Measuring Droplet Sizes Generated by 3D-Printed Stirrers in a Lean Gas–Liquid–Liquid System Using Borescopy

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ABSTRACT: Photo-optical probes with automated image analysis are among the most promising measurement techniques for gas–liquid–liquid systems, but the literature on the application in lean water-in-oil dispersions is scarce. Taking water-in-1-octene as a model system, adhering droplets that severely cloud the images are successfully prevented using a polymeric disc made of polycarbonate or fluorinated ethylene propylene. Sharp bright-field imaging is enabled using a neither fully diffuse nor specular reflection pane. Various 3D-printed stirrers are characterized by their energy input, including gas-inducing modifications. Gas induction impairs the energy input of the stirrer and the imaging of droplets. Nevertheless, the measurement technique reliably provides droplet size distributions that exclude gas bubbles. Axial stirrers are preferable if one position at high stirring speeds is measured, as the homogeneity of droplet sizes along the reactor height is promoted. Radial stirrers are preferable if the stirring speed is varied, as the same trend of the droplets sizes is measurable along the reactor height. For radial stirring, the most beneficial position of the probe is close or slightly above the stirrer. The use of borescopy is thus found to be feasible if adhering droplets can be prevented and vision on droplets is not obstructed by too many bubbles.

INTRODUCTION

Multiphase systems are common within process engineering and its related disciplines, with the applications including multiphase reaction systems, most separation processes, biotechnology, and wastewater treatment. While the phase boundaries play a significant role in transport phenomena and hydrodynamics of these multiphase systems and can have a large impact on product quality, the measurement of particle size distributions is not a well-established standard procedure, especially when the size distribution in question is transient.1

Such transient particle size distributions appear in coalescing systems such as gas–liquid–liquid–liquid (GLL), gas–liquid–solid, or gas–liquid–liquid (GLL) systems. In comparison, the complex GLL systems are rarely addressed in the engineering-related literature, making valuable data scarce.2

The measurement of transient particle size distributions is challenging. The unsteady nature of the interfacial area in these systems makes an in situ measurement necessary to avoid distortion of the data by sampling.3 When multiple dispersed phases are present, including unwanted or trapped gas bubbles in LL systems, the measurement technique must be able to distinguish the present particles to avoid a distortion of the data. The literature suggests that the use of photo-optical probes in conjunction with automated image analysis is among the most promising measurement techniques for transient particle size distributions, as it is more precise than laser-based and acoustic methods and is oftentimes used as a reference for the calibration of these.1,4–10 Image analysis offers the possibility to differentiate particles based on optical characteristics such as the difference in refractive indices or opaqueness.

Table 1 gives an overview of applications of photo-optical probes in the analysis of LL and GLL systems. Most applications focus on systems in which water is the continuous phase and not the dispersed phase. In addition, the droplet and bubble size distributions are oftentimes influenced by additives such as surfactants or nanoparticles. Thus, the literature on the application in lean water-in-oil dispersions is scarce. Applications that include a gas phase are mostly reduced to the gas phase being air at atmospheric pressure and thus the influence of pressure as well as the effect of dissolved gases such as hydrogen or carbon monoxide is not covered. It is further known that gas induction has a negative effect on mixing—regardless of whether it was achieved by vortex formation or aeration—reducing the energy input of the stirrer significantly.11–13 This is rarely addressed, especially if both GL and LL mixing is important at the same time. In addition,
the inhomogeneity within the vessel of the droplet sizes of lean surfactant-free systems is rarely addressed and the probe is mostly placed close to the stirrer only. To our understanding, it is important to clarify the feasibility of using a photo-optical probe in these challenging GLL systems so that it can be implemented in future studies as a tool that allows deeper insights into the mechanisms at hand.

An emerging field of LL and GLL systems is water-based recycling of homogeneous catalysts. One of the most prominent and industrially relevant uses of homogeneous catalysis is the Ruhrchemie/Rhône-Poulenc process, an aqueous biphasic reaction system. However, the underlying hydroformylation reaction is limited to short-chained olefins as the decreased solubility of higher olefins is detrimental to the reaction rates. In an effort to increase the reaction rates of longer-chained olefins, various strategies have been deployed that make use of additives to influence the physical behavior of the multiphase system. These strategies, however, come with their own set of challenges such as increased catalyst leaching, difficult separation, or high prices. The modification of the physical behavior in these strategies precedes the understanding of the fundamental physical behavior of the lean system and its influence on the chemistry itself. It is not fully clear where the reaction takes place, although the interfacial area between the two liquid phases is regarded as the most probable location. Due to the challenging nature of lean GLL systems discussed above, to our knowledge, the interfacial area and its influence on the lean aqueous biphasic hydroformylation of long-chained olefins has not been quantified so far. Therefore, a model system of 1-octene and water is used in this work based on the interesting hydroformylation reaction.

