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Measurement of maximum stable drop size in aerated dilute liquid–liquid dispersions in stirred tanks

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HIGHLIGHTS

- We established a method to measure maximum stable drop size in aerated stirred tanks.
- The dispersed and continuous phases applied enable break-up controlled conditions.
- Maximum stable drop sizes are shown to be representative and highly reproducible.
- Method can be applied to correlate hydromechanical stress in aerated stirred tanks.
- Application in large scale and with high aeration and agitation rates possible.

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ABSTRACT

Turbulence intensity, or hydromechanical stress, is a controlling parameter in many industrially relevant processes. Especially fermentation processes are often characterized by intense aeration and agitation, operating conditions for which the measurement of turbulence intensity is extremely difficult. Since the maximum stable drop diameter in a break-up controlled dispersion is directly correlated with turbulence intensity, the measurement of drop sizes can enable an indirect access to the intensity of turbulence under such operating conditions. This work presents the constraints and the development of a method for the measurement of maximum stable drop size in aerated liquid–liquid dispersions in stirred tanks for the purpose of characterizing turbulence intensity. Continuous and dispersed phase properties were selected to achieve a break-up controlled dispersion with negligible coalescence. This was accomplished mainly by applying a dilute dispersion, a low ionic strength and by incorporating a dispersed phase, paraffin oil, with a negative spreading coefficient. The negative spreading coefficient prevents coalescence due to drop–bubble interactions for aerated operating conditions. It was demonstrated that the off-line measured drop size distributions are representative for the conditions in the bioreactor and are not altered by sample handling. The sampling and measurement procedure was found to be highly reproducible with a standard deviation for the maximum stable drop size for independent experiments of approximately 10%. Relevant constraints for the application of the method in large-scale experiments were discussed and accounted for during method development to allow a later application in production scale equipment.

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1. Introduction

The intensity of turbulence in stirred tank reactors is an important design parameter for a number of industrially relevant processes. Prominent examples can be found in the bioprocess industries. Jüsten et al. (1996, 1998a, b), e.g. showed in a series of publications that growth and productivity of submerged fungal cultures of *Penicillium chrysogenum* in stirred fermenters can be correlated with turbulence intensity. Other reports preceded the work of Jüsten et al. that identified hydromechanical forces to influence submerged fungal cultures (e.g. Makagiansar et al., 1993; Smith et al., 1990; Van Suijdam and Metz, 1981). Gregoriades et al. (2000) showed that Chinese hamster ovary cells immobilised on microcarriers are damaged by hydromechanical forces of magnitudes that can be expected in stirred tank processes. Further examples from the processing industries are suspension polymerisation (Vivaldo-Lima et al., 1997) and processes where interfacial area can become rate limiting like solvent extraction (Kumar and Hartland, 1996).

The intensity of turbulence in a stirred tank can be characterized by the maximum local energy dissipation rate. The energy

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dissipation rate is the rate at which kinetic energy introduced by agitation dissipates into heat. Energy dissipation is distributed inhomogeneously in the reactor volume and a maximum value exists that defines the most severe effects of the flow field on dispersed particles like oil drops and gas bubbles. This is the maximum local energy dissipation rate. Since industry scale processes are influenced by the maximum local energy dissipation rate, three important questions arise that demand for experimental access to this parameter: (1) what is the influence of the type of equipment used? In the fermentation industries, process development often starts in shake flasks. A transfer of results from shake flasks to the typical stirred tank reactor in the next development step is only possible if the characteristics and restrictions of these different types of processing equipment can be anticipated (Büchs, 2001; Humphrey, 1998). Peter et al. (2006) and Büchs and Zoels (2001) described a method to characterize hydromechanical stress in shake flasks. Mollet et al. (2004) give an overview on hydromechanical stress in different process equipments used during the propagation of cell cultures. However, there is a particular lack of available methods and data for the characterization of aerated stirred tanks under conditions that are close to process conditions. (2) What is the influence of scale and geometry? Exact geometric similarity is typically not conserved throughout scale-up from lab to pilot to production scale (Einsele, 1978; Junker, 2004). Therefore, it is important to be able to experimentally assess the influence of geometrical differences and scale on maximum local energy dissipation rate for a more rational approach to process scale-up and for systematic experiments during process development in small scale. (3) What is the influence of operating conditions? The major part of publicly available experimental data related to the maximum local energy dissipation rate is restricted to operating conditions with low agitation intensity in the range below 1 kW/m^3 and single phase operation (e.g. Zhou and Kresta, 1996). However, many processes are conducted under very different operating conditions. E.g. in most bioprocesses intense aeration is necessary to support the oxidative metabolism of microorganisms. Aeration is accompanied by strong agitation with volumetric power inputs typically in the range of 1–5 kW/m^3. Volume averaged gas hold-ups of 10–20% (v/v) are typical in process equipment (Junker, 2004). The influence of aeration on turbulence under such intense operating conditions has – to our knowledge – not been investigated experimentally up to now. Therefore, there is a need for a measurement method that enables the comparison of different agitators in different scales with respect to hydromechanical stress in “real life equipment” under operating conditions that are comparable to process conditions. This report focuses on the development of a measurement method that addresses the questions raised above. The method is based on the measurement of drop size distributions and maximum stable drop sizes in aerated dilute liquid–liquid dispersions in stirred tanks.

