the shear frequency and shear rate has been proposed by Porcelli (2002). This proposal considered the number of rotor blades and stator openings.

Kowalski (2009) reported the power consumption of in-line rotor-stator devices.

#### **1.1.3 Summary of current knowledge for mean drop diameter**

Mechanisms of liquid-liquid dispersion and breakup of oil drops in agitating vessels were conducted by Ali *et al.* (1981), Chang *et al.* (1981), Tatterson and Stanford (1981). They reported that two different dispersion mechanisms, ligament stretching and turbulent fragmentation were observed to occur in the vortex systems of

impeller discharge. And they also investigated the dispersion of low viscosity liquid into high viscosity liquid.

Clark (1988a, 1988b) explained that the local viscous effect in the breakup of droplets smaller than the Kolmogoroff micro-scale and inertial effects in the breakup of droplets larger than Kolmogoroff micro-scale.

With regard to the mean drop diameter, many researches have been carried out. Especially, many researchers were interested in the relationship between Weber number and drop size (Mlynek and Resnick (1972), Konno *et al.* (1977), McManamey (1979), Gnanasundaram et al. (1979), Konno et al. (1983), Konno and Saito (1987), Baldyga and Bourne (1993), Konno *et al.* (1993)). Generally, the average drop size in agitated liquid-liquid dispersion can be correlated as following equation.

$$
\frac{d_{32}}{D} = C'(We)^{-0.6}
$$
 (1-1)

Here, d*32;* Sauter mean diameter [m], D: impeller diameter [m], C': constant [-] and We: Weber number [-]. Zhou and Krestra (1998) reviewed results of many similar works.

For the scale-up, several index which estimate the mean drop diameter were proposed. These indexes are based on the energy dissipation rate. Average drop size in an agitated liquid-liquid dispersion can be correlated as

$$
d_{32} = C_1 \varepsilon^{-2/5} \left( D/D_0 \right)^{6/5} f(\phi) f(\mu) (\sigma/\rho)^{3/5}
$$
 (1-2)

or

$$
d_{32} / D = C_2 W e^{-3/5} N_p^{-2/5} f(\phi) f(\mu)
$$
 (1-3)

in the breakup-dominant region, and the average energy dissipation rate per unit mass of liquid  $\epsilon$  and the Weber number in mixing vessel *We* are shown as follows.

$$
\varepsilon = (4N_p / \pi) N^3 D^2 \left( D^3 / (D_0 H) \right) \tag{1-4}
$$

$$
We = N^2 D^3 \rho / \sigma \tag{1-5}
$$

 $f(\Phi)$  and  $f(\mathcal{L})$  in these equations present the effect of volume fraction and the effect of liquid viscosity, respectively.

Average drop size in the coalescence region is also shown by the following equation.

$$
d_{32} = C_1^{\ \prime} \varepsilon^{-1/4} \big( D/D_0 \big)^{3/4} f'(\phi) f(\mu) (\sigma / \rho)^{3/8} \tag{1-6}
$$

or

$$
d_{32} / D = C_2 W e^{t-3/8} N_p^{-1/4} D^{-3/8} f'(\phi) f(\mu)
$$
 (1-7)

Then interfacial area of drops in dispersion, a, is obtained by

$$
a = 6\phi/d_{32} \tag{1-8}
$$

It has been reported by the presence authors from turbulent measurements using hot-film anemometry that the distribution pattern of the local energy dissipation rate in a mixing vessel is independent of vessel scale when geometrical similarity is preserved and that the average value throughout the vessel is shown by the following equation to give (2).

$$
\varepsilon = P/(\rho \cdot V) \tag{1-9}
$$

where P and V are agitation power consumption and product volume in the vessel.

Nishikawa *et at.* (1987) reported following index for scale-up.

$$
\left(N^3 D^2 D_0\right) \cdot \left(\frac{4N_p}{\pi}\right) \tag{1-10}
$$

Here, *N*: rotational speed [1/s],  $D_0$ : Vessel diameter [m] and  $N_p$ : Power number [-]. They also reported that the average energy dissipation rate, Weber number in the mixing vessel and Reynolds number is not a satisfying scale-up standard.

On the other hands, Pacek *et at.* (1999) indicated that the energy dissipation rate per swept volume was well account for the mean drop diameter in the agitated vessel.

Hong and Lee (1985) was developed a general correlation for

the changes of the Sauter-mean droplet diameter with respect to mixing time during the initial period of liquid-liquid dispersions in agitated vessels as follows:

$$
\frac{d_{32} - d_{32}^*}{d_{32}^*} = \alpha (N \cdot t_m)^{-0.7}
$$
 (1-11)

Here,  $d_{32}^*$ : the steady-state drop size [m]  $\alpha$ :constant [-] and  $t_m$ : mixing time [s].

Most researches were focused on normal agitated vessels, not on rotor-stator mixes. Thus far, the investigation of concerning about mean drop diameter for rotor-stator mixers has been required.

## **1.1.4 Main problem of rotor-stator mixers in production fields**

Although a rotor-stator mixer is a highly efficient device, its drop-size distribution is generally considerably broader than that of a fine-clearance valve homogenizer. This is why rotor-stator mixers are usually used in pre-emulsification processes in an actual production line. From an engineering viewpoint, for the design of production lines, obtaining information about the decreasing trend of the mean drop diameter is more important than obtaining information about the maximum stable drop size, which is obtained over a long mixing time.

Thus far, researchers have focused on pilot-scale mixers and the maximum stable drop size that is obtained over a long mixing time. However, studies have not been conducted on the decreasing trend of the mean drop diameter and the correspondence between the scale-up

factor and mixing time of a production-scale mixer.

Further, many studies have focused on the comparison of different types. of emulsification devices under experimental conditions; however, a theoretical evaluation to address the effect of different configurations of rotor-stator mixers has not been carried out.

# **1.2 Objectives of this study**

The objective of this study is to propose a theoretical evaluation method of a homogenization effect for different stator configurations of internally and externally circulated batch rotor-stator mixers. Another aim of this study is to derive the scale-up factor for the mean drop diameter **in** relation to the mixing time and to confirm the validity of the scale-up procedure proposed in this study.

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# **Chapter 2 Scale-Up Factor for Mean Drop Diameter in Externally Circulated Batch Rotor-Stator Mixers**

# **2.1 Introduction**

Rotor-stator mixers are widely employed in the chemical, food, cosmetic, and pharmaceutical industries to produce emulsions and liquid-liquid dispersions. A typical feature of these mixers is the presence of a high-speed rotor (which drives the mixing process) in close proximity to.a stator (a fixed element with slots). These mixing units generate a high shear rate that ranges from 20,000 to 100,000  $s^{-1}$ (Atiemo-Obeng and Calabrese, 2004). Therefore, these devices are also called high-shear mixers.

Although a rotor-stator mixer is a highly efficient device, its drop size distribution is generally much broader as compared to that of a fine clearance valve homogenizer. This is why rotor-stator mixers are usually used in pre-emulsification processes in actual production. From an engineering viewpoint, obtaining information about the decreasing trend of the mean drop diameter is more important than obtaining that about the maximum stable drop size, which is obtained over a long mixing time, for the design of production lines.

Over the past few decades, several studies have been conducted on rotor-stator mixers. With regard to the maximum stable drop size, Davies (1985, 1987) reported that the drop size decreases with an increase in the energy dissipation rate per unit mass. This phenomenon

has been confirmed in different types of emulsification devices (static mixers, turbine impellers, colloid mills, and fine valve homogenizers). Davies (1985, 1987) also indicated that a turbulent fluctuation velocity is responsible for drop breakup, and not the shear rate.

Calabrese *et al.* (2000, 2002) have reported further details of the drop breakup mechanism in a rotor-stator mixer. From their investigations, it was found that a shear gap between the rotor and the stator was not a major factor responsible for the drop breakup. It is more likely that breakup occurs in the stator slots or in the jet emanation from the stator slot. A narrow gap in the stator slot, however, contributes to a high level of turbulent kinetic energy in it. From this fact, they deduced that this narrow gap is necessary for efficient dispersion. Maa and Hsu (1996) and Barailler *et al.* (2006) reported the importance of shear gap.

A study on the energy dissipation rate of a rotor-stator mixer was conducted by Utomo *et al.* (2008). From their results of complete three-dimensional computational fluid dynamics simulations, it was found that the highest energy dissipation rate occurred around 'the leading and trailing edges of the stator slots. However, the energy dissipated in the slot region is  $7.6\%$  of the total energy supplied by the rotor. They also reported that the high energy dissipation rate could be found on the stator wall. This result suggests that the gap between the rotor and the stator cannot be disregarded in the processes of dispersion and emulsification.

Utomo *et al.* (2008) also indicated that a scale-up procedure should be based on the constant energy dissipation rate per unit mass

and geometrical similarities, rather than the constant tip speed and constant gap width, which have been proposed by other researchers (Bourne and Studer, 1992; Atiemo-Obeng and Calabrese, 2004). In addition to the above factors, another scale-up factor termed shear number derived from the shear frequency and shear rate has been proposed by Porcelli (2002). This proposal considered the number of rotor blades and stator openings; however, the associated scale-up accuracy and coverage were not known.

Thus far, studies have focused on the maximum stable drop size that is obtained over a long mixing time, however, studies on the decreasing trend of the mean drop diameter and the scale-up index correspondence with mixing time have still not been conducted.

In this chapter, we derive the scale-up index in relation to the mixing time. This index can also explain the mean drop diameter for different configurations of rotor-stator mixers as well as different operating conditions and production scales.

## **2.2 Theory**

#### **2.2.1 Power consumption for homogenization**

In order to intensify a homogenization effect, power consumption must be increased. This then requires that we consider the structure of power consumption and determine the power consumption which contributed to homogenization. The net power consumption  $P_n$  [W] is calculated by Eq. (2-1).

$$
P_{\rm n} = \eta (P_{\rm g} - P_{\rm a}) \tag{2-1}
$$

Here,  $\eta$  is the motor efficiency [-],  $P_g$  is the gross electrical power [W], and  $P_a$  is the power loss [W] as determined by running the mixer in air (Kowalski, 2009). Although  $\eta$  is normally a function of different motor power and rotor speeds, we assumed that the motor power and rotor speed are relatively high and that  $\eta$  is fairly constant (in this study,  $\eta$  is 1.0).  $P_{\rm g}$  and  $P_{\rm a}$  are measured values. Power consumption is usually measured by strain gauges or torque transducers. In this study, we determined motor power consumption through direct measurement of the electrical current. We understand that this method is not generally recommended, particularly on a small scale, as losses tend to be greater than the power delivered to the fluid (Brown *et* al., 2004). However, for a mixer in large-scale production, it is difficult to attach strain gauges and torque transducers to the mixer driving shaft. Thus, even as the method introduces a certain margin of error, using the measurements of electrical current enables us to practically estimate the power consumption of actual mixers.