The goal of this work is thus to find and evaluate a testing setup to measure and follow the trend of the droplet size distribution (DSD) in the presence of bubbles for lean multiphasic systems such as the ones encountered in biphasic catalysis using high-pressure borescopy with automated image analysis. For this, it is targeted to selectively measure the trend of the organic/aqueous interfacial area in the additive-free aqueous biphasic hydroformylation model system consisting of 1-octene, water, and synthesis gas at temperatures up to 100 °C and pressures up to 80 bar in a stirred tank reactor. Furthermore, this work aims to quantify which flow patterns and stirrer geometries in a stirred tank benefit the photovoltaic measurement, using 3D-printing to manufacture gas-inducing modifications of stirrers. The evaluation of this measurement technique will allow the quantification of the influence of the fundamental physical behavior of the mixing in a lean GLL system, which will give important insights on which new promising strategies can be based on.

### METHODS

**Borescopy.** High-pressure capable borescopy in conjunction with an automated image evaluation software both provided by SOPAT GmbH is used for the measurement of liquid droplets and the subtraction of bubbles. The used technology is commercially available and its principle for particle detection and/or exclusion is discussed in detail by the works of Maaß et al. and Panckow et al. The automated image analysis needs to be trained to the studied system, which is a service provided by SOPAT GmbH. An exemplary raw

| continuous phase | dispersed phase(s) | dispersed fraction [vol %] | equipment | probe position | refs |
|------------------|--------------------|---------------------------|-----------|----------------|-----|
| water            | kerosene           | 1.3–78.05                 | mixer-settler | close to stirrer | 1   |
| water            | toluene, air       | 10–50                     | static mixer | after the mixer | 1   |
| water            | styrene, poly styrene, air | 40                  | stirred tank | close to stirrer | 17  |
| water            | various organic substrates with polyvinyl alcohol | 1–45                   | stirred tank | close to stirrer | 17  |
| water            | various organic substrates with polyvinyl alcohol | 20–90                  | stirred tank | not stated      | 18  |
| water            | various organic substrates, oxygen | 1–100                | stirred tank | half of liquid level | 19  |
| water            | n-butanol, Shellsol-T | 10                   | Taylor-Couette disc contactor | middle of column height | 19  |
| water            | hexadecane, air    | 10                       | stirred tank | close to stirrer | 22  |
| water            | 1-dodecene with non-ionic surfactants, micellar phase | diverse               | stirred tank | close to stirrer | 23  |
| water            | water or nitric acid solution with surfactant | 2–15                  | pulsed column | middle of column height | 24  |
| various organic substrates with hydrophobic silica nanoparticles | water | 22–80 | mixer-membrane | before and after filtration | 25  |
| water            | microorganisms, hexadecane, air | 10 (hexadecane) | stirred bioreactor | not stated | 26  |
| water            | silicon or paraffin oil | 1–5                   | plate droplet separator | in- and outlet | 26  |
| water            | water              | 23                       | stirred tank | close to stirrer | 27  |
| Esssol D60 with red dye | water with 3.2 wt % NaCl | 30–70              | parallel pipe oil-water separator | separator inlet | 28  |
| water            | n-heptane with silica nanoparticles | 15–85 wt % | stirred tank | close to stirrer | 29  |
| various aqueous solutions | kerosene with additives | 1–24               | mixer-settler | close to stirrer | 30  |
| water with surfactant | hexadecane with dye | 10                   | stirred tank | close to stirrer | 31  |
| water            | isododecane        | <40                      | extraction column | not stated | 32, 33 |

*Based on Emmerich et al. and extended.*

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**Table 1. Overview of Applications of Intrusive Photo-optical Image Acquisition Together with Automated Image Analysis for (Gas–)Liquid–Liquid Systems**

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image with the respective evaluated image is given in Figure 1. The algorithm is set to first detect all spherical objects on the image, after which the particles are classified into bubbles and droplets. The bubbles are then subtracted from the particle size distribution to achieve a pure DSD. A comparison to a manual image evaluation verified the performance of the algorithm in measuring the Sauter mean diameter of the droplet swarm. It can be found in the Supporting Information in Table S1 and Figure S14.

