Contents lists available at ScienceDirect

Review

Chinese Journal of Chemical Engineering

journal homepage: www.elsevier.com/locate/CJChE

**Chinese Journal of
CHEMICAL ENGINEERIN**

Mixing time in stirred vessels: A review of experimental techniques☆

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article info abstract

Article history: Received 13 December 2013 Received in revised form 2 July 2014 Accepted 28 October 2014 Available online 3 April 2015

Keywords: Mixing time Stirred vessel Homogeneity

Mixing time is defined as the time required for achieving a certain degree of homogeneity of injected tracer in a unit operation vessel. It has been used as a key parameter for assessing the performance of a mixing system. From an experimental standpoint, several techniques have been developed for measuring the mixing time. Based on the disturbances to flow, they can be classified into two groups: non-intrusive and intrusive. However, depending on the type of data generated, they can be also classified into direct measurements and indirect measurements (Eulerian and Lagrangian). Since the techniques available for measuring mixing times in an agitated tank do not provide the same information, its choice depends on several factors, namely: accuracy, reproducibility, suitability, cost, sampling speed, type of data, and processing time. A review of the experimental techniques reported in the literature in the last 50 years for the measurement of mixing time in stirred vessels under single and gas–liquid flow conditions with Newtonian and non-Newtonian fluids in the laminar and turbulent regime is made, and a comparison between these techniques is also presented.

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1. Introduction

Mixing time is a key parameter used for analyzing the performance and the hydrodynamics of a stirred vessel. In general terms, the mixing time is defined as the time required for achieving a certain degree of homogeneity of tracer inserted in a stirred vessel [\[1\].](#page--1-0) From a macromixing standpoint, the bulk mixing time is the time required to get all points in the vessel uniformly distributed, while the local mixing time is the measure of how fast a material is distributed in a particular region of the vessel, which depends on the local turbulence.

Therefore, local measurements are time and space dependent, while bulk mixing time is based on time dependent measurements (temporal measurements) [\[2\]](#page--1-0). Mixing time can be expressed in its nondimensional form:

$$
N\theta_{\rm m} = K \tag{1}
$$

where $\theta_{\rm m}$ is the mixing time in s, N is the impeller speed in revolutions per second and K is a constant which depends on the size, geometry of the tank and the flow regime [\[3\].](#page--1-0) Constancy of K is valid in the laminar and turbulent regimes but not in the transitional

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one. Moo-Young et al. [\[4\],](#page--1-0) the mixing time can be correlated in a dimensionless manner as

$$
N\theta_{\rm m} = \alpha (Re)^{\beta} \tag{2}
$$

where α and β are the adjustable parameters and Re is the Reynolds number,

$$
Re = \frac{\rho N D^2}{\mu} \tag{3}
$$

where ρ is the fluid density in kg·m⁻³, D is the impeller diameter in m and μ is the fluid dynamic viscosity in Pa \cdot s.

In the case of non-Newtonian fluids, such a number is defined by

$$
Re = \frac{\rho N^{2-n} D^2}{k} \tag{4}
$$

where k and n are the consistency index and the flow behavior index, respectively, for fluids obeying the Ostwald-de Waele model (powerlaw model), in which the rate of deformation has been taken as $\gamma = N$. However, the Reynolds number for non-Newtonian can also be defined in terms of the effective viscosity, which is a function of the shear rate. Nowadays, the most used definition of the shear rate in stirred vessels

Supported by DGAPA-UNAM through the grant IN-108312.

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is the one developed by Metzner and Otto [\[5\]](#page--1-0), which is calculated from the power-law model:

$$
\dot{\gamma} = \left(\frac{\mu}{k}\right)^{1/[n-1]} = k_s N \tag{5}
$$

where the shear rate is proportional to the impeller rotational speed, being k_s the proportionality constant. Based on this approach, the Reynolds number is defined as

$$
Re = \frac{\rho N D^2}{\mu} = \frac{\rho N^{(2-n)} D^2}{k k_s^{(n-1)}}.
$$
\n(6)

A number of mixing time correlations have been reported in the literature taking into account the effect of baffles on mixing time [6–[8\]](#page--1-0), as well as the relationship between circulation and mixing times [\[9\].](#page--1-0) In the turbulent regime, the dimensionless mixing time is independent on the Reynolds number and it can be correlated by empirical expressions involving the size and geometry of the tank as well as some hydrodynamic parameters [\[10,11\]](#page--1-0). Experimental techniques for measuring mixing time can be classified depending basically on two different scenarios, namely: (1) level of disturbance to flow and (2) type of data collected. Based on the first scenario, the techniques are classified as non-intrusive and intrusive. With respect to the intrusive techniques, they are based on local and global measurements. Although they are accurate, its use is limited because the flow pattern is modified by the presence of probes in the vessel. Mavros [\[12\]](#page--1-0) reviewed the experimental techniques available for the study of flow patterns in stirred vessels. Although the aim of his paper was to give a general overview of intrusive and non-intrusive techniques for the flow visualization in tanks, some of them can be also applied also for estimating mixing times.

