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Investigation of emulsification in static mixers by optical measurement techniques using refractive index matching

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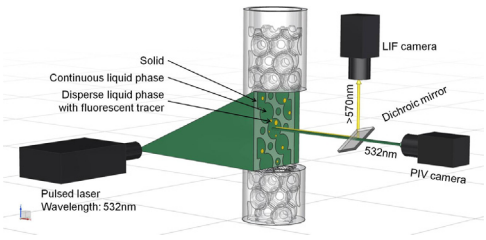
HIGHLIGHTS

- Two immiscible fluids with the same RI as porous structure produced by rapid prototyping.
- A setup for simultaneous PIV and LIF in two-phase flow through porous structures.
- Dependence of droplet size on flowrate, volumetric transport fraction and position in the bed.
- The porous structure with larger changes in the free cross-section produces smaller droplets.

GRAPHICAL ABSTRACT



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ABSTRACT

This study compares a two-phase flow through a foam-like porous structure and a Sulzer SMX^{TM} static mixing element. Refractive index matching between two immiscible fluids and the internal structures enables optical measurements to be performed. The droplet size and position within the internal structures were observed by laser-induced fluorescence. The results show that at low flowrates, droplets follow preferred paths, whereas at higher flowrates, they are more homogeneously distributed within the structures. The droplet size distribution was found to be well represented by the Sauter mean diameter. Measuring along the axis of the two static mixers, we found that droplets disintegrate more quickly in the foam-like porous structure. As both geometries have the same porosity and hydraulic diameter, we conclude that the change in the free cross section is also an important parameter. We observed that in the geometry with large changes in the free cross section, the droplets are smaller.

1. Introduction

Contact between two fluids and a solid is required for heterogeneous catalysis with a two-phase educt (e.g., hydrogenation, oxidation, and hydroxylation). An extensive review of the reactions that are frequently encountered in the pharmaceutical and fine-chemicals industries is provided in Mills and Chaudhari (1997). These reactions are conventionally conducted in batch vessels that are used as multipurpose plants. However, many alternative reactors are available (Stitt, 2002). A continuous tubular reactor with a porous structure is frequently proposed as an alternative (e.g., Stankiewicz and Moulijn, 2004). It enables new chemical routes by providing good heat-/mass-transfer and improved safety. In addition, the continuous operation mode allows heat integration and therefore reduces energy consumption. The internal structures in tubular reactors can be used as a catalyst support and as static mixing elements. In multiphase reactions, these structures can also serve to produce small droplets. In this study, we investigate a two-phase flow through a foam-like porous structure and a Sulzer SMX^{TM} static mixing element.

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Studies of multiphase flow through porous structures can be macroscopic (system scale) or microscopic (pore scale). Many studies on multiphase flows in porous structures have considered macroscopic quantities such as the pressure drop, hold-up, mass transfer, residence time distribution, or interfacial area density (Edouard et al., 2008; Losey et al., 2001; Molga and Westerterp, 2012). These macroscopic quantities are strongly affected by the microscopic composition of the two-phase flow. Therefore, to understand the transport processes on a macroscopic scale, the microscopic effects need to be investigated. In many microscopic studies of two-phase flows in porous structures, the droplet size was larger than the pore size (Ovdat and Berkowitz, 2006; Darnault et al., 1998; Ng et al., 1978; Krummel et al., 2013; Montemagno and Gray, 1995; Kong et al., 2011; Stöhr et al., 2003). Such situations are encountered in oil recovery, for example. In this case, a single-phase flow can occur at the pore scale even though a two-phase flow occurs at the macroscopic scale.

In a tubular reactor, it could be beneficial for the droplets to be smaller than the pores as this would lead to more homogeneous reaction conditions within the reactor. Droplets smaller than the pores are also encountered in emulsification processes using static mixing elements. Owing to the limited access to measurement equipment, the droplet size is frequently measured at the exit of a static mixing element (Al Taweel and Walker, 1983; Fradette et al., 2007). Alternatively, it is measured offline. By microencapsulation via an interface polymerization reaction, the droplet size of an emulsion can be preserved for later analysis by optical microscopy or laser diffraction (Legrand et al., 2001; Lemenand et al., 2003; Theron and Le Sauce, 2011).

In the present study, we investigate a two-phase flow inside a porous structure by optical measurements. This is enabled by matching the refractive index (RI) of the two liquids and the solid. Below, we review optical measurement techniques for multiphase flows.