For the image acquisition, the photo-optical probe SOPAT VI Sc is used. Preliminary experiments were conducted using the SOPAT VI Pa to find that the increased zoom level of the SOPAT VI Sc is necessary to detect smaller droplets. The fields of view are compared in Figure 2. The used probe has a width of 3/4” and a maximum working temperature of 130 °C. It is installed from the top of the reactor using the reversible process connection Spectite PF supplied by TC Mess- und Regeltechnik GmbH. This allows the variation of the immersion depth of the probe. The field of view of the PN250 SOPAT VI Sc has a diameter of 2550 μm, while that of the PN10 SOPAT VI Pa has 5900 μm. The images can be taken with a resolution of 5 MPixel at a maximum of 15 Hz.

Behind the probe’s window, a stroboscope acts as the light source for image acquisition. The light rays provided by the stroboscope pass the studied fluid and are reflected by a reflection pane that is mounted on the tip of the probe. This results in bright-field imaging which is required to measure small droplets. The gap between the window and reflection pane is named the measuring gap and its height is set, unless noted otherwise, to 3 mm. This height was chosen based on preliminary experiments so that most bubbles are excluded from entering the gap, which would otherwise obstruct the vision on droplets. To improve the image acquisition, an array of transparent plastics is tested in this work as discussed in the Results and Discussion. The considered materials are polycarbonate (PC) provided by APEX plastic solutions GmbH, polyvinylchloride, and polystyrene both supplied by Herrmann, Papenfuß, Ziebold GbR, and fluorinated ethylene propylene (FEP) provided by Holscot Europe. Circular panes of 0.5 mm in thickness are mounted in front of the sapphire glass using either PTFE tape or a custom-built reflection pane. Holes are drilled into the panes to alleviate pressure gradients that may damage or deform the panes under reaction conditions.

The image analysis includes the possibility to classify particles such as, in this work, droplets and bubbles. The bubbles are excluded from the measurement, and only the DSD is measured. Following eq 1, the Sauter mean diameter \( d_{32} \) can be derived from the DSD, wherein \( V_d \) denotes the total volume and \( A_d \) the total surface area of the dispersed droplets. The Sauter mean diameter is defined as the diameter of spheres of a theoretical monodisperse particle swarm that has the same ratio of volume to surface area as the original swarm.

\[
d_{32} = \frac{6 \cdot V_d}{A_d} \tag{1}
\]

\[
a_{LL} = \frac{A_d}{V_{total}} = \frac{A_d \cdot \phi_d}{V_d} = \frac{6 \cdot \phi_d}{d_{32}} \tag{2}
\]

Data on \( d_{32} \) are often correlated with the Weber number \( W_e \), which relates the kinetic energy of the droplet to its interfacial energy, as shown in eq 3. For systems in which coalescence is not inhibited or neglected, the literature typically suggests the form shown in eq 4. Herein, \( C_1 \) depends on the used stirrer and \( C_2 \) quantifies the influence of coalescence. For the exponent of \( W_e C_3 \), the literature further suggests a value of 0.6 for breakage-dominated dispersions, which is common in diluted dispersions, and a value of 0.375 for coalescence-dominated dispersions.

\[
W_e = \frac{\rho n^2 D^3}{\sigma} \tag{3}
\]

\[
d_{32} \frac{D}{C} = C_1(1 + C_2 \phi_d) W_e^{-C_3} \tag{4}
\]
While the baffle holder was printed in PEEK, the stirrers were printed in polylactide (PLA) as the high durability and chemical corrosion resistance of PEEK was not required for the experiments conducted in this work. The used model of the 3D printer further lacks a heated chamber that is required to minimize warping when printing PEEK. The PEEK filament was provided by Intamsys Technology Co. Ltd., and the PLA filament is the EasyPrint PLA provided by PrimaCreator. For the parts printed in PEEK, a BuildTak polyether imide printing surface manufactured by Ideal Jacobs Corp was used. PLA was printed on a borosilicate glass plate. The 3D models of the printed parts were created using a CAD software. Technical drawings of the printed stirrers are provided in the Supporting Information in Figures S1–S7.