On the other hand,  $P_n$  can also be described as follows with Eq.  $(2-2).$ 

$$
P_{\rm n} = Q \cdot H_{\rm t}
$$
  
=  $Q \cdot (H_{\rm k} + H_{\rm pr} + H_{\rm pt})$  (2-2)

The total energy loss  $H_t$  [kg/m/s<sup>2</sup>] is correlated with three factors: the kinetic energy loss  $H_k$  [kg/m/s<sup>2</sup>], the pressure energy loss  $H_{\text{pr}}$  [Pa] (= pressure), and the potential energy loss  $H_{\text{pt}}$  [kg/m/s<sup>2</sup>] (= 0).

The flow rate is represented by  $Q \text{ [m}^3/\text{s}]$ . For all others, the kinetic energy loss  $H_k$  is separated into two losses shown by Eq. (2-3).

$$
H_{\mathbf{k}} = H_{\mathbf{k}, \text{avg}} + H_{\mathbf{k}, \text{fluc}} \tag{2-3}
$$

Here,  $H_{k,avg}$  is the average velocity loss [kg/m/s<sup>2</sup>] and  $H_{k,flux}$  is the fluctuation velocity loss  $[kg/m/s^2]$ caused by turbulence (Yamaguchi, 1984). From Eqs. (2-2) and (2-3), *Pnis* given by Eq. (2-4).

$$
P_{\rm n} = Q \cdot (H_{\rm k, ave} + H_{\rm k, fluc} + H_{\rm pr}) \tag{2-4}
$$

In these energy losses, it is surmised that  $H_{k,fluc}$  and  $H_{pr}$  are the main factors contributing to homogenization, and the power consumption which contributed to homogenization  $P_h$  [W] is defined by Eq. (2-5).

$$
P_{\rm h} = Q \cdot (H_{\rm k,fluc} + H_{\rm pr}) \tag{2-5}
$$

On the other hand, the pumping power consumption  $P_p$  [W] is also defined by the conservation of angular momentum as Eq. (2-6).

$$
P_{\rm p} = Q \cdot H_{\rm k, ave}
$$
  
=  $2\pi N \rho Q \left(\frac{D}{2}\right)^2 \varpi$   
=  $\rho Q (\pi D N)^2$  (2-6)

Here, *N* is the rotational speed [1/s],  $\rho$  is the density [kg/m<sup>3</sup>], *D* is the rotor diameter [m], and  $\omega$  is the angular velocity [rad/s]. The purpose is to know the  $P_{\text{h}}$ , but both energy losses in Eq. (2-5) are difficult to measure directly. Therefore,  $P_h$  is estimated by subtracting  $P_p$  from  $P_n$ as shown in Eq.  $(2-7)$ .

$$
P_{\rm h} = P_{\rm n} - P_{\rm p}
$$
  
=  $\eta (P_{\rm g} - P_{\rm a}) - \rho Q (\pi D N)^2$  (2-7)

All variables in Eq. (2-7) can be derived from experiments.

#### **2.2.2 Homogenization volume and circulation number**

According to Utomo *et al.* (2008), the highest energy dissipation rate occurs around the leading and trailing edges of the stator slot due to the stagnation of fluid on these edges. A high energy dissipation rate was also predicted on the stator wall. From these results, the volume that contributed to homogenization  $v<sub>h</sub>$  [m<sup>3</sup>] is defined as the entire slot volume on the stator  $v_s$  [m<sup>3</sup>] and the gap in the volume between the rotor and the stator  $v_{\rm g}$  [m<sup>3</sup>], which was calculated by subtracting the swept-out volume of the rotor diameter from the swept-out volume of the stator diameter.

$$
v_{\rm h} = v_{\rm g} + v_{\rm s} \tag{2-8}
$$

The circulation number  $N_c$  [-] is defined as follows by Eq. (2-9).

$$
N_{\rm c} = \frac{Q}{V} \cdot t_{\rm m} \tag{2-9}
$$

Here, *V* is the total product volume  $[m^3]$ , and  $t_m$  is the mixing time [s]. N*<sup>c</sup>* signifies how many times the product passed through the homogenization unit during the time it was operating.

#### 2.2.3 Homogenization index

In past experimental results, it is difficult to explain the mean drop diameter by N*c,* except for at the same rotational speed condition. This means it is required to evaluate the homogenization intensity for a single pass through a batch-mixing unit. Davies (1985, 1987) reported that the drop size decreases with an increase in the energy dissipation rate per unit mass. Sumi and Kamiwano (2000) indicated that the power consumption per unit volume and the circulation time affect the drop breakup. From these facts, it is supposed that main factors affecting homogenization are energy dissipation rate per unit mass and *N<sub>c</sub>*. Based on this hypothesis, the homogenization index *H.I.*  $[m^{2}/s^{3}]$  (=[W/kg]) is defined by Eq. (2-10).

$$
H.I. = \left(\frac{P_{h}}{\rho \cdot v_{h}}\right) \cdot \left(N_{c}\right)
$$

$$
= \varepsilon_{1} \cdot N_{c} \qquad (2-10)
$$

Here,  $\varepsilon_1$  is the local energy dissipation rate of turbulence  $[m^2/s^3]$  (the

energy dissipation rate per unit mass of the homogenization region). The  $\varepsilon_1$  signifies how much power the product received in the homogenization region. In other words, *H.I.* was obtained from the circulation number weighted with the local energy dissipation rate of turbulence.

## 2.3 Experimental

#### 2.3.1 Experimental apparatus

The mixer head used for this study is shown in Figure 2-1. Two sizes of rotor-stator mixers were employed. In order to compare the effect of different rotor-stator configurations, we used two rotors with two different gap widths in the small mixer (Magic Lab, lKA Works Inc.). The diameter of the standard mixer was 30.1 mm and the gap width was 0.15 mm. In the wide gap mixer, the diameter and the gap width were 29.9 mm and 0.25 mm, respectively. Both the standard and the wide gap mixers were used with the same diameter stator (30.4 mm). The diameter and the gap width of the large mixer (MP-10, IKA) Works Inc.) were 57 mm and 0.25 mm, respectively. More details on the mixer configuration are shown in Table 2-1.

Figure 2-2 shows the apparatus of the batch rotor-stator mixing unit. Small and large scales of rotor-stator mixers were installed at the bottom of a vessel and the throughput from the mixer was circulated to the vessel. Total volume of each vessel were 1.5 L and 9 L, respectively.

#### 2.3.2 Materials

In order to simulate the emulsion of food products, milk protein concentration powder (MPC-80, DMV International bv.) and rapeseed oil (Cocolin rapeseed oil, Taiyo Yushi Corp.) were used for the model product. Their composition and properties are presented in Table 2-2. The composition ratios of protein to water, oil to protein, and oil to water were similar to those found in typical dairy products.

#### 2.3.3 Preparation and Evaluation Methods

The preparation procedure was as follows. First, hot water  $(40-50\degree C)$  and rapeseed oil  $(15-25\degree C)$  were added to a mixing pail (10 L). Then, a vertical mixer (IKA-ROTOTRON, IKA Works Inc.) was turned on in order to obtain a rough dispersion of the oil and water. After pre-mixing, the milk protein powder was added to the mixing pail, and was mixed for ten min. After this preparation, the model product was moved to the batch mixing unit. The decreasing trend of the mean drop diameter was evaluated in drop size measurement increments (mean drop diameter:  $d_{50}$ [m]) using a laser diffraction particle size analyzer (SALD-2000, Shimadzu Corp.).

The time at which the rotor reached full speed was considered the start of the mixing time. Samples were taken at certain time intervals for measurement of the mean drop diameter.

## 2.4 Results and Discussion

#### 2.4.1 Mean drop diameter

All experimental conditions and calculated values are summarized in Table 2-3. Discussion of the accuracy of the scale-up

factor is based on  $H.I.$  shown Table 2-3 and the results of drop size measurement.

**Figure 2-3** shows the mean drop diameter under different operating conditions and with rotors of different diameters in the small rotor-stator mixer. The drop size decrease much faster at a high rotor speed, narrow gap width, and long mixing time.

With regard to the effect of the gap width, Calabrese *et al.* (2000, 2002) indicate that the shear gap between the rotor and the stator is not a major factor that is responsible for the drop breakup. However, in the model product and the mixer, it was confirmed that gap width strongly affects the mean drop diameter.

**Figure 2-4** shows the relationship between the circulation number and the drop size under different operating conditions and with rotors of different diameters, as obtained by Magic Lab. All data are the same as those shown in Figure 2-3. From this figure, it can be inferred that the circulation number is not necessarily responsible for the mean drop diameter under different rotational speed conditions or different gap widths. This result also indicates that it is necessary to evaluate the homogenization intensity for a single pass through a mixer unit.

#### **2.4.2 Influence of rotor speed**

In order to confirm the validity of the scale-up index using  $H.I.$ comparisons of the mean drop diameter obtained under different operating conditions were carried out.

**Figure 2-5** shows the mean drop diameter, which is ordered by
the H.I., under different rotor speed conditions. It is found that the tendency varies, even as changes in drop size show a similar slope under different operating conditions. This result suggests that *H.I.* can be used to describe the mean drop diameter in terms of the influence of the rotor speed.

#### 2.4.3 Influence of the gap

Figure 2-6 shows the mean drop diameter, which is arranged by the H.I., for different gap widths. By comparing Figures 2-6, 2-3, and 2-4, it is found that, although it is difficult to correlate the mean drop diameter with the mixing time and the circulation number, *H.I.* can account suitably for the mean drop diameter, considering the influence ofthe gap. This result also suggests that *H.I* can be used to predict the effect of different rotor-stator configurations.

#### 2.4.4 Influence of the mixer size and batch volume

Figure 2-7 shows the mean drop diameter, which is ordered by the *HI,* for different-sized mixers. As shown in Figures 2-5 and 2-6, the mean drop diameter is in good agreement with both the mixer sizes and the different rotor speed settings.

The ratio of the rotor diameter between Magic Lab (29.9-30.1 mm) and MP-10 (57 mm) is approximately 1.9. Further, the volume ratio of Magic Lab (1.5 L) to MP-10 (9 L) is 6. We can explain the mean drop diameter using *HI.* by considering the influence of the mixer size and the batch volume. However, the large mixer produced a wider drop size distribution. As shown in Figure 2-8, the standard

deviation of the production scale mixer was much higher ( $\sigma = 0.188$ )  $\mu$  m) compared with the standard deviation of the pilot scale ( $\sigma$  =  $0.128 \mu$  m) although the mean drop diameter of the production scale mixer was smaller than the pilot scale mixer (Production scale mixer  $d_{50}$  = 0.499  $\mu$  m, Pilot scale mixer  $d_{50}$  = 0.578  $\mu$  m).

We then plotted all results onto the same graph, which are shown in **Figure 2-9.** These results clearly indicate that *HI* is a good index for estimating the mean drop diameter for different product volumes, different rotor speeds, and different mixer configurations (diameter and gap). In other words, *HI.* can be applied to a wide range of operating conditions. This implies *H.I.* can be used to evaluate the homogenization intensity for a single pass through a mixing unit, adequately using the local energy dissipation rate. From the result, it can be inferred that the scale-up procedure should be based on *H.I.,* which is calculated from the local energy dissipation rate and circulation number.