2. Relevance of the maximum stable drop size

Drop sizes in dispersions evolve by the impact of the turbulent flow field of the continuous phase on the non-soluble dispersed phase. Turbulence causes the break-up of larger drops into smaller fragments, resulting in a drop size distribution that depends on geometry, operating conditions, physico-chemical properties of the continuous and dispersed phases and the age of the dispersion. Coalescence of drops may be superimposed which then further complicates the process. Chemical engineering aspects of such complex systems have been investigated for many years. Hinze (1955) and Kolmogorov (1949) were amongst the first to model the break-up process of drops in a turbulent flow field. Their pioneering work was later extended by Arai et al. (1977) and by Calabrese et al. (1986a) to viscous dispersed phases. Their break-up model is based on the concept that a drop exposed to a turbulent flow field experiences hydromechanical forces that cause a deformation. The deformation is counteracted by interfacial tension and viscosity of the drop. The drop breaks up if the deforming stress exceeds the cohesive forces from interfacial tension and viscosity. Drop break-up will persist until a drop size is reached for which the hydromechanical stresses are too weak to overcome the cohesive forces. The size of these largest drops that can resist break-up is called the “maximum stable drop size” (d_{max}). If coalescence can be neglected, the maximum stable drop size is directly related to the maximum local energy dissipation rate for low dispersed phase viscosity by the well known correlation of Hinze (1955):

$$d_{max,0} \sim \left(\frac{\sigma}{\rho_c}\right)^{0.6} \cdot \varepsilon_{max}^{-0.4} \tag{1}$$

With the maximum stable drop diameter of an inviscid dispersion $d_{max,0}$. Viscosity can be accounted for by a correction factor introduced by Arai et al. (1977):

$$d_{max} = d_{max,0} \cdot \left(1 + 2.5 \cdot \frac{\eta_d}{\sigma}(\varepsilon_{max} \cdot d_{max})^{1/3}\right)^{0.6} \tag{2}$$

The classical concept of maximum stable drop size was extended by Baldyga et al. (2001) to account for the observation that even for very long agitation times the maximum stable drop size keeps drifting slowly towards smaller values. This was explained by the intermittent character of turbulence which results in rare but strong bursts of turbulence. For practical applications the effect of turbulence intermittency must be judged in comparison to measurement accuracy and relevant time scales. During typical dispersion times of 1–3 h, the effect might be below the reproducibility of consecutive measurements.

The theory of turbulent drop dispersion is applicable if the drop size is in the size range of the turbulent eddies. Based on Kolmogorov's theory of isotropic turbulence, the size range of turbulent eddies can be estimated to fall within the macroscale of turbulence Λ and the microscale of turbulence λ. The macroscale of turbulence is a measure for the largest eddies that can be estimated to scale proportionally with the impeller blade height h_b (Liepe et al., 1988):

$$\Lambda = 0.4 \cdot h_b \tag{3}$$

By decaying, these large eddies pass on their energy to smaller eddies in an energy cascade until an eddy size is reached for which viscous forces become predominant. This smallest scale of eddies is the microscale of turbulence λ which can be estimated as

$$\lambda = \sqrt[4]{\frac{\nu^3}{\varepsilon}} \tag{4}$$

where ν is the kinematic viscosity and ε is the local energy dissipation rate per unit mass. Whereas the macroscale of turbulence only depends on impeller geometry, the microscale of turbulence depends on the physical properties of the liquid (ν) and the operating conditions (ε). Typical values for the macroscale of turbulence can be calculated as approx. 10^{-2} m for a 0.1 m^3 stirred tank and 10^{-1} m for a 100 m^3 stirred tank. The microscale of turbulence is typically of an order of 10^{-5} m (Zhou and Kresta, 1998). The relationship between maximum local energy dissipation rate and maximum stable drop size can be applied to compare the turbulence intensity for different geometries and operating conditions by measuring d_{max}. From the theory explained above it can be concluded that the prerequisites for the validity of this method are a break-up controlled dispersion and a maximum stable drop size between the microscale and the macroscale of

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