**Studied Stirrer Types.** In this work, a six-blade RT and a four-blade 45° pitched blade impeller (PB) both 1/3 of the tank diameter in width (33 mm) are used for radial and axial mixing, respectively. An overview of the studied stirrer geometries is given in Table 2. The PB was printed in two versions, one pumping the fluid upward (PBup) and one downward (PBdown). An advantage of additive manufacturing is the ability to 3D-print internal structures which are more challenging to obtain using conventional manufacturing techniques. This allows for internal shafts that can be beneficial for gas induction. Both the RT as well as the PBup were modified for gas induction and combined with a hollow stirrer shaft that is perforated above the liquid level. The stirrers are connected to the shaft with an M6 thread. The gas induction is achieved by angling the surfaces of the blades’ tips to 45° and connecting them to a system of internal shafts, as shown in the Supporting Information in Figures S3–S6. The pressure drop that occurs at the blades’ tips drags in the gas phase at sufficiently high rotational speeds.

RTgas was further studied with a larger diameter of 40 mm (RTgasL). A mirrored design of RTgas with (RTgasM) and without (RTpitched) the internal shafts was used to study the effect of the direction of rotation as well as the effect of the pitched tips of the blades.

### EXPERIMENTAL SETUP

The experiments were conducted in a custom-built 730 mL stirred tank reactor. A technical drawing of the reactor can be found in the Supporting Information in Figures S8 and S9. The vessel has a diameter and a height of 100 mm. A 3D-printed baffle holder is used to hold four stainless-steel blades 10 mm in width that act as baffles. The reactor facilitates a dished bottom to aid with emptying. In each experiment, 500 mL of the liquid phase was used, resulting in a liquid height to diameter ratio that was measured to be 0.75.

As mentioned previously, a hollow stirrer shaft was used to enable gas induction. The stirrer shaft is 6 mm in diameter and is connected to the magnetic coupling bmd 075 manufactured by Büchi AG. In the magnetic coupling, plain bearings made of PEEK are used. For agitation, the ViscoPakt-rheo-X7 manufactured by HiTec Zang GmbH was used that enables the measurement of the stirring torque with an accuracy of ±0.5 Nmm and thereby the calculation of the energy dissipation rate of mixing following eqs 5 and 6, wherein \( \epsilon \) denotes the energy dissipation rate, \( P \) the power input, \( n \) the rotational speed, and \( \tau \) the torque.

\[
P = 2\pi \cdot n \cdot \tau \tag{5}
\]

\[
\epsilon = \frac{P}{V_{\text{total}}} \tag{6}
\]

The measurement of the power input further enables the calculation of the Newton number \( Ne \) as a function of the Reynolds number \( Re \) in accordance with eqs 7 and 8, which is used to characterize the printed stirrers. In these equations, \( \rho \) denotes the density of the liquid, \( D \) the diameter of the stirrer, and \( \eta \) the dynamic viscosity of the liquid.


\[ Ne = \frac{P}{\rho n^2 D^5} \]  

(7)

\[ Re = \frac{\rho n D^2}{\eta} \]  

(8)

The \( Ne \) tabulated in the literature for various stirrer types under turbulent conditions generally does not account for deviations in a stirrer’s geometry or in the experimental setup. An estimation of the energy input of the 3D-printed stirrers that also includes gas-inducing variants in the presence of a borescope is therefore difficult. The 3D-printed stirrers are instead characterized by their energy input by measuring the stirring torque and calculating \( Ne \) over a varying \( Re \). The experimental setup differs from typical standard testing conditions in three ways:

1. A dished bottom is used.
2. The liquid height to diameter ratio is 0.75.
3. There are reactor internals present such as the 3/4” optical probe.

The experimental setup further enables the variation of both temperature and pressure. A piping and instrumentation diagram can be found in the Supporting Information in Figure S10. For heating, a KM-HJ-450 heating jacket in conjunction with the KM-RD3011 PID temperature controller both manufactured by SAF Wärmetechnik GmbH were used. A Pt100 temperature sensor in the reactor is used for the temperature measurement. The pressure control is facilitated using the pressure regulator PR-57 manufactured by GO Regulator. Two mass flow controllers allow the continuous feed of synthesis gas at a variable ratio. In this work, the ratio is set to 1:1. For hydrogen, the MINI CORI-FLOW ML120V00 and for carbon monoxide, the MINI CORI-FLOW M13 both supplied by Bronkhorst Deutschland Nord GmbH are used.

**Chemicals.** The olefin used in this work is 99+\% pure 1-octene supplied by ACROS Organics. Ultrapure water was procured using the MilliQ water purification system supplied by Merck KGaA. Carbon monoxide with a purity of 99.997\% and hydrogen with a purity of 99.999\% were supplied by Westfalen AG. Density, kinematic viscosity, and interfacial tension of the water—1-octene system are shown in Table 3.