In the production scale, it is difficult to satisfy the same conditions as in the laboratory scale, such as constant dissipation rate per unit mass, geometrical similarities, constant tip speed, and constant gap width. The results suggest that these constant conditions and geometrical similarities are not necessary for achieving successful scale-up for mean drop diameter in the pre-homogenization process carried out in the mixers for the model product. In Figure 2-8, the data correspond well with Eq. (2-11).

$$
d_{50} \propto H.I.^{-0.328} \tag{2-11}
$$

The coefficient of variation is  $R = 0.926$ . If the stress on the drop is inertial, as shown from the results of Calabrese *et al. (2002),* the correlation of  $d_{32}$  and  $\varepsilon$  in low viscosity liquid is given by Eq. (2-12).

$$
d_{32} \; \propto \; \sigma^{1/3} \rho^{-2/3} \varepsilon^{-1/3} \mu^{1/3} \tag{2-12}
$$

Here,  $d_{32}$  is the Sautar mean diameter [m],  $\mu$  is the viscosity [Pa · s],  $\varepsilon$ is energy dissipation rate  $[m^2/s^3]$  and  $\sigma$  is the surface tension [N/m]. The fitted coefficient of  $\varepsilon_1$  in the study is the same coefficient of  $\varepsilon$  that was reported by Calabrese *et al.* (2000). If we were to assume that  $d_{32}$ and  $\varepsilon$  have a proportional relationship with  $d_{50}$  and  $\varepsilon_1$  respectively, the result could be explained by the theory of Calabrese et al. (2000). It is assumed the mechanism' for the breakup of drops is based on the interaction of inertial sub-range eddies, which could take place either on a similar scale or on the smaller Kolmogorov microscale.

The Power consumption that was calculated from the electrical current, however, contains some measurement errors; therefore, it might not be applicable to all types of rotor-stator mixer. Further studies to confirm the coverage of scale-up using  $H.I$ . are required.

#### **2.5 Conclusions**

In this chapter, a scale-up factor (homogenization index:  $H.I.$ ) was proposed for the mean drop diameter in batch rotor-stator mixers. This index is based on the concept that the circulation number and local energy dissipation rate of turbulence could well account for the mean drop diameter at different rotor speeds, with different mixer configurations (gap width and rotor diameter), and for different volumes of production. In other words, mean drop diameter could be estimated by using *H.I.* This result also suggested that *H.I.* accurately corresponds with the homogenization intensity for a single pass through a batch-mixing unit. The scale-up criteria for the mean drop diameter in terms of mixing time should be based on *H.I,* and not necessarily on geometrical similarities, same rotor tip speed, or the same gap width in the case of similar mixer configurations and the experimental range (1 to 10 L) used for the model product.

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Fig. 2-1 Schematic representation of the rotor and the stator



Fig. 2-2 Flow diagram of the batch mixing unit







Fig. 2-4 Profiles of the circulation number vs. the mean drop diameter by Magic Lab



**Fig. 2-5** Profiles of the homogenization index vs. the mean drop diameter in different rotor speed by Magic Lab











the mean drop diameter in all experimental data



# Table 2-1 General information of mixing unit



# Table 2-2 Composition and properties of the model product

ر اسمع



## **Table 3** Experimental conditions and calculated values

::r' 5  $\mathcal{\mathcal{P}}$ ٤ o <u>ة</u> 'ji  $\bar{z}$ S x· (ll

## **Chapter 3 Evaluating** Method **of Homogenization Effect for Different Stator Configurations of Internally Circulated Batch Rotor-Stator Mixers**

### **3.1 Introduction**

High-speed rotor-stator mixers have been broadly used in process industries as a process intensification tool to accelerate mixing, dissolution, homogenization, dispersion, and emulsification. In a typical configuration, these mixers consist of a high-speed rotor (that drives the mixing process) in close proximity to a stator (a fixed element with slots). These mixing units generate a high shear rate that ranges from 20,000 to 100,000  $s^{-1}$  (Atiemo-Obeng and Calabrese, 2004). Therefore, these devices are also called high-shear mixers.

Many manufacturers have developed and provided a variety of rotor-stator mixers that have, for the most part, a different design. However, methods of comparing emulsification and homogenization from different rotor-stator configurations have not been developed. Therefore, a quantitative evaluation of the homogenization performance with different types of rotor-stator mixers has been difficult thus far. In order to predict the mean drop diameter on a production-scale, a method to evaluate the homogenization effect from different rotor-stator configurations is required.

Over the last few decades, many studies have focused on the homogenization or emulsification process with different types of high-shear devices. Bourne and Garcia-Rosas (1986) studied the micro-mixing performance of different rotor-stator configurations in

the same mixing device (six types of rotors and two types of stators). They concluded that there is an optimum combination of rotor-stator set; however, the stators had a negligible effect on both the predicted energy dissipation and the measured product distributions in their mixing system.

Davies (1987) reported on the maximum stable drop size for different types of emulsification devices (static mixers, turbine impellers, colloid mills, and fine-valve homogenizers). He concluded that the drop size decreases with an increase in the energy dissipation rate per unit mass. Maa and Hsu (1996) compared the equilibrium drop size between different sizes and configurations of rotor-stator sets (four-blade rotors with 12 rectangular slots and two-blade rotors with 8 slant slots). They also compared the trend in drop size reduction of the different sets; however, they did not discuss the effects of the differences in mixer configurations theoretically.

Calabrese *et al.* (2000, 2002) reported the effect of different clearance gaps between the rotor and the stator (standard clearance: 0.5 mm, wide clearance: 1.0 mm). They found that the drop size for wide clearance geometry is smaller than that for standard clearance geometry. While the reason for this is not apparent, they concluded that the mechanical shear force created in the shear clearance does not control the ultimate drop size.

With regard to the theoretical effect of different mixer configurations, Porcelli (2002) reported an evaluation index called the shear number that was derived from the shear frequency and shear rate. Porcelli (2002) considered the number of rotor blades and stator

openings; however, the scale-up accuracy and the scope of their theories were unknown.

Urban (2006) reported the drop size for different types of emulsification devices (rotor-stator system, disc system, and high-pressure system). Although he compared the mean drop diameter experimentally, he did not discuss the theoretical effects of differences in mixer configurations.

Kamiya *et al.* (2009) proposed a homogenization index to scale-up and estimated the mean drop diameter in relation to the mixing time for externally circulated batch rotor-stator mixers. However, this index is not applicable to various mixer designs and stator configurations.

As noted above, many studies have focused on the comparison of different types of emulsification devices under experimental conditions; however, a theoretical evaluation to address the effect of different configurations of rotor-stator mixers has not been carried out.

In this chapter, a theoretical evaluation method of the homogenization effect from different stator configurations of internally circulated batch rotor-stator mixers is proposed. This evaluation method allows a comparison' and assessment of the homogenization effect for different configurations of rotor-stator mixers by water mixing operations without actual product operation.

## 3.2 Theory

3.2.1 Calculation methods of power consumption, average shear stress, and average force

Kamiya *et al.* (2009) defined the power consumption that contributed to a homogenization as  $P_h$  [W]. They assumed that the homogenization effect was related to *Ph. Ph* is calculated by subtracting the net power consumption  $P_n$  [W] from the pump power consumption  $P_p$  [W] as follows:

$$
P_{\rm n} = N_{\rm p} \rho N^3 D^5 \tag{3-1}
$$

$$
P_{\rm p} = \rho \pi^2 N^2 D^2 Q
$$

$$
= N_{\rm qd} \pi^2 \rho N^3 D^5 \qquad (3-2)
$$

$$
P_{\rm h} = P_{\rm n} - P_{\rm p}
$$
  
=  $(N_{\rm p} - \pi^2 N_{\rm qd}) \rho N^3 D^5$  (3-3)

where N is the rotational speed [1/s];  $N_{\rm p}$ , the power number [-];  $N_{\rm qd}$ , the flow number [-];  $\rho$ , the density [kg/m<sup>3</sup>]; Q, the flow rate [m<sup>3</sup>/s]; and D, the rotor diameter [m]. All variables in Eq.  $(3-3)$  can be derived experimentally.

According to Tatterson (2002), the power consumption was calculated by multiplying the flow rate (impeller pumping) by the shear stress (head). From this relationship, the average shear stress at a stator hole  $\tau_{\rm a}\,[\mathrm{N/m}^2]$  is calculated by Eqs. (3-1)– (3-3) as follows:

Chapter 3 Evaluation of different stator configurations with internally circulated mixers

$$
\tau_{a} = \frac{P_{h}}{Q}
$$
  
=  $\rho N^{2} D^{2} \left( \frac{N_{p}}{N_{qd}} - \pi^{2} \right)$  (3-4)

**Figure 3-1** shows the schematic diagram of the rotor-stator mixing unit and the details of a stator hole.

According to Utomo *et al.* (2008), the energy dissipation rate in the rotor swept volume was low. The energy dissipation rate around the leading and trailing edges of the stator holes was higher than that in the other parts due to stagnation of fluid on these edges.

From these facts, it is assumed that the area subjected to the shear stress  $S_s$  [m<sup>2</sup>] is the sum of the area of the side wall  $S_l$  [m<sup>2</sup>] and the area of a stator hole cross section  $S_d$  [m<sup>2</sup>].

$$
S_{s} = S_{d} + S_{1}
$$
  
=  $(\pi dl + \frac{\pi}{4}d^{2})$  (3-5)

Here,  $d$  is the stator hole diameter [m] and *l*, the thickness of the stator wall [m].

Therefore, the average force at a stator hole  $F_a$  [N] is calculated by multiplying the shear stress (Eq. (3-4)) by the area subjected to the shear stress  $(Eq. (3-5))$  as follows:

$$
F_{a} = \tau_{a} \cdot S_{s}
$$
  
=  $\rho N^{2} D^{2} \left( \frac{N_{p}}{N_{qd}} - \pi^{2} \right) (\pi d l + \frac{\pi}{4} d^{2})$  (3-6)

## 3.2.2 Calculation methods of total energy dissipation rate and homogenization coefficient

Davies (1987) reported that the drop size decreases with an increase in the energy dissipation rate per unit mass. Utomo *et al.* (2008) also suggested that the energy dissipation rate is the highest when the blade overlaps with the leading edges. It is considered that the energy dissipation rate to be one of the key factors for homogenization. And it is also considered that the energy dissipation rate is the homogenization intensity in a single pass through a stator hole. From the result of the dimension analysis to lead an energy dissipation rate, the energy dissipation rate at a local volume  $\varepsilon_1$  [m<sup>2</sup>/s<sup>3</sup>] is calculated by the average force (Eq.(3-6)), rotor tip velocity  $U$  [m/s], density  $\rho$ , and volume of stator hole  $v_s$  as follows:

$$
\varepsilon_{\rm l} = \frac{F_{\rm a} U}{\rho v_{\rm s}}
$$

$$
= \frac{\pi^4 dN^3 D^3}{v_s} \left( l + \frac{d}{4} \left( \frac{N_p}{N_{qd} \pi^2} - 1 \right) \right)
$$
(3-7)

As the other key factor for homogenization with rotor-stator mixers,

Porcelli (2002) defined the shear frequency  $f_s$  [1/s]. This factor expresses the frequency of overlaps with a rotor blade and a stator hole as follows:

$$
f_{\rm s} = n_{\rm r} n_{\rm s} N \tag{3-8}
$$

where  $n_r$  is the number of rotor blades  $[-]$  and  $n_s$ , the number of stator holes [-]. When the opening ratio of the stator is defined as *A* [-], *n;* can be expressed as follows:

$$
n_{s} = A \frac{4(D+2\delta)h}{d^{2}}
$$
 (3-9)

where  $\delta$  is the clearance between the rotor and the stator [m] and h, the height of the stator  $[m]$ .