1.1. Optical measurement techniques

Many tomographic methods including X-ray radiography, neutron transmission tomography, nuclear magnetic resonance imaging, electrical capacitance tomography, and optical methods have been applied for visualizing the phase distribution (Chaouki et al., 1997). In the present study, we use an optical measurement technique with RI matching. Below, we review optical measurement techniques with a focus on studies that employed RI matching. We start with two-phase flows in ducts, continue to single-phase flows in porous structures, and end with two-phase flows in porous structures.

1.1.1. Two-phase flow in ducts

For phenomenological studies such as column flooding (Stemmet et al., 2005) or phase inversion (Piela et al., 2006), a standard camera can be used without RI matching. For quantitative measurements by laser-induced fluorescence (LIF) and particle image velocimetry (PIV) a system consisting of an illuminating laser and a camera is needed. When used for investigating multiphase flows, the main challenge faced with these optical measurement techniques is the refraction of light at phase boundaries. Some authors have applied optical measurement techniques without RI matching (Unadkat et al., 2009; Lindken et al., 1999; Fujiwara et al., 2004). In this case, a laser-based measurement technique is frequently combined with shadowgraphy. The limitation of the shadowgraphy method is that it is only applicable to two-phase flows in a bubbly flow regime with low hold-up of the dispersed phase. This limitation can be overcome by matching the RI of the disperse phase and the continuous phase.

RI matching was applied by Svensson and Rasmuson (2006) for two-phase flow in stirred tanks. RI-matched two-phase flow through ducts was investigated by LIF (Liu et al., 2005) and PIV (Conan et al., 2007). Augier et al. (2003) applied simultaneous PIV and LIF measurements in RI matched liquid/liquid flow through a duct. The images of the particles and fluorescent dye were separated based on their gray values. This allowed simultaneous PIV and LIF with only one camera. Similarly, Morgan et al. (2013) investigated RI-matched liquid/liquid flow in a duct. They simultaneously measured the droplet size by LIF and the velocity field by PIV/PTV.

1.1.2. Single-phase flow in porous structures

RI matching between a solid and a liquid allows optical measurements inside porous structures. Budwig (1994) reviewed RI matching techniques for a single-phase flow. Wiederseiner et al. (2011) recently reviewed RI matching for particle suspensions. These two reports provide an overview of fluid recipes corresponding solid materials for RI matching. Whereas many studies have focused on RI matching between one fluid and one solid, few have focused on using RI matching for two-phase flow in porous structures.

1.1.3. Two-phase flow in porous structures

Ng et al. (1978) investigated liquid/liquid two-phase flow through a porous structure by RI matching of the wetting liquid and the solid. The motion of single droplets was observed by viewing the setup from two perpendicular directions. Kong et al. (2011) investigated gas/liquid two-phase flow inside a porous structure by RI matching of the continuous liquid phase and the solid. A moving laser sheet allowed an incremental 3D scan of the gas phase, in turn allowing a 3D reconstruction of the distribution of the gas phase.

Few studies have employed RI matching between two fluids and a solid. Burdett et al. (1981) was the first to match the RI of two fluids and a solid for optical measurements. Silicone rubber with $n=1.422$ was used owing to its low RI. This allowed the use of methylcyclohexane and a 67.9% w/w glycerine/water mixture. The mean hold-up along a line was estimated using a light absorption technique. Montemagno and Gray (1995) were the first to apply RI matching for investigating two-phase pore scale flow. Two aqueous and two nonaqueous liquids were used in a random packing of fused silica ($n=1.46$). Tracer dyes were added to both phases. The aqueous phase was doped with a dye that congregates along the fluid–fluid interface. Plane illumination by a coherent laser enabled identification of the two phases. A 3D scan of the two fluids in the porous structures was obtained by using precision translators for the optical measurement equipment. Alternatively, Stöhr et al. (2003) measured the 3D field by the translation of the porous structure. They used silicone oil and a zinc chloride solution as RI matching fluids for fused silica. Two optical filters were mounted on a PC-controlled filter wheel to allow separate LIF measurements of the phases with one camera. Ovdat and Berkowitz (2006) obtained similar measurements from polymethyl methacrylate (PMMA). The phases were distinguished using a fluorescent dye in the disperse phase. Finally, Krummel et al. (2013) used two fluids that closely match the RI of glass beads to measure the 3D phase distribution. This was achieved using a confocal microscope that (slice-by-slice) observed the fluorescence of a tracer in the wetting continuous liquid phase.

In comparison to the abovementioned studies that used particles with random packing, the porous structure investigated in our study has a defined geometry that is manufactured by rapid prototyping. This enables optical measurement techniques for two-phase flow in designed porous structures.