**RESULTS AND DISCUSSION**

The goal of this work is to find and optimize a suitable testing setup to measure the DSD in a lean water-in-oil dispersion in the presence of gas bubbles. As two particle classes are present, namely, droplets and bubbles, the used measuring technique has to be able to both detect and distinguish these classes. Due to the fast rate of coalescence and demixing of the studied system, the measurement must take place in situ. For the application in the model system of aqueous biphasic hydroformylation performed in a stirred tank reactor, some challenges need to be overcome to find an optimal testing setup. Firstly, most applications of borescopy to measure droplet sizes use either oil-in-water systems or water-in-oil systems with additives. The challenge that arises from water not being the continuous phase is its high interfacial energy and its polarity. Aqueous droplets are more prone to coalesce and adhere on untreated glass surfaces than most organic solvents, clouding the images. Furthermore, the contrast of the LL and gas—liquid interfaces on the images is tunable by modifying the illumination. Secondly, the stirred tank reactor allows for a simple manipulation of the degree of mixing by varying the stirrer speed. The mixing conditions in the reactor are additionally tunable by changing the geometry of the stirrer. This also affects the fraction of particles that enter the measuring gap. For stirrers that introduce more bubbles into the system, which is relevant for a GLL system, the synergy with a measurement technique that aims to measure droplets is to be evaluated. Thirdly, the stirred tank reactor is, depending on installed stirrer type and stirring speed, inhomogeneously mixed. It is therefore necessary to evaluate the validity of trends measured with a point measurement for the rest of the reactor.

**Image Acquisition.** The probe window, which is glued on the tip of the probe, consists of sapphire glass. It seals and protects the probe against pressure. In the studied GLL system, adhering droplets occur that cloud the image acquisition (see Figure 3). To minimize the adhering of aqueous droplets, an array of readily available, colorless, and transparent plastics with increased hydrophobicity in comparison with sapphire glass are tested as an additional protection layer. In addition to the aforementioned attributes, the materials are required to be resistant to the studied chemicals and future process conditions encountered in aqueous biphasic hydroformylation of 1-octene, which are at least 100 °C, synthesis gas pressure of up to at least 80 bar, and the presence of the product nonanal. The first considered material is PC which is mounted in front of the sapphire glass window using PTFE tape. While PC offered sufficient protection from adhered aqueous droplets at room temperature, it is not resistant to water at 100 °C. Of the further studied materials polyvinylchloride, polystyrene, and FEP, which were mounted in front of the sapphire glass using a custom-built reflection pane, as shown in Figure 3, only FEP withstands the operating conditions of the model reaction system. FEP is chemically similar to PTFE concerning composition and corrosion resistance. Unlike PTFE, however, it is transparent. Images of the materials after the contacting as well as a technical drawing of the custom-built reflection pane are shown in the Supporting Information in Figures S11 and S12.

For the imaging of the droplets, the reflection pane may reflect the stroboscope’s light specularly, diffusely, or in between. In the images acquired using PTFE as a diffuse reflector, a sectional plane is visible and particles behind this plane are hidden. As the focal plane of the optics can be moved to this sectional plane, diffuse reflection provides distinct and sharp bright-field images. However, as the number of passes of the light rays through the phase boundaries is reduced, the boundaries of droplets become less distinct. This effect is more pronounced if the borescope is further zoomed in (SOPAT VI Sc). Therefore, while taking the possibility of an exchangeable PC or FEP disc into account, a custom-built reflection pane using sanded 1.4571 stainless steel as the reflective material is built in accordance with Figures 3 and 4. The custom-built pane provides reflection that has a larger specular component than the PTFE that provides a purely diffuse reflection. As it can be seen on Figure 4, this enhances the contrast of the

| substance | density \( \rho \) [kg m\(^{-3}\)] | dynamic viscosity \( \eta \) [mPa s] | interfacial tension \( \sigma \) [mN m\(^{-1}\)] |
|-----------|-------------------------------|-----------------|-------------------|
| water     | 997.0                         | 0.90            | 46.6              |
| 1-octene  | 710.6                         | 0.45            |                   |

Table 3. Physical Properties of Water and 1-Octene at 25 °C
phase boundaries while retaining the positive characteristics discussed before. However, the measuring gap height cannot be varied with this design and is set to 3 mm.

The custom-built reflection pane is more restrictive to the streamlines of the flow compared to the installation using PTFE tape to fix the polymer disc. This hampers the flushing of the surface of the disc so that adhering droplets are observed when PC is used. This does not occur when using FEP. It is therefore concluded that FEP is more effective at preventing the adhering of aqueous droplets than PC which requires a sufficient flushing.