In this study, Porcelli's shear frequency theory for a unit hole was modified. The number of holes  $n_v$  in the total volume  $V$  [-] is calculated as follows.

$$
n_{\rm v} = \frac{V}{v_{\rm s}} \tag{3-10}
$$

In order to estimate the number of times the product receives the shear stress at a hole, the shear frequency for a unit hole  $f_{s,h}$  [1/s] is calculated by dividing  $f_s$  by  $n_v$ .

#### Chapter 3 Evaluation of different stator configurations with internally circulated mixers

$$
f_{s,h} = \frac{n_s n_r N}{n_v}
$$
  
=  $A \frac{4(D+2\delta)h v_s}{d^2} n_r N$  (3-11)

Davies (1987) experimentally indicated that the mean drop diameter depends on the energy dissipation rate per unit mass. Utomo *et al.* (2008) numerically suggested that the energy dissipation rate around the leading and trailing edges of the stator holes was higher than that at the other parts. Porcelli (2002) theoretically reported the importance of the shear frequency for the mean drop diameter in rotor-stator mixers. From these facts, it is assumed that the main factor for homogenization is the total energy dissipation rate  $\varepsilon_t$  [m<sup>2</sup>/s<sup>3</sup>], which contains information about the local energy dissipation rate  $\varepsilon_{\rm b}$ , shear frequency of a unit hole  $f_{\rm sh}$ , and mixing time  $t_{\rm m}$  [s]  $t_{\rm m}$  is added to the equation used in the dimension analysis.  $\varepsilon_t$  is expressed as follows:

$$
\varepsilon_{\rm t} = \varepsilon_{\rm l} f_{\rm s,h} t_{\rm m}
$$

$$
= \left[ \frac{\pi^4 dN^3 D^3}{v_s} \left( l + \frac{d}{4} \right) \left( \frac{N_p}{N_{qd} \pi^2} - 1 \right) \right] \left( A \frac{4(D + 2\delta) h}{d^2} \frac{v_s}{V} n_r N \right) t_m
$$
  

$$
= \left[ A \pi^4 n_r (D + 2\delta) D^3 h \left( \frac{4l}{d} + 1 \right) \left( \frac{N_p}{N_{qd} \pi^2} - 1 \right) \right] \left( \frac{N^4}{V} t_m \right)
$$

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$$
=C_{\rm h}\frac{N^4}{V}t_{\rm m}\tag{3-12}
$$

$$
C_{\rm h} = A\pi^4 n_{\rm r} (D + 2\delta) D^3 h \left( \frac{4l}{d} + 1 \right) \left( \frac{N_{\rm p}}{N_{\rm qd} \pi^2} - 1 \right)
$$
 (3-13)

where  $C_h$  is the homogenization coefficient  $[m^5]$ . As this coefficient depends on the rotor-stator configurations, it is possible to evaluate the stator performance from  $C_h$ .

#### 3.3 Experimental

#### 3.3.1 Experimental apparatus

The rotor-stator mixer investigated in this study is Turbo Mixer (SPM-100V, Tetra Pak Scanima  $A/S$ ). The mixer head is shown in Figure 3-2.

The stator, which is installed at the bottom of a tank, is a dynamic one. It can be moved up and down while being operated. In the up state, the stator is in the circulation mode, and in the down state, it is in the homogenization mode.

In order to compare the effect of different rotor-stator configurations, 5 different configuration stators for the internally circulated batch rotor-stator mixer were used. Details about the mixer specifications and configurations are listed in Table 3-1. Rotor-stator mixers generally have external and internal circulation systems. The schematic diagrams of both circulation systems are shown in Figure 3-3. Over the last few decades, batch mixing systems that employ external circulation have been extensively used in various fields. More

recently, in the chemical, food, and pharmaceutical fields, users have preferred an internally circulated batch mixing system because it is possible to build a less complex system and the system exhibits good performance on a large production scale. Therefore, this study mainly focused on an internal circulation system; however, it is difficult to directly measure the circulated flow rate in an internal circulation system. Thus, the flow rate of the external circulation system with a similar mixing head as the internal circulation system is used.

#### 3.3.2 Materials

In order to simulate the emulsion of food products, milk protein concentration powder (MPC-80, DMV International bv.) and rapeseed oil (Cocolin rapeseed oil, Taiyo Yushi Corp.) were used for the model product. Their composition and properties are listed in Table 2-2. The composition ratios of protein to water, oil to protein, and oil to water were similar to those found in typical dairy products.

#### 3.3.3 Preparation and evaluation methods

The preparation procedure was as follows. First, hot water  $(40-50^{\circ}C)$  and rapeseed oil  $(15-25^{\circ}C)$  were added to the mixing tank. Then, the mixer was turned on at low rotational speed and the dynamic stator was set to the up state (circulation mode) in order to obtain a rough dispersion of oil and water. After premixing, the vacuum pump was turned on and the milk protein powder was vacuum-sucked into the mixing tank. Finally, the dynamic stator was set to the down state (homogenization mode) and the rotational speed was increased to the setting value. The mean drop diameter was evaluated in drop size measurement increments (mean drop diameter d*so*[m]) using a laser diffraction particle size analyzer (SALD-2000, Shimadzu Corp.). The time at which the rotor speed reached the setting value was considered as the start of the mixing time. Samples were taken at certain time intervals to measure the mean drop diameter.

#### 3.4 Results and Discussion

All measured and calculated values for the operation with the model product are summarized in Table 3-2. The discussion of the adequacy of the mixer performance estimation is based on  $C<sub>h</sub>$  shown in Table 3-2 and the results of the drop size measurement.

#### 3.4.1 Mean drop diameter for different stators

Figure 3-4 shows the mean drop diameters for different stators under the same rotational speed conditions for the model product. Homogenization effects of stators No. 3 and No. 4 are more or less equivalent, and stator No.5 is confirmed to be the most efficient stator (narrow clearance and smaller hole diameter) in the experiment. Table 3-2 lists the stators in order of the homogenization effect based on  $C_{h}$ .

It is clear from Fig. 3-4 that the mean drop diameter of the model product decreases with  $C<sub>h</sub>$ . From this result, it is considered that  $C<sub>h</sub>$  could be used to predict the mean drop diameter in relation to the different stator configurations.

#### 3.4.2 Influence of stator hole diameter

In order to explain the effect of the stator hole diameter, the influence of each hole diameter was examined.

Figure 3-5 shows the relationship between the stator hole diameter and  $C_h/C_{h, std}$ , which is obtained by dividing each stator  $C_h$ from the standard stator (No. 4)  $C_{h, std}$  under the same operating conditions.

All the plotted trials were carried out using an externally circulated batch mixer that has four different hole diameter stators (d *=* 1, 2, 4, 6 mm) under the same opening ratio  $(A = 0.24)$  and clearance  $(\delta = 1$  mm). In the case of rotor-stator mixers of this study,  $C_h/C_{h, std}$ increases when the stator hole diameter is small. For example,  $C_h/C_h$ , std for 2 mm hole diameter is 1.75 times that for 4 mm hole diameter. This suggests that the stator hole diameter strongly affects the homogenization process. From Eq. (3-9), a smaller stator hole diameter implies a larger number of stator holes (n*s)* for the same opening ratio.

In order to confirm the adequacy of quantity estimation by  $C<sub>h</sub>$ , the measured and estimated values of the mean drop diameter for the model product were compared, as shown in Figure 3-6. The verification experiment using the model product was carried out using an internally circulated mixer that has two different hole diameter stators  $(d = 1$  and 4 mm).

The estimated mixing time of stator No. 5, which has the same mean drop diameter as stator No.4, was calculated by dividing the mixing time of stator No. 4 by  $C_h/C_{h, std}$  of stator No. 5. The estimated decreasing trend of the mean drop diameter of stator No. 5 was the same as that obtained experimentally. This confirms that the quantity estimation using  $C<sub>h</sub>$  for the homogenization effect has good adequacy.

### 3.4.3 Influence of stator opening ratio

In order to demonstrate the effect of the stator opening area, the influence of the different opening ratio is examined.

Figure 3-7 shows the relationship between the stator opening ratio and  $C_h/C_{h, std}$  for the same stator hole diameter and rotor-stator clearance. All the. plotted trials were carried out using an externally circulated batch mixer that has three different opening ratio stators (A  $= 0.12, 0.24, 0.36$  under the same hole diameter ( $d = 4$  mm) and clearance ( $\delta = 1$  mm).  $C_h/C_{h, std}$  is much higher when the stator opening ratio is larger. For example, in the case of an opening of 0.36, the opening ratio is 1.5; however,  $C_h/C_{h, std}$  increases to 1.74 for an opening of 0.24. This suggests that the stator opening ratio strongly influences the stator performance. With respect to the hole diameter, a large opening ratio also implies a larger number of stator holes  $(n_s)$ , by Eq.  $(3-9)$ . From these results, it is clear that the number of stator holes is an important factor for assessing the stator performance. In order to confirm the adequacy of the quantity estimation by  $C<sub>h</sub>$ , the decreasing trend in the mean drop diameter for the measured and estimated values were compared. The verification experiment using the model product was carried out using an internally circulated mixer that has three different opening stators  $(A = 0.11, 0.20, \text{ and } 0.31)$ .

Figure 3-8 shows the estimated decreasing trend of the mean drop diameters of stators No. 1 and No. 3, which were obtained by the experimental results of stator No.2. The estimated mixing time of each stator, which has the same mean drop diameter as stator No.2, was calculated by dividing the mixing time of stator No. 2 by  $C_h/C_{h, std}$ values of stators No. 1 and No.3. It was found that the performance estimation using  $C_h$  has a high degree of adequacy.

### 3.4.4 Influence of clearance between rotor and stator

In order to demonstrate the relationship between the rotor-stator clearance and  $C_h/C_{h, std}$ , the same comparison was carried out. In the case of 1 and 2 mm rotor-stator clearances,  $C_h/C_{h, std}$  was slightly large (1.07 times) when the rotor-stator clearance was reduced by half (2 mm to 1 mm). This suggests that the clearance influences the stator performance; however, the effect is lesser than that of the hole diameter and the stator opening ratio.

With regard to the effect of the clearance, Calabrese *et al.* (2000, 2002) and Utomo *et al.* (2008) indicated that the shear clearance between the rotor and the stator is not a major factor responsible for drop breakup. It is more likely that breakup occurs in the stator slots or in the jet emanation from the stator slot. A narrow clearance in the stator slot, however, contributes to a high level of turbulent kinetic energy. Therefore, they deduced that this narrow clearance is necessary for efficient dispersion. Results of this study could be similarly explained.

