An Experimental Methodology for Bubble Diameter Estimation in a Gas-Inducing Reactor: Application to CO_2 Cycloaddition of Bio-Based Epoxides

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Highlights

- Power number characterization of a gas-inducing impeller across a wide viscosity range
- High-speed camera imaging of CO_2 bubbles in bio-based epoxides
- Bubble size distributions and gas holdup measurements
- Gas-liquid mass transfer coefficients and interfacial area estimation

1. Introduction

Gas-liquid mass transfer is a critical factor governing the performance of multiphase reactors, particularly when gaseous reactants must dissolve into a viscous liquid phase to reach catalytic sites. The volumetric mass transfer coefficient kLa is the standard parameter for characterizing gas-liquid mass transfer, however, interpreting kLa in terms of reactor design and scale-up requires separating the liquid-side mass transfer coefficient kL from the interfacial area A, the latter being directly governed by bubble size and gas holdup. Interfacial area is notoriously difficult to measure directly, particularly in pressurized stainless steel reactors where optical access is not available, making reliable methodologies for bubble diameter and gas holdup estimation a key challenge in reactor engineering.

Gas-inducing impellers are a common solution in pressurized batch gas-liquid systems, as they self-aspirate gas from the headspace without requiring external sparging, producing fine bubbles that enhance interfacial area. However, characterization of this type of impeller, particularly in terms of power consumption and bubble onset speed, remains scarce for viscous non-aqueous systems [1,2], and direct bubble imaging at autoclave conditions is generally not feasible.

Bubble diameter has been correlated to specific power input and fluid physical properties through expressions of the form $D_{32} = C\,(\Pi/V)^a\,\rho^b\,\mu^c\,\sigma^d$ [3,4]. Once fitted to experimental data, such expressions can estimate bubble size where direct measurement is not possible. The present work develops and validates this methodology for a laboratory-scale autoclave reactor equipped with a gas-inducing impeller operating with viscous bio-based fluids, integrating power number characterization, high-speed camera bubble imaging, and kLa measurements to separate kL from kLa at process conditions — applied to CO_2 cycloaddition to epoxidized vegetable oils and fatty acid esters.

2. Methods

A 300-mL laboratory-scale autoclave reactor equipped with a gas-inducing impeller was used throughout this study. The working fluids were P401 (epoxidized methylhexyl linoleic ester) and CP401 (its carbonate derivative), along with additional reference fluids covering a wide viscosity range. Density, viscosity, and surface tension were measured as a function of temperature.

Power number characterization was performed across the full laminar-to-turbulent range at ambient conditions. Shaft power was measured with a precision power analyser and corrected for mechanical losses via air blank subtraction. Np and Re were computed for both ungassed and gassed conditions, with bubble onset speed determined experimentally for each fluid.

Bubble size distributions and gas holdup were measured in a glass reactor of identical geometry to the autoclave vessel, using a high-speed camera and a diode line laser as the illumination source. D_{32} and gas holdup ε were extracted from image analysis and fitted to a power-law scaling expression in Π/V, ρ, μ, and σ.

3. Results and discussion

Figure 1A shows the power number curve obtained for the gas-inducing impeller across a Reynolds number range spanning nearly six orders of magnitude, covering the full laminar-to-turbulent regime. Data from all working fluids collapse onto a single master curve, confirming that Np is a function of Re alone and that the impeller behaves consistently across the wide viscosity range studied. The curve is well described by $Np = 77.60/Re^{1.04} + 1.77$ ($R^2 = 0.9753$), recovering the expected limiting behaviors: a viscous-dominated regime at low Re and a turbulent plateau at high Re. The collapse of data from fluids as different as CLSO (Re ~ 0.1) and water (Re ~ 10^4) onto a single correlation validates the dimensional approach and provides a reliable power input estimation tool across all operating conditions of interest.

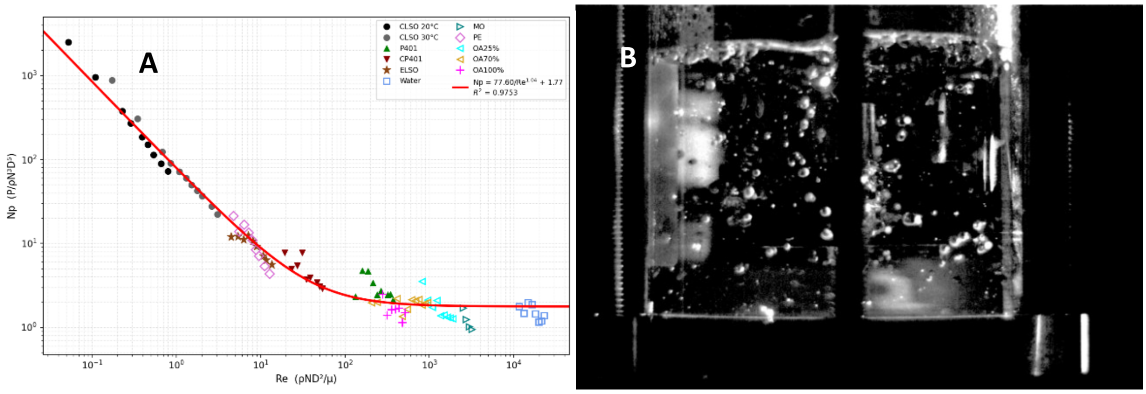

Figure 1. Power number characterization of the gas-inducing impeller (A) and high-speed camera image of CO_2 bubble dispersion in P401 epoxide substrate.

Figure 1B shows a representative high-speed camera image from the bubble imaging setup, illustrating the quality of optical access achieved in the glass reactor. Individual bubbles are clearly resolved, demonstrating the feasibility of the image analysis approach for extracting bubble size distributions and gas holdup. Systematic bubble imaging experiments across the working fluids and impeller speeds are ongoing, and the resulting D_{32} and ε data will be fitted to the scaling expression to enable estimation at process conditions.

4.Conclusions

A power number correlation for a gas-inducing impeller was established across nearly six orders of magnitude in Reynolds number, covering the full laminar-to-turbulent regime for a wide range of viscous bio-based fluids. The data collapse onto a single Np–Re master curve, providing a reliable power input estimation tool for all operating conditions of interest. A high-speed camera imaging setup in a glass reactor of identical geometry to the autoclave vessel was developed and validated for bubble size distribution and gas holdup measurement. Together, these results form the experimental foundation of a scaling methodology that will enable bubble diameter and interfacial area estimation at elevated temperature and pressure conditions, ultimately allowing kL to be separated from kLa for CO_2 cycloaddition to epoxidized vegetable oils and fatty acid esters.

References

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Keywords
gas-inducing impeller; bubble diameter scaling; gas-liquid mass transfer; bio-based epoxides