The aforementioned findings result in two setups used in this work. In setup A, the polymeric disc made of PC is installed using PTFE tape to fix the polymer disc. This hampers the flushing of the surface of the disc so that adhering droplets are observed when PC is used. This does not occur when using FEP. It is therefore concluded that FEP is more effective at preventing the adhering of aqueous droplets than PC which requires a sufficient flushing.

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Choice of Stirrer and Flow Pattern. With the two available setups that successfully prevent adhering droplets, the influence of stirrer geometry, stirring speed, and gas induction can be evaluated. Varying the stirrer geometry has an influence on both the flow pattern and the energy input for mixing. While the flow pattern is apparent from the used standard geometries for radial and axial mixing, the energy input is not. Characterizing the 3D-printed stirrers in this regard allows, other than comparing their mixing performance with conventionally manufactured stirrers, a better understanding of the results obtained with the borescope. This is crucial for a GLL system as gas induction greatly decreases the amount of dissipated energy. The findings of the characterization are therefore associated with the results of the borescopic measurements that follow.

A general approach to characterize stirrers is by plotting $N_e$ over $Re$. $N_e$ generally becomes constant at sufficiently high levels of turbulence around the stirrer, that is, for $Re$ over $10^4$, as long as gas induction through vortex formation is prevented by the setup. An example of the results using the RT, as well as the dead torque, that is, without a stirrer, over the course of an experiment is given in the Supporting Information in Figure S13. $N_e$ is calculated from the averaged stirring torque of the last minute of each step.

Figure 5 compares the $N_e$ over $Re$ plots for the studied radial and axial stirrers. For the system studied in this work, both the RT and RTpitched show a constant $N_e$ until an $Re$ of around 40,000, after which a sharp decrease occurs. This decrease is attributed to gas induction by gas vortex formation and is already reported in the literature. In this range, RT averages to 6.55 for RT and 5.87 for RTpitched, which is in agreement with the textbook value for the RT of 5.5. With the exclusion of the lowest studied $Re$, the $N_e$ of PBup remains around an average of 2.12, which is again in agreement with the textbook value for the PB of 2. PBDown starts by following a similar trend to PBup until Re over 40,000, after which a downward trend similar to RT and RTpitched occurs that is again accompanied by gas induction. As expected from the literature reports, the gas-inducing variants RTgas, RTgasL, and PBupgas show a decreased $N_e$ compared to their nongassing counterparts once the rotational speed is sufficiently high to initiate the gas induction. This is marked by a distinct drop in $N_e$. For the studied gas-inducing geometries, this happens at...
1250 min⁻¹. RTgasM, on the other hand, closely follows the trend of the geometrically similar RTpitched, owing to a reduced gas induction. RTgasL shows an unexpectedly low Ne that is on a level with PBup. However, the increase in gas induction of RTgasL is negligible.

Table 4. Performance of Borescopic Measurement for Varying Flow Pattern and Gas Induction

| Stirrer   | n [min⁻¹] | $d_{32}$ [μm] | Recognized droplets | Detections per image | Comments                                                                                                                                 |
|-----------|-----------|---------------|---------------------|----------------------|------------------------------------------------------------------------------------------------------------------------------------------|
| RT        | 1500      | 391.94        | 1620                | 2.7                  | At 1500 rpm, large non-spherical droplets were not detected                                                                                |
|           | 1700      | 415.82        | 2187                | 3.6                  |                                                                                                                                              |
|           | 2000      | 404.76        | 2258                | 3.8                  |                                                                                                                                              |
| RTgas     | 1500      | 594.29        | 802                 | 1.3                  | Adhering droplets, increased gas induction, bubbles obstruct vision on droplets                                                          |
|           | 1700      | 606.81        | 917                 | 1.5                  |                                                                                                                                              |
|           | 2000      | 567.77        | 1144                | 1.9                  |                                                                                                                                              |
| RTgasL    | 1500      | 417.75        | 1244                | 2.1                  | Poorer dispersion in comparison to RT while no increase in gas induction is observable                                                   |
|           | 1700      | 505.15        | 1335                | 2.2                  |                                                                                                                                              |
|           | 2000      | 434.80        | 1936                | 3.2                  |                                                                                                                                              |
| RTgasM    | 1500      | 440.68        | 1703                | 2.8                  | Large bubbles present in comparison to RT; Small portion of bubbles detected as droplets; Scale of recognized bubbles similar to large droplets |
|           | 1700      | 444.73        | 1812                | 3.0                  |                                                                                                                                              |
|           | 2000      | 443.78        | 2136                | 3.6                  |                                                                                                                                              |
| RTpitched | 1500      | 445.55        | 2038                | 3.4                  |                                                                                                                                              |
|           | 1700      | 436.73        | 2420                | 4.0                  |                                                                                                                                              |
|           | 2000      | 420.81        | 2864                | 4.8                  |                                                                                                                                              |
| PBup      | 1500      | 502.59        | 1925                | 3.2                  | Presence of oil-in-water-in-oil double emulsion                                                                                             |
|           | 1700      | 468.63        | 2645                | 4.4                  |                                                                                                                                              |
|           | 2000      | 441.64        | 3696                | 6.2                  |                                                                                                                                              |
| PBdown    | 1500      | 500.46        | 554                 | 0.9                  | Presence of large bubbles; poorly dispersed; many images show no aqueous phase; DSD assumed to be outside of detection range of borescope |
|           | 1700      | 558.34        | 692                 | 1.2                  |                                                                                                                                              |
|           | 2000      | 555.60        | 879                 | 1.5                  |                                                                                                                                              |