#### 3.4.5 Total energy dissipation rate and mean drop diameter

Figure 3-9 shows the mean drop diameter as a function of the total energy dissipation rate  $\varepsilon_t$ . All data are the same as those shown in Fig. 3-4. The mean drop diameter is similar when  $\varepsilon_t$  is the same regardless of the stator configuration and mixing time. As indicated in Eq.  $(3-12)$ ,  $\varepsilon_t$  contains information about the mixer configuration, power consumption, flow rate, and mixing time. The mean drop diameter, therefore, can be determined using  $\varepsilon_t$  in relation to the mixing time and stator configuration.

This implies that  $\varepsilon_t$  can be adequately used to evaluate the homogenization intensity for a single pass through a stator hole, suggesting that  $\varepsilon_t$  can be applied to a wide range of different stator configurations for estimating the mean drop diameter.

This information could be useful for determining the optimum mixer design and the adequate mixer selection.

## 3.5 Conclusions

In this chapter, a method of evaluating the homogenization effect for different stator configurations of batch rotor-stator mixers with internal circulation was proposed. The homogenization effect for different stator configurations was evaluated by the homogenization coefficient  $(C_h)$  based on the power number, flow number, and shear frequency derived from the number of rotor blades and stator holes. Using a water mixing operation, it was found that the homogenization effect was much higher in the case of a smaller stator hole diameter and larger number of stator holes. Based on the  $C_h$  evaluations, it was

also found that the clearance between the rotor and the stator had less influence on the homogenization effect than the hole diameter and number of holes. It is verified that the adequacy of the mean drop diameter estimation by  $C_h$  using model product trials. The total energy dissipation rate that was contained by the  $C<sub>h</sub>$  could well account for the mean drop diameter in relation to the mixing time and the stator configuration differences. Results of this study indicate that we can compare and evaluate the homogenization effect for different configurations of rotor-stator mixers using  $C_h$  by a water mixing operation without actual product trials.

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Chapter 3 Evaluation of different stator configurations with internally circulated mixers



Fig. 3-1 Schematic diagram of the mixing unit



Top view of rotor Side view of rotor Top view of stator Fig. 3-2 Photographs of the rotor and the stator



**Fig. 3-3** Flov diagram of external and internal circulation system







Fig. 3-5 Influence of the stator hole diameter evaluated by the ratio of homogenization coefficient (Externally circulated mixer)















Fig. 3-9 Profiles of total energy dissipation rate vs. mean drop diameter for all experimental data



# Table 3-1 Details of mixer specifications and configurations

Rotor diameter *D* :198 mm Number of rotor blades  $n_r$ : 6 Stator height  $h: 32$  mm





 $\overline{\phantom{a}}$ 

 $\mathcal{A}$ 

#### **Chapter** 4 **Scale-Up Factor for Diameter in Circulated Batch Rotor-Stator Mixers Mean Drop Internally**

#### **4.1 Introduction**

Rotor-stator mixers are used in chemical, biochemical, food-processing, cosmetic, and pharmaceutical industries to produce emulsions and liquid-liquid dispersions. The distinguishing feature of rotor-stator mixers is that a high-speed rotor (a driven mixing element) operates in close proximity to a stator (a fixed mixing element). These mixing units generate a high shear rate that ranges from 20,000 to  $100,000 \text{ s}^{-1}$  (Atiemo-Obeng and Calabrese, 2004). Therefore, these devices are also called high-shear mixers.

Although a rotor-stator mixer is a highly efficient device, its drop-size distribution is generally much broader than that of a fine-clearance valve homogenizer. This is why rotor-stator mixers are usually used in pre-emulsification processes in an actual production line. From an engineering viewpoint, obtaining information about the decreasing trend of the mean drop diameter is more important than obtaining information about the maximum stable drop size, which is obtained over a long mixing time, for the design of production lines.

Over the past few decades, externally circulated batch mixing systems have been extensively used in various fields. However, in recent times, internally circulated batch mixing systems are preferred, especially in the chemical, food, and pharmaceutical industries, because these systems are less complex and they show good

performance on a large production-scale. For these reasons, this study has mainly focused on internally circulated mixing systems.

Over the last several decades, a number of studies on the rotor-stator mixer have been carried out. Davies (1985, 1987) investigated the maximum stable drop size and reported that it decreases with an increase in the energy dissipation rate per unit mass. This phenomenon has been observed in different types of emulsification devices (static mixers, turbine impellers, colloid mills, and fine-valve homogenizers). Davies (1987) also indicated that the turbulent fluctuation velocity and the shear rate are not responsible for drop breakup.

Calabrese *et al.* (2000, 2002) studied the drop breakup mechanism in a rotor-stator mixer in detaiL Their investigations revealed that a shear gap between the rotor and the stator does not play a significant role in drop breakup. Breakup is more likely to occur in the stator slots or during jet emanation from the stator slots. A narrow gap in the stator slots, however, contributes to the turbulent kinetic energy produced therein. From this observation, Calabrese *et al.* (2000, 2002) deduced that this narrow gap is necessary for efficient dispersion.

Utomo *et al.* (2008) conducted a study on the energy dissipation rate of a rotor-stator mixer by performing complete three-dimensional computational fluid dynamics simulations, and they found that the energy dissipation rate was the maximum around the leading and trailing edges of the stator slots. However, the energy dissipated in the slot region was 7.6% of the total energy supplied by

the rotor. They also found that energy dissipation rate on the stator wall was high, suggesting that the gap between the rotor and the stator cannot be disregarded in the processes of dispersion and emulsification.

Utomo *et al.* (2008) also indicated that the scale-up procedure for mixers should be based on the constant energy dissipation rate per unit mass and the geometrical similarities rather than constant tip speed and constant gap width, as proposed by other researchers (Bourne and Studer, 1992; Atiemo-Obeng and Calabrese, 2004).

In addition to the abovementioned scale-up factors, another factor, termed shear number, which is derived from the shear frequency and the shear rate, has been proposed by Porcelli (2002). This proposed scale-up factor takes into account the number of rotor blades and stator openings; however, the adequacy of scale-up and scope of their theories are unknown.

Kamiya et al. (2009a) proposed a homogenization index for scaling up and estimated the mean drop diameter in terms of the mixing time for pilot-scale externally circulated batch rotor-stator mixers. However, this index is not applicable to internally circulated mixers and mixers that are less affected by the clearance between the rotor and the stator.

Thus far, researchers have focused on pilot-scale mixers and the maximum stable drop size that is obtained over a long mixing time. However, studies have not been conducted on the decreasing trend of the mean drop diameter and the correspondence between the scale-up factor and mixing time of a production-scale mixer.

In this chapter, the scale-up factor in terms of the mixing time is derived. This factor can also be used to explain the mean drop diameter in relation to the mixing time for different configurations of rotor-stator mixers as well as under different operating conditions and production scales.

### 4.2 Theory

### 4.2.1 Estimation method of internal circulation flow rate for production-scale mixers

Figure 4-1 shows the estimation method of circulation flow rate for production-scale mixers. In general, it is difficult to estimate the internal circulation flow rate of a conventional mixing tank. However, in the case of the pilot-scale rotor-stator mixer, an inline type of mixing pump (externally circulated mixing pump) is used; the mixing unit of this pump is similar to that of an internally circulated batch rotor-stator mixer. Therefore, the circulation flow rate of the pilot-scale rotor-stator mixer can be estimated using the inline type of mixing pump (Fig. 4-1(a) and (b)).

The diameter of the mixing unit of the production-scale mixer is in the range of 300-400 mm and it were unable to use an inline type of mixing pump, such as that used in the pilot-scale mixer. Thus, it was impossible to estimate the flow rate of the production-scale mixer. Hence, the flow rate of the production-scale mixer was estimated using the time required for complete mixing-termed "completely mixed time", which is given in Eq. (4-1). This equation was proposed by Kamiwano et al. (1967).

$$
\frac{1}{T_{\rm M} \cdot N} = k \left( \left( \frac{D}{D_{\rm t}} \right)^3 \cdot N_{\rm qd} + 0.21 \left( \frac{D}{D_{\rm t}} \right) \cdot \sqrt{\frac{N_{\rm p}}{N_{\rm qd}}} \right) \cdot \left( 1 - \exp(-13 \left( \frac{D}{D_{\rm t}} \right)^2 \right) \tag{4-1}
$$

Here,  $T_M$  [s] denotes the completely mixed time;  $N$  [s<sup>-1</sup>], the rotational speed;  $k$  [-], a constant that depends on the method of the measurement system; D [m], the rotor diameter;  $D_t$  [m], the tank diameter;  $N_{qd}$  [-], the flow number; and  $N_p$  [-], the power number.

Except for  $N_{qd}$ , all values can be obtained from the experiment of the completely mixed time and measurement of the power consumption. The flow rate Q [m<sup>3</sup>/h]: is a function of  $N_{qd}$  (Q =  $N_{\text{qd}}ND^3$ ); thus, the flow rate of the production-scale mixer could be estimated experimentally (Fig. 4-1 (b) and (c)).

In the case of normal agitating vessels, the circulation flow rate  $Q_c$  [m<sup>3</sup>/h] is suitable for the calculation of  $\varepsilon_t$ .  $Q_c$  is the function of  $N_{\rm qc}(Q_{\rm c} = N_{\rm qc}ND^3)$ . But  $N_{\rm qc}$  is difficult to estimate directly. In this study, we assume  $Q_c$  is nearly equal to  $Q$ . Hence, it is considered that  $Q$ could be used for the calculation of  $\varepsilon$ <sub>t</sub>, alternatively.

#### 4.2.2 Calculation method of total energy dissipation rate

Figure 3-1 shows a schematic diagram of the rotor-stator mixing unit. According to Utomo *et al.* (2008), the energy dissipation rate around the leading and trailing edges of the stator holes was of a higher order of magnitude owing to the stagnation of the fluid therein.

Davies (1987) indicated that the mean drop diameter depends on the energy dissipation rate per unit mass. In chapter 3 it was stated that the mean drop diameter can be determined from the total energy dissipation rate  $\varepsilon_t$  [m<sup>2</sup>/s<sup>3</sup>] in terms of the mixing time for pilot-scale batch rotor-stator mixers. From these results, it is assumed that the main factor influencing homogenization is the total energy dissipation rate  $\varepsilon_t$ .  $\varepsilon_t$  was derived as follows.

$$
\varepsilon_{\rm t} = C_{\rm h} \frac{N^4}{V} t_{\rm m} \tag{4-2}
$$

Here,  $t_m$  [s] is the mixing time,  $V[m^3]$  is the total product volume, and  $C_h$  [m<sup>5</sup>] is a homogenization coefficient, which depends on the rotor-stator configuration.  $C_h$  is calculated from Eq. (4-3). More details have been provided by Chapter 3.

$$
C_{\rm h} = A\pi^4 n_{\rm r} (D + 2\delta) D^3 h \left( \frac{4l}{d} + 1 \right) \left( \frac{N_{\rm p}}{N_{\rm qd} \pi^2} - 1 \right)
$$
(4-3)

Here,  $A$  [-] denotes the opening ratio of the stator;  $n_r$ [-], the number of rotor blades;  $\delta[m]$ , the width of the gap between the rotor and stator; h [m], the stator height;  $l$  [m], the thickness of the stator wall; and  $d$  [m], the stator hole diameter.