On the other hand, considering the type of data collected, these techniques are classified as direct measurements or indirect measurements (Eulerian data or Lagrangian data). In the case of indirect measurements, mixing time is to be inferred from physical measurements of conductivity or velocity for instance, while direct techniques provide real time results or at least requiring minimum processing. Table 1 summarizes the classification of experimental techniques as a function of both scenarios.

Table 1

Classification of measurement techniques of mixing times in stirred vessels

Nere et al. [\[8\]](#page--1-0) performed a critical review of the literature dealing with liquid-phase mixing in stirred vessels in the turbulent regime. Although, the limitations of the techniques employed for measuring mixing times were described therein, they focused on the effects of different parameters such as impeller geometry, impeller position on liquid-phase mixing as well as the mathematical models developed

for analyzing the hydrodynamics in stirred vessels. In this paper, the experimental techniques reported in the literature over last 50 years for the measurement of mixing time in stirred vessels have been reviewed by highlighting their advantages and drawbacks along with the most outstanding findings. This review covers single phase and gas–liquid mixing of Newtonian and non-Newtonian fluids in the laminar and turbulent regimes.

2. Experimental Techniques

2.1. Colorimetry

From a practical standpoint, colorimetry is by far the most common technique employed for measuring mixing times in stirred vessels [\[13](#page--1-0)-15]. It is a non-intrusive technique extensively reported in the literature, which is used not only to determine the time required to achieve the desired degree of homogenization, but also to visualize qualitatively flow patterns and to reveal the presence of secondary flows generated under steady stirring such as well-mixed regions (caverns), islands and other segregated regions like stagnant of dead flow zones [16–[20\].](#page--1-0) The technique basically consists of injecting a liquid tracer and observing how it is dispersed in the fluid contained in stirred vessels. A variant of this technique is based on the color-decolorization approach by using a pH sensitive tracer. The reader is referred to Ascanio et al. [\[18\]](#page--1-0) for a detailed description of the measuring protocol. [Fig. 1](#page--1-0) shows an example of a typical mixing sequence by the colorimetric technique.

Norwood and Metzner [\[22\]](#page--1-0) reported for the first time the use of an acid–base neutralization reaction for measuring mixing time in baffled stirred tanks equipped with turbines operating in the turbulent regime, which has been extensively adopted in the research. Hari-Prajitno et al. [\[23\]](#page--1-0) used also this technique for measuring mixing time under aerated and unaerated conditions for dual and triple coaxial impeller configurations in the turbulent regime. The decolorization method has not only been used for measuring mixing time, but also for getting the insight of flow patterns generated by different impellers such as close-clearance impellers, multiple and coaxial arrangements, inclined impeller, etc. [\[24](#page--1-0)–34].

Although colorimetry is a very simple technique to implement, mixing time estimation depends strongly on the subjectivity of the human observer. For that reason, complementary techniques based on image processing have been developed [\[35](#page--1-0)–40]. Kouda et al. [\[35\]](#page--1-0) dissolved phenolphthalein as pH indicator in bacterial cellulose and CMC solution, which became dark purple when NaOH being present. Then, $H₂SO₄$ was added at time zero and the images were captured every 0.5 s. Afterwards, the digital brightness of images was analyzed by image processing, and the mixing time was determined in terms of the decolorization ratio (output/reference).

[Table 2](#page--1-0) summarizes the operating principle, advantages and disadvantages of the estimation of mixing time by colorimetry.

2.2. Electrical resistance tomography

Electrical Resistance Tomography (ERT) is a non-intrusive and noninvasive technique from which cross-sectional images showing the distribution of electrical conductivity of gas–liquid flows in a stirred vessel from Lagrangian measurements taken at the boundary of the vessel. It can be used where the continuous phase is conductive and the other phases have different values of conductivity. A typical ERT setup consists basically of a set of electrodes, the data acquisition system and a user interface [\[41\].](#page--1-0) The array of sensors are usually a series of rings in which the electrodes are equally spaced around the vessel circumference. Electrical measurements are made without affecting the flow in the vessel. A typical measurement protocol consists of applying the current to the next electrode pair and the resultant potential differences on all other electrode pairs are measured. This step is repeated until all electrode pair combinations have been used. A reconstruction technique converts the raw peripheral potential difference measurements into a

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