“Setup and algorithm not optimized for individual stirrers. Sauter mean diameter $d_{32}$ and number of recognized droplets under ambient conditions on 600 images taken at 3 Hz for varying stirrer speed and geometry with $\phi_d = 5$ vol % using setup A.”

Figure 5. Newton number Ne over Reynolds number Re for modifications of (a) the RT and (b) the pitched blade impeller with the RT for reference. Calculation based on stirring torque measurements.
With the Ne quantified for the various stirrers and for the studied vessel, it is now possible to interpret changes in the measured \(d_{32}\) for varying stirrer speeds in Table 4, especially when gas induction is present. The table further shows the number of droplets that are recognized by the algorithm on the 600 images that are acquired for each data point. For a fixed amount of acquired images and an otherwise similar system, the number of recognized droplets signals the relative difficulty of the measuring task for the image analysis for the various stirrer geometries. To detect a droplet, it has to enter the measuring gap, be of sufficient sphericity, be in line of sight, and not be obstructed by a bubble. It must be noted that a change in droplet count with increasing stirring speed or with a change in stirrer geometry can be attributed to multiple reasons. On the one hand, the relative difficulty of the measuring task is affected. This includes the probability of a droplet to enter the measuring gap or for the droplet to be visually obstructed by a bubble. On the other hand, higher stirring speeds generally correspond to smaller droplets and smaller droplets correspond to a higher number of droplets to be measured.

For the RT, increasing stirring speeds \(n\) increase the number of recognized droplets, while the changes in \(d_{32}\) are regarded as negligible. This is against the expectation that increasing \(n\) leads to smaller droplets. At 1500 min\(^{-1}\), large nonspherical droplets can be seen. These liquid bulks are outside of the measuring range and are not detected by the algorithm. It may be argued that these droplets form a secondary droplet swarm and are generated by droplet coalescence in regions of low turbulence within the reactor. Of all studied geometries, RT generated the smallest \(d_{32}\) at around 400 \(\mu\)m. The gas-inducing stirrer RTgas leads to a significant increase in bubbles. This indicates that the internal structures are printed successfully and the gas holdup is increased over the standard RT. With the RTgas, however, droplets are more prone to adhere on the tip of the probe, leading to faulty detections. Furthermore, the present bubbles obstruct the vision on the dispersed droplets, greatly reducing the number of recognized droplets. The \(d_{32}\) measured with RTgas is the largest of all studied stirrers at around 600 \(\mu\)m. This is explained by less energy being dissipated into the liquid phase due to the increased gas holdup, as evident in its Ne as shown in Figure 5. The gas-inducing stirrer with an increased diameter RTgasL leads to a poorly dispersed system. Unlike RTgas, the amount of bubbles is not visibly increased. This is in line with RTgasL having the lowest Ne of all tested radial stirrers. RTgasM shows a similar trend to RT, albeit the measured \(d_{32}\) being increased by around 40 \(\mu\)m. Compared to RT, large bubbles are visible on the images with some of them being detected as droplets. The wrongly detected bubbles are of a scale similar to other larger dispersed droplets and therefore increase \(d_{32}\). RTpitched shows a decreasing \(d_{32}\) with increasing \(n\), as would be expected for this type of experiment. The measured \(d_{32}\) is of a similar scale to RTgasM. At 2000 min\(^{-1}\), around 20 \(\mu\)m separate RTpitched from RT. The increased \(d_{32}\) over RT is in line with the slightly reduced Ne as seen in Figure 5. The axial stirrer PBup shows a decreasing \(d_{32}\) with increasing \(n\). With the exception of RTgas, the axial stirrer generates a larger \(d_{32}\) than the radial stirrers. The number of recognized droplets is the highest of all studied geometries. The axial flow pattern therefore benefits the homogeneity of the droplet swarm within the reactor and thus the measurement. On the other hand, an oil-in-water-in-oil double emulsion is recognizable with this stirrer type, especially when the dispersed phase fraction is increased (see Figure 6). This occurrence is explained by decreased shear forces within the vessel. Similar to RTgas, the use of PBupgas hampered the droplet detection significantly. On many of the acquired images, no droplets are visible. This leads to the assumption that either the DSD is outside the probe’s detection range or only a small portion of the dispersed droplets enters the measuring gap.