#### **4.3 Experimental**

**4.3.1 Experimental apparatus of internally circulated batch**

#### rotor-stator mixers

The internally circulated batch rotor-stator mixer investigated in this chapter is the Turbo Mixer; the pilot-scale mixer (SPM-500V) and the production-scale mixer (SPV-10000V) were sourced from Tetra Pak Scanima *AlS.* The mixer head is shown in Figure 3-2. The stator, which was installed at the bottom of the tank, was a dynamic stator: it could be moved up and down during operation. In the "up" position, the stator operated in the circulation mode, and in the "down" position, it operated in the homogenization mode. More detailed information about the mixer specifications and configurations are given in Table 4-1.

#### 4.3.2 Materials for the emulsification experiment

In order to simulate the emulsion of food products, a mixture of milk protein concentrate powder (MPC-80, DMV International) and rapeseed oil (Cocolin rapeseed oil, Taiyo Yushi Corp.) was used as a model product. Their composition and properties are listed in Table 2-2. The composition ratios of protein to water, oil to protein, and oil to water were similar to those found in typical dairy products.

# 4.3.3 Measurement of completely mixed time and estimation procedure of the flow rate

The completely mixed time (the criterion for sufficient mixing) was measured by adding saturated KCL solution until the concentration of the mixture became 0.1% (conductivity: 1.343  $\text{mS/cm}^2$ ) at final consistency.

The saturated KCL solution was quickly poured into the tank from the top. The consistency was measured using an electrical conductivity meter (Smartec S CLD 132, Endress+Hauser), which was installed on a side wall of the tank (Fig. 4-1 (b) and (c)).

The completely mixed time was defined as the deviation from the normalized concentration difference. In this study, the time at which the deviation value of the normalized concentration difference reduced below 0.5% was defined as the completely mixed time.

The estimation procedure of the flow rate is as follows.  $T_M$ ,  $N$ , Nqd, and *N*<sup>p</sup> were measured using the pilot-scale mixer. The only unknown value in Eq.  $(4-1)$ , *k*, was calculated from Eq.  $(4-1)$  for the pilot-scale mixer. Because *k* is a constant that depends on the type of measuring system, it was considered that  $k$  was not influenced by the measurement scale. In this study, it was assumed that  $k$  for the production-scale mixer was the same as that for the pilot-scale mixer. Thus, by measuring  $T_M$ , N, and N<sub>p</sub> for the production-scale mixer and using  $k$  from the pilot-scale measurement,  $N_{qd}$  could be obtained from Eq. (4-1). The flow rate Q is a function of  $N_{qd} (Q = N_{qd} ND^3)$ ; thus, the flow rate of the production-scale mixer could be estimated experimentally.

# **4.3.4 Preparation procedure of emulsification experiment and evaluation of the mean drop diameter**

The preparation procedure was as follows. First, hot water  $(40-50^{\circ}$ C) and rapeseed oil  $(15-25^{\circ}$ C) were added to the mixing tank. Then, the mixer was turned on at a low rotational speed and the

dynamic stator was set to the "up" position (circulation mode) in order to obtain a coarse dispersion of oil and water. After premixing, a vacuum pump was turned on and the milk protein powder was vacuum sucked into the mixing tank. Finally, the dynamic stator was set to the "down" position (homogenization mode), and the rotational speed was increased to a setting value.

The mean drop diameter was evaluated in drop size increments (mean drop diameter  $d_{50}$ ) using a laser diffraction particle size analyzer (SALD-2000, Shimadzu Corp.). The time at which the rotor speed reached the setting value was considered the start time of mixing. Samples were taken at certain time intervals to measure the mean drop diameter.

#### 4.4 Results and Discussion

# 4.4.1 Completely mixed time and circulation flow rate of production-scale mixer

The measured completely mixed time and the estimated circulation flow rates are shown in Table 4-2. The value of *k* for the production-scale mixer, given in Table 4-2, was used as the average value for the pilot-scale mixer (0.033). The completely mixed time at the production-scale into the same experimental condition was 31 and 32 sec respectively. Thus the average value (31.5sec) was used for the flow rate calculation. Using average value of *k* and the completely mixed time, the circulation flow rate of the production-scale mixer was estimated as 234 m<sup>3</sup>/h.

### 4.4.2 Influence of mixing time and production volume on mean drop diameter

All the experimental conditions and calculated values are summarized in Table 4-3. The adequacy of the scale-up factor based on  $\epsilon_t$  is shown in Table 4-3, and the results of drop size measurements are discussed.

Figure 4-2 shows the relationship between the mean drop diameter and the mixing time at different rotational speeds and product volumes for the pilot-scale mixer. The drop size decreases rapidly at a high rotor speed, small product volume, and long mixing time. From the result of comparison between the stator No.2 and 4, it is clearly indicated that the mean drop diameter is strongly affected by the rotor tip speed.

In order to confirm the influence of the product volume, the relationship between the circulation number and the drop size was examined, as shown in Figure 4-3. All data are the same as those shown in Figure 4-2. The circulation number  $N_c$  [-] is defined as follows:

$$
N_c = \frac{Q}{V} \cdot t_m \tag{4-4}
$$

The circulation number includes information about the mixing time  $t_m$  and product volume V. From this figure, it can be seen that the mean drop diameter is the same at a given rotational speed, irrespective of the product volume

In the case of the same rotational speed, the mean drop diameter was determined by the circulation number. However, it was inferred that the circulation number did not necessarily influence the mean drop diameter at different rotational speeds. This result indicates that it is necessary to evaluate the homogenization intensity during a single pass through the mixing unit.

### 4.4.3 Relationship between the mean drop diameter and total energy dissipation rate

In order to confirm the validity of the evaluation method of the homogenization effect using  $\varepsilon_t$ , the mean drop diameters obtained under different operating conditions were compared.

Figure 4-4 shows the mean drop diameter, which is ordered by  $\varepsilon_t$ . All data are the same as those shown in Figures 4-2 and 4-3. It was found that the drop size showed a similar slope under different product volumes and rotational speeds.

This result suggests that  $\varepsilon_t$  can be used to describe the influence of the rotor speed and product volume on the mean drop diameter. This result also suggests that  $\varepsilon_t$  adequately corresponds with the homogenization intensity during a single pass through a batch-mixing unit. It is clear from Eqs. (4-2) and (4-3) that  $\varepsilon_t$  includes information about the difference in the mixer configurations  $(C_h)$ ,  $t_m$ , *N,* and *V.* It was found that the mean drop diameter reduced in proportion to  $C_{\rm h}$ ,  $t_{\rm m}$ ,  $N^4$ , and  $V^{-1}$ , in theory. Because  $\varepsilon_{\rm t}$  can account for the mean drop diameter, it was considered the evaluation theory of the homogenization effect to be validated.

# **4.4.4 Validity of scale-up method for mean drop diameter using total energy dissipation rate**

**Figure 4-5** shows the mean drop diameter, ordered by  $\varepsilon_t$ , for different-sized mixers and at various rotational speeds. As shown in Figure 4-4, the mean drop diameter was in good agreement for both the mixers at different rotor speeds and product volumes. The ratio of the rotor diameter of the pilot-scale mixer (0.198 m) to the rotor diameter of the production-scale mixer (0.396 m) was approximately 2.

Further, the volume ratio of the pilot-scale mixer  $(200 \text{ L})$  to the production-scale mixer (7000 L) was almost 35. The mean drop diameter could be explained using  $\varepsilon_t$ , by considering the influence of the mixer size and the batch volume. However, the large mixer produced a wider- drop-size distribution, although its mean drop diameter was the same as that of the small mixer. For example, the standard deviation of production-scale mixer is much higher value ( $\sigma$ )  $= 0.19$ ) compare with the pilot-scale mixer ( $\sigma = 0.11$ ) at similar mean drop diameter  $(d_{50} = 0.50 \,\mu \text{ m})$ .

# **4.4.5 Estimation method of equivalent mixing time for production-scale mixer**

From an engineering viewpoint, obtaining information about an equivalent mixing time, i.e., the time after which the mean drop diameter for both the pilot- and production-scale mixers will be the

same, is more important than obtaining information about the maximum stable drop size, which is obtained over a long mixing time, for the design of production lines.

**Figure 4-6** shows the relationship between the rotational speed and  $\varepsilon_t$ . This figure indicates that  $\varepsilon_t$  of the production-scale mixer was smaller than  $\varepsilon_t$  of the pilot-scale mixer at the same rotational speed. This implies that the production-scale mixer required a longer mixing time to obtain the same mean drop diameter as that obtained in the pilot-scale mixer.

**Table 4-4** summarizes the estimated results of the equivalent mixing time for the production-scale mixer. The theoretically estimated equivalent mixing time for the production-scale mixer at 15  $s^{-1}$  and 17  $s^{-1}$  was 42 min and 18 min, respectively. These mixing times corresponded to a mixing time of 5 min at 27  $s^{-1}$  for the pilot-scale mixer.

In order to confirm the adequacy of estimation using  $\varepsilon_t$ , the decreasing trend in the mean drop diameter for the measured and estimated values was compared.

**Figure 4-7** shows the estimated mean drop diameters at 15  $s^{-1}$ and 17  $s^{-1}$  for the production-scale mixer, which were obtained from the experimental data for the pilot-scale mixer at  $27 \text{ s}^{-1}$ . The estimated mean drop diameter of the production-scale mixer showed a decreasing trend, as in the case of the measured values. It was found that the mean drop diameter estimated using  $\varepsilon_t$  showed a high degree of adequacy. This implies that it is possible to scale-up the mean drop diameter of production-scale mixers in terms of the mixing time. It is

considered that the estimation method proposed in this study to be correct.

### **4.5 Conclusions**

In this chapter, the total energy dissipation rate  $(\varepsilon_t)$  was proposed as a scale-up factor for the mean drop diameter in internally circulated batch rotor-stator mixers. This factor was selected on the basis of the concept that the shear frequency and the local energy dissipation rate of turbulence account for the mean drop diameter at different rotor speeds for mixers with different configurations (different gap widths, rotor diameters, and opening areas) and for different product volumes. In other words, the mean drop diameter could be estimated by using  $\varepsilon_t$ . Results of this study also suggested that  $\varepsilon_t$  accurately corresponded with the homogenization intensity during a single pass through a batch-mixing unit. The scale-up criteria for the mean drop diameter in terms of the mixing time should be based on  $\varepsilon_t$  and not necessarily on geometrical similarities, constant rotor tip speed, or constant gap width, in the case of similar mixer configurations and the experimental range (200-7000 L) used for model products.

