Testing of Phase Transition and Bubble Dynamics
Using A Four-Point Optical Probe
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Introduction

❖ By taking advantage of the difference in refractive index, an optical fiber can be used to determine when a single or multiple phases are present in a system. When multiple fibers are clustered together, bubble properties, as explained in the goals, can also be determined using a data processing algorithm developed in CREL at Washington University in St. Louis.

Project Goals

❖ Develop a diagnostic tool for detecting phase transition of expanded solvent/CO$_2$ systems from multi-phase to single phase.

❖ Evaluate the suitability of a four-point optical probe and the algorithm for data processing in a stirred autoclave reactor for measuring the following properties:

- Bubble chord length distribution, local gas hold-up, bubble velocity, and specific interfacial area.

Relevant Work

❖ Phase behavior of expanded solvent/CO$_2$ systems with acetone$^{2,3}$, ethanol$^3$, cyclohexane$^4$ and n-decane$^{5,6}$ has been studied by visual confirmation of phase separation.
Methodology

- The optical probe uses the difference in refractive index of liquid, gas, and optical fiber to distinguish between the gas and liquid phase.

- The output voltage is low when the probe tip is in the liquid phase and it is high when it is in the gas phase. From the output signals, bubble velocity vector, bubble chord length, specific interfacial area and local gas holdup are calculated.
Achievements

- The developed four-point optical probe, and the algorithm for data processing have been used to investigate the bubble velocity distribution, bubble chord length distribution, specific interfacial area and local gas holdup in a 6-inch high pressure bubble column reactor. The effect of pressure (up to 1.0 MPa), superficial gas velocity (up to 60 cm/s), and sparger have been studied.¹

- The four-point optical probe for application at high pressure (up to 10 MPa) was manufactured in our laboratory. The implementation of such probe in a 1 liter stirred autoclave and needed modification of the developed algorithm for data processing in the stirred tank have been initiated.

Figure 4. Schematic Diagram of the Application of the Probe in a Bubble Column
Experiments have begun with n-decane/CO$_2$ for probe evaluation and preliminary results are presented in Figures 6 and 7.

The data in Figure 6 displays spikes which represent bubbles striking the probe tip, hence demonstrating a two-phase system. The lack of spikes in the data in Figure 7 indicates that bubbles are no longer present in the system, hence demonstrating a single-phase system.

The bubbles at the conditions indicated in Figure 6 are of such small size that they only strike one probe tip and the data processing algorithm is unable to determine the bubble properties. Work is currently underway to identify the window of operation at which bubble properties can be measured by the probe. One potential solution to measure such small size of bubbles is to use plastic optical fibers which can be manufactured in smaller sizes and would allow for the probe tips to be located closer to one another.

Figure 5. Flow diagram of the application of the four-point optical probe in an autoclave stirred tank reactor for an expanded solvent/CO$_2$ system.
Figure 6. Probe response for decane/CO$_2$ at 43 °C and ~1000 psi

Figure 7. Probe response for decane/CO$_2$ at 32 °C and ~1050 psi
Milestones

- Report the transition point from two phases to single phase of the expanded solvent system of CO$_2$ and n-decane.
- For validation, compare results of two expanded solvent/CO$_2$ systems with previous experimental and computational studies of phase transitions and mixture critical conditions.
- Once the probe is validated, perform studies using selected solvents from the test bed systems of the Center for Environmentally Beneficial Catalysis (CEBC).
- At the conditions of the two phase system, report the bubble dynamics under high pressure (>1 MPa).

Summary

- A four point optical probe is proposed for determining bubble chord length distribution, bubble velocity distribution, specific interfacial area, and local gas holdup at high pressures in an autoclave reactor.
- This probe has shown to be capable of detecting the transition from multi-phase to single phase as operating conditions are varied.

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References