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the estimation method of the flow rate for the production-scale mixer



Fig. 4-2 Profiles of mixing time vs. mean drop diameter for the pilot-scale mixer



Fig. 4-3 Plot of circulation number vs. mean drop diameter for the pilot-scale mixer



Fig. 4-4 Plot of total energy dissipation rate vs. mean drop diameter for the pilot-scale mixer







Fig. 4-6 Plot of rotational speed vs. total energy dissipation rate for the pilot and production mixer



Mixing time  $T_m$  [min]

Fig. 4-7 Estimated mixing time for the production-scale mixer



 $\hat{\boldsymbol{\gamma}}$ 

**Table 4-1** Specifications of mixing units

Stator hole diameter  $d: 4$  mm Number of stator blades  $n_r$ : 6

 $\sim$ 

un production-scale inixer					
			Pilot scale		Production scale
Rotational frequency	$\overline{N}$	$\lceil 1/s \rceil$	27	14	20
Power consumuption	$P_{n}$	[kW]	10.1	1,2	108
Power number	$N_{\rm p}$	$\lceil - \rceil$	1.63	1.59	1.39
Completely mixed time	$T{_{\rm M}}$	[s]	13	22	31.5
Nondimensional mixed time	$T{_{\rm M}}$ . $\overline{N}$	$[\cdot]$	355	300	630
Flow number	$N_{\mathsf{qd}}$	$\lceil - \rceil$	0.067	0.067	0.052
Flow rate	$\varrho$	$\left[\text{m}^3/\text{h}\right]$	51	26	234
Constant	$\boldsymbol{k}$	$\left  - \right $	0.030	0.036	0.033

**Table 4-2** Measured values and estimated flow rate of the production-scale mixer

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 $\ddot{\phantom{a}}$
			<b>Table 4-3</b> Experimental conditions, measured and calculated values of emulsification experiments							
			Pilot scale				Production scale			
				$\overline{2}$	$\overline{3}$	4	5.	6	$\tau$	
Volume	$\boldsymbol{V}$	$\lceil m^3 \rceil$	0.2	0.3	0.5	0.3	7.0	7.0	7.0	
Rotational speed	$\boldsymbol{N}$	[1/s]	27.3	27.3	27.3	20.5	15.0	16.0	17.5	
Rotor tip velocity	U	[m/s]	17.0	17.0	17.0	12.7	18.7	19.9	21.8	
Net power consumption	$P_{\rm n}$	[kW]	10.3	10.3	10.3	4.3	46.7	56.3	73.2	
Power number	$N_{\rm p}$	$\left[ -\right] % \includegraphics[width=0.9\columnwidth]{figures/fig_2b.pdf} \caption{The graph $\mathcal{N}_1$ is a function of the parameter $\mathcal{N}_1$ and $\mathcal{N}_2$ is a function of the parameter $\mathcal{N}_1$ and $\mathcal{N}_2$ is a function of the parameter $\mathcal{N}_1$ and $\mathcal{N}_2$ is a function of the parameter $\mathcal{N}_1$ and $\mathcal{N}_2$ is a function of the parameter $\mathcal{N}_1$ and $\mathcal{N}_2$ is a function of the parameter $\mathcal{N}_1$ and $\mathcal{N}_2$ is a function of the parameter $\mathcal{N}_1$.} \label{fig:1}$	1.61	1.61	1.61	1.61	1.38	1.37	1.36	
Estimated flow rate	$\varrho$	$\lceil m^3/h \rceil$	51.3	51.3	51.3	38.3	175.5	187.2	204.8	
Flow number	$N_{\text{qd}}$	$\left[ -\right]$	0.067	0.067	0.067	0.067	0.052	0.052	0.052	
Pump power consumption	$P_{\rm p}$ .	[kW]	4.1	4.1	4.1	1.7	16.9	20.6	26.9	
Homogenization power consumption	$P_\mathrm{\,h}$	kW	6.1	6.1	6.1	2.6	29.8	35.8	46.3	
Ratio of cotributed to homogenization	$P_{\rm h}$ / $P_{\rm n}$	$\left[\begin{array}{c}\n\end{array}\right]$	0.60	0.60	0.60	0.60	0.64	0.63	0.63	
Total energy dissipation ratio	$\varepsilon_{\rm t}$	$\rm [m^2/s^3]$	$1.05 \times 10^{5}$				$7.00 \times 10^{4}$ 4.20 $\times 10^{4}$ 2.21 $\times 10^{4}$ 1.08 $\times 10^{4}$ 1.38 $\times 10^{4}$		$1.95 \times 10^{4}$	

 $\bullet$ 



 $\overline{\phantom{a}}$ 

**Table 4-4** Estimation result of equivalent mixing time for production-scale mixer

### **Chapter 5 Conclusion**

#### **5.1 General conclusion of this study**

In this study, a theoretical evaluation method of the homogenization effect was proposed for different stator configurations of internally and externally circulated batch rotor-stator mixers. Further, the scale-up factor was derived in relation to the mixing time and the validity of the scale-up procedure proposed in this study was confirmed using a model product.

In **chapter 1,** the background of this study and the current knowledge was explained about general rotor-stator mixers. Further, the objectives of this study were described. Problems related to the actual operation of rotor-stator mixers and the necessity of a solution to these problems was also discussed.

In **chapter** 2, a scale-up factor (homogenization index: *H.I)* for the mean drop diameter was proposed in externally circulated batch rotor-stator mixers. This index is based on the concept that the circulation number and local energy dissipation rate of turbulence could well account for the mean drop diameter at different rotor speeds, with different mixer configurations (gap width and rotor diameter), and for different production volumes. In other words, the mean drop diameter could be estimated by using *HI.* This result also suggested that *H.I.* accurately corresponded with the homogenization intensity for a single pass through a batch-mixing unit. The scale-up criteria for the mean drop diameter in terms of mixing time should be based on H.I., and not necessarily on geometrical similarities, the

same rotor tip speed, or the same gap width in the case of similar mixer configurations and the experimental range of production volume (1 to 10 L) used for the model product.

In **chapter** 3, a method of evaluating the homogenization effect was proposed for different stator configurations of batch rotor-stator mixers with internal circulation. The homogenization effect for different stator configurations was evaluated using the homogenization coefficient  $(C_h)$  based on the power number, flow number, and shear frequency derived from the number of rotor blades and stator holes. Using a water mixing operation, it was found that the homogenization effect was considerably higher in the case of a small stator hole diameter and a large number of stator holes. Based on the  $C_h$  evaluations, it was also found that the clearance between the rotor and the stator had less influence on the homogenization effect than the hole diameter and the number of holes. the adequacy of the mean drop diameter estimation by  $C_h$  was verified using model product trials. The total energy dissipation rate that was contained by the  $C<sub>h</sub>$  could well account for the mean drop diameter in relation to the mixing time and the stator configuration differences. The results indicated that it is possible to compare and evaluate the homogenization effect for different configurations of rotor-stator mixers using  $C<sub>h</sub>$  in a water mixing operation without actual product trials.

In **chapter** 4, I proposed the total energy dissipation rate ( $\epsilon$ <sub>t</sub>) as a scale-up factor for the mean drop diameter in internally circulated batch rotor-stator mixers. This factor was selected on the basis of the

concept that the shear frequency and the local energy dissipation rate of turbulence account for the mean drop diameter at different rotor speeds for mixers with different configurations (different gap widths, rotor diameters, and opening areas) and for different product volumes. In other words, the mean drop diameter could be estimated by using  $\epsilon$ <sub>t</sub>. The results also suggested that  $\epsilon$ <sub>t</sub> accurately corresponded with the homogenization intensity during a single pass through a batch-mixing unit. The scale-up criteria for the mean drop diameter in terms of the mixing time should be based on  $\epsilon_t$  and not necessarily on geometrical similarities, constant rotor tip speed, or constant gap width, in the case of similar mixer configurations and the experimental production volume range (200-7000 L) used for the model products.

#### **5.2 Future work**

The use of rotor-stator mixers in various fields is expected to increase year after year. From an engineering viewpoint, it is considered that the necessity for an adequate evaluation of the rotor-stator mixer performance will also increase. Further, I believe that the results of this study can be adapted to various engineering needs.

In this study, in order to estimate the mean drop diameter, I proposed the use of *HI.* for the mixer that is strongly affected by the gap between the rotor and the stator. On the other hand, I also proposed the use of  $\varepsilon_t$  for the mixer that is mainly affected by stator holes. However, I could not clarify the distinction criterion that can be used for categorizing mixers into various types. I think it will be useful if we can find the distinction criterion and develop a common

index that can evaluate all types of mixers using the same theory.

At the end of this thesis, I mention the possibility of new index which merges both scale-up index  $H.I$  and  $\varepsilon$ <sub>t</sub>. The both index has same dimension  $(m^2/s^3)$ . This dimension means energy dissipation rate.

The calculation theory of *H.I.* is simple, but it is difficult to be reflected the difference of mixer configurations directly. And  $\varepsilon_t$  is calculated under the ideal condition. Actually, the calculation of  $\varepsilon$ <sup>t</sup> should be considered the leakage flow from gap between the rotor and stator. Based on these improvements, I look over the leading process of *H.I.* and  $\varepsilon$ <sub>t</sub> again, and I propose a new scale-up and performance estimation index which connotes the concept of *H.I* and  $\varepsilon$ <sub>1</sub>. Detail information of the leading process about new index is shown in appendix.

$$
\varepsilon_{\rm a} = \varepsilon_{\rm g} + \varepsilon_{\rm s} \tag{5-1}
$$

$$
\varepsilon_a = \left[ \left( N_p - N_{qd} \pi^2 \right) \cdot n_r \right] \cdot \left[ D^3 \left( K_g + K_s \right) \right] \cdot \left( \frac{N^4 \cdot t_m}{V} \right) \tag{5-2}
$$

$$
K_g = \left(\frac{D^3 b}{\delta(D+\delta)}\right) \tag{5-3}
$$

$$
K_s = \frac{\pi^2 n_s^2 d(d+4\ell)}{4N_{qd}} \cdot \left(\frac{d^2}{n_s \cdot d^2 + 4\delta(D+\delta)}\right)
$$
 (5-4)

 $\epsilon_a$  [m<sup>2</sup>/s<sup>3</sup>] is overall energy dissipation rate [m<sup>2</sup>/s<sup>3</sup>],  $\epsilon_g$  [m<sup>2</sup>/s<sup>3</sup>] is energy dissipation rate at gap which expresses similar effect of the *H.I.*, and  $\varepsilon$  s [m<sup>2</sup>/s<sup>3</sup>] is energy dissipation rate at stator openings which has similar trend of  $\varepsilon$ <sub>t</sub>  $K_{\rm g}$  [m<sup>2</sup>] is the influence factor of gap,  $K_{\rm s}$  [m<sup>2</sup>] is the influence factor of stator openings.

I guess the reason why the *H.I.* and  $\varepsilon_t$  could well account for mean drop diameter in the definite type of mixers, using Eq. (5-2), to (5-4) as follows. In the mixer which was used at Chapter 2,  $K<sub>g</sub>$  is dominant, *K,* could be neglected. On the hands, it is considered that in the mixer which was used at chapter 3 and 4, *Ks* is dominant, the effect of  $K_{\rm g}$  is small.

However, the new index  $\varepsilon_a$  have just led recently. The validity of the theory and accuracy of estimation and scale-up will confirm in near future.

I hope that results of this study will be used for a new mixer design that will improve the production efficiency. If we can obtain detail information of mixer configuration, power consumption, and flow rate, we can estimate the mixer performance and equivalent mixing time for production scale mixers.

Finally, this study proposes the basic idea for an evaluation method of mixer performance.

In this appendix, we proposed a scale-up and evaluation method for estimating mean drop diameter for various batch rotor-stator mixers. We assumed that homogenization and emulsification occurs at the gap between the rotor and the stator and at the stator openings. The energy dissipation rate at the rotor tips and the stator openings is calculated experimentally using the local homogenization power consumption that is defined using the homogenization intensity of the liquid received in each area and the shear frequency, which is the number of times the product receives the shear stress in each area. We defined the overall energy dissipation rate  $\varepsilon_a$  in terms of the homogenization intensity and the shear frequency. This theoretical scale-up method has not confirmed the validity and accuracy for scale-up and performance estimation yet. It is just a concept of integration with *H.I.* and  $\varepsilon$ <sub>t</sub>.

#### **A-I Theory**

## **A-I.I Calculation methods of the local energy dissipation rate at rotor tip**

In chapter 2, authors defined the power consumption that contributed to a homogenization as *Ph.* They assumed that the homogenization effect was related to  $P_h$ .  $P_h$  is calculated by subtracting the net power consumption  $P_n$  from the pump power consumption  $P_p$  as given in Eqs. (A-1) to (A-3).

$$
P_{\rm n} = N_{\rm p} \rho N^3 D^5 \tag{A-1}
$$

$$
P_{\rm p} = \rho \pi^2 N^2 D^2 Q
$$
  
=  $N_{\rm qd} \pi^2 \rho N^3 D^5$  (A-2)

$$
P_{\rm h} = P_{\rm n} - P_{\rm p}
$$
  
=  $(N_{\rm p} - \pi^2 N_{\rm qd}) \rho N^3 D^5$  (A-3)

Here, N is the rotational speed;  $N_{\text{p}}$ , the power number;  $N_{\text{qd}}$ , the flow number;  $\rho$ , the density;  $Q$ , the flow rate; and  $D$ , the rotor diameter. All variables in Eq. (A-3) can be derived experimentally.

According to Tatterson (2003), the power consumption was calculated by multiplying the flow rate (impeller pumping) by the shear stress (head). From this relationship, the average shear stress at a stator hole  $\tau_a$  is calculated by using Eqs. (A-1)- (A-3) as given in Eq.  $(A-4)$ .

$$
\tau_{a} = \frac{P_{h}}{Q}
$$

$$
= \rho N^{2} D^{2} \left( \frac{N_{p}}{N_{qd}} - \pi^{2} \right)
$$
(A-4)

**Figure A-I** shows a schematic diagram of the rotor tip. We

assume that the area subjected to the shear stress  $S_b$  is the cross section of the tip of the rotor blade.

$$
S_b = b \cdot h_b \tag{A-5}
$$

Here, *b* is the thickness of the rotor blade [m],  $h<sub>b</sub>$  is the rotor height [m]. Therefore, the average force at a stator hole  $F<sub>b</sub>$  is calculated by multiplying the shear stress (Eq. (A-4)) with the area subjected to the shear stress (Eq.  $(A-5)$ ) as given in Eq.  $(A-6)$ .

$$
F_b = \tau_a \cdot S_b
$$
  
= 
$$
\frac{(N_p - N_{qd}\pi^2)\rho N^2 D^2}{N_{qd}} \cdot b \cdot h_b
$$
 (A-6)

The homogenization intensity at the tip of the rotor blade  $W<sub>b</sub>$  is defined in terms of the average force  $F_b$  [N], velocity  $U$  [m/s], and number of rotors  $n_r$  [-] as shown in Eq. (A-7).

$$
W_{b} = F_{b} \cdot U \cdot n_{r}
$$
  
=  $\left(\frac{(N_{p} - N_{qd}\pi^{2})\rho\pi \cdot n_{r}}{N_{qd}}\right) \delta \cdot h \cdot D^{3} \cdot N^{3}$  (A-7)

This factor indicates the homogenization intensity in a single pass through a product.

As the other key factor for homogenization with rotor-stator mixers, Porcelli (2002) defined the shear frequency  $f[1/s]$ . In order to estimate the number of times the product receives the shear stress at certain gap volume, the shear frequency for the gap volume  $f_g$  is calculated using Eq. (A-8). The gap volume shown in Figure 2 is calculated by subtracting the swept-out volume of the rotor diameter from the swept-out volume of the stator diameter.

$$
f_g = \frac{N_{qd}ND^3}{\pi \cdot h_b \cdot \delta(D+\delta)}
$$
 (A-8)

Here,  $\delta$  is the clearance between the rotor and the stator and  $h<sub>b</sub>$  is the height of the stator.

Davies (1987) experimentally indicated that the mean drop diameter depends on the energy dissipation rate per unit mass. Utomo *et al.* (2008) numerically suggested that the energy dissipation rate around the leading and trailing edges of the stator holes was higher than that at the other parts. Porcelli (2002) theoretically reported the importance of the shear frequency for the mean drop diameter in rotor-stator mixers. In chapter 3 and 4, authors also reported that the decreasing trend of the mean drop diameter could well account for the total energy dissipation rate. From these facts, we assume that the main factor for homogenization is the local energy dissipation rate  $\varepsilon_{\rm g}$ , which contains information about the local homogenization intensity  $W_{\rm b}$ , shear frequency at the rotor tip  $f_{\rm g}$  and mixing time  $t_{\rm m}$ . The local energy dissipation rate  $\varepsilon_{\rm g}$  is expressed as follows in Eq. (A-9).

$$
\varepsilon_{g} = \frac{W_b \cdot f_g \cdot t_m}{\rho \cdot V}
$$
  
=  $\left[ \left( N_p - N_{qd} \pi^2 \right) \cdot n_r \right] \cdot \left( \frac{D^6 b}{\delta (D + \delta)} \right) \left( \frac{N^4 \cdot t_m}{V} \right)$  (A-9)

# **A-t.2 Calculation of local energy dissipation rate at stator openings**

The local energy dissipation rate at stator openings can be calculated in a manner similar to that mentioned in the above theories. The area subjected to the shear stress  $S_s$  is the sum of the area of the side wall  $S_w$  and the area of a stator-hole cross section  $S_d$ .

$$
S_s = S_w + S_d
$$
  
= 
$$
(\pi dl + \frac{\pi}{4}d^2)
$$
 (A-10)

Here,  $d$  is the diameter of the stator hole and  $l$  is the thickness of the stator wall.

Therefore, the average force at a stator hole  $F_s$  is calculated by multiplying the shear stress (Eq. (A-4)) with the area subjected to the shear stress (Eq.  $(A-10)$ ), as given in Eq.  $(A-11)$ .

$$
F_s = \tau_a \cdot S_s
$$
  
= 
$$
\frac{(N_p - N_{qd}\pi^2)\rho N^2 D^2}{N_{qd}} \pi \cdot d\left(\frac{d}{4} + \ell\right)
$$
 (A-11)

The homogenization intensity at stator openings  $W_s$  is defined in terms of average force  $F_s$  [N] and velocity  $U$  [m/s], as given in Eq. (12).

$$
W_s = F_s \cdot U
$$
  
= 
$$
\left[ \frac{(N_p - N_{qd}\pi^2)\pi^2}{N_{qd}} \right] \rho N^3 D^3 d \left( \frac{d}{4} + \ell \right)
$$
 (A-12)

The shear frequency  $f[1/s]$  at the stator openings is defined by Porcelli (2002) as follows;

$$
f = n_s \cdot n_r \cdot N \tag{A-13}
$$

In the case of actual mixers, there are some leakage flows at the gap between the rotor and the stator. It is considered that the leakage flows from the gap are in proportion to the ratio of the gap cross section  $S_{g}$  [m<sup>2</sup>] to the total cross section of the stator wall openings  $S_{h}$ [m<sup>2</sup>]. The leakage ratio  $\alpha$ <sub>L</sub>[-] is calculated as follows:

$$
S_h = \frac{\pi}{4} d^2 \cdot n_s \tag{A-14}
$$

$$
S_g = \pi \delta (D + \delta) \tag{A-15}
$$

$$
\alpha_{\rm L} = \frac{S_{\rm h}}{S_{\rm h} + S_{\rm g}} \tag{A-16}
$$

$$
= \frac{n_s \cdot d^2}{n_s \cdot d^2 + 4\delta(D+\delta)} \tag{A-17}
$$

Therefore, the shear frequency at the stator openings is calculated as given in. (A-I7).

$$
f_s = n_s^2 \cdot n_r \cdot N \cdot \left(\frac{d^2}{n_s d^2 + 4\delta(D + \delta)}\right) \tag{A-18}
$$

The local energy dissipation rate at stator openings  $\varepsilon_{\rm s}$ , which contains information about the local homogenization intensity *Ws,* shear frequency at stator openings  $f_{\rm g}$  and mixing time  $t_{\rm m}$  is expressed as follows in Eq. (A-I8).

$$
\varepsilon_{s} = \frac{W_{s} \cdot f_{s} \cdot t_{m}}{\rho \cdot V}
$$
  
=  $\left[ \frac{\left(N_{p} - N_{qd}\pi^{2}\right)\pi^{2}n_{s}^{2} \cdot n_{r}}{N_{qd}} \right] D^{3}d\left(\frac{d}{4} + \ell \right) \left(\frac{d^{2}}{n_{s}d^{2} + \delta(D + \delta)} \right) \left(\frac{N^{4} \cdot t_{m}}{V}\right)$  (A-19)

### **A-l.3 Calculation** ofthe **overall energy dissipation rate**

We assumed that the homogenization and the emulsification take place at the gap between the rotor and the stator and at the stator openings. As the performance evaluation index and the scale-up index, we defined the overall energy dissipation rate  $\varepsilon_a$  as follows:

$$
\varepsilon_{\rm a} = \varepsilon_{\rm g} + \varepsilon_{\rm s}
$$

$$
= \left[ \left( N_p - N_{qd} \pi^2 \right) \cdot n_r \right]
$$
  
 
$$
\times \left\{ D^3 \left[ \left( \frac{D^3 b}{\delta (D + \delta)} \right) + \frac{\pi^2 n_s^2 d(d + 4\ell)}{4N_{qd}} \cdot \left( \frac{d^2}{n_s \cdot d^2 + 4\delta (D + \delta)} \right) \right] \right\} \left( \frac{N^4 \cdot t_m}{V} \right)
$$
  
(A-20)

Here

$$
K_g = \left(\frac{D^3 b}{\delta(D+\delta)}\right) \tag{A-21}
$$

$$
K_s = \frac{\pi^2 n_s^2 d(d+4\ell)}{4N_{qd}} \cdot \left(\frac{d^2}{n_s \cdot d^2 + 4\delta(D+\delta)}\right) \quad (A-22)
$$

$$
K_h = \left(N_p - N_{qd}\pi^2\right) \cdot n_r \cdot D^3 \cdot \left(K_g + K_s\right) \qquad (A-23)
$$

$$
\varepsilon_a = K_c \cdot \left(\frac{N^4 \cdot t_m}{V}\right) \tag{A-24}
$$

Here,  $K_{\rm g}$  [m<sup>2</sup>] is the influence factor of the gap,  $K_{\rm s}$  [m<sup>2</sup>] is the influence factor of the stator openings, and  $K_c$  [m<sup>5</sup>] is the configuration evaluation coefficient.



**Fig. A-1** Schematic diagram of the rotor tip



Fig. A-2 Schematic diagram of the mixing unit

# **Nomenclature**

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