Conclusively, 3D-printed gas-inducing stirrers increase the gas holdup but do not synergize well with a borescopic image acquisition that aims to detect droplets. Gas induction promotes adhering droplets even with the optimized setup. The automated image analysis successfully neglects bubbles in the acquired DSD, with the only exception being RTgasM. With RTgasM, some bubbles were detected as droplets which is explained by the need to tune the detection algorithm for this specific measuring task. Furthermore, bubbles obstruct the vision on droplets, decreasing the number of recognized droplets per image. Thus, when using gas-inducing stirrers, longer measurement times, an increased number of images, and a fine-tuned algorithm is required in comparison with standard stirrers. The gas induction of gas-inducing stirrers lowers the energy dissipation rate for mixing in comparison with standard stirrers, as evident in Figure 5, which increases the droplet sizes. However, as both the generated droplet swarm and the gas induction are a function of stirrer speed, the resulting trend of \(d_{32}\) over increasing stirring speeds is of interest. As it can be seen in Table 4, the measured trend is inconsistent with the trend of standard stirrers. For standard stirrers, the \(d_{32}\) decreases with increasing stirring speeds. This inconsistency in the measurements is, however, explained by the aforementioned challenges when using gas-inducing stirrers and requires further study. Radial stirrers, while having an increased Ne over the axial pitched blade stirrers, generate smaller droplets and therefore larger interfacial areas. Axial stirrers, on the other hand, benefit the borescopic image acquisition.

The results of RTgas may be enhanced by the optimization of the measuring gap height to exclude a portion of the interfering bubbles from entering the gap, as compared in Table 5. Reducing the height leads to a significant decrease in the number of droplets that are recognized on the same number of images and under otherwise the same conditions. This goes in hand with a reduction in the calculated \(d_{32}\) of more than 100 \(\mu\)m. Table 5 also compares the \(d_{90}\) of the respective DSD from which it can be concluded that by minimizing the measuring gap height, a significant portion of

Figure 6. Oil-in-water-in-oil double dispersion as observed with setup B and PBup at 1500 min\(^{-1}\) and \(\varphi_d = 10\ \text{vol}\%\).
is important that the trend of $d_{32}$ measured at one point in the reactor matches the trend of the whole reactor. The latter is of high importance for the biphasic hydroformylation model system to evaluate the influence of the interfacial area at varying stirring speeds. The stirrer with the highest $N_e$, namely, the RT, is used as a basis and is studied in the range of 1250 to 2000 min$^{-1}$. For the other studied stirrers in this chapter, an additional stirring speed is studied that matches the measured torque of the RT at 2000 min$^{-1}$ to accommodate for their lowered energy input per rotational speed, approximated in 250 min$^{-1}$ steps.

In Figure 8, the development of $d_{32}$ for varying stirring speeds and probe positions is shown for measurements using the RT installed at 1/3 and 1/4 of the liquid level, measured from the bottom. With the stirrer installed at 1/3 of the liquid level (25 mm), the smallest $d_{32}$ is measured below the stirrer followed by the measurement at stirrer height. In the studied range of stirring speeds, all probe positions follow a similar downward trend. While the average $d_{32}$ is reduced with increasing stirring speeds, the maximum relative difference of $d_{32}$ along the reactor axis remains unchanged, meaning that the change in the homogeneity of the mixing is negligible for increasing rotational speeds. If the RT is installed at 1/4 of the liquid level (18.75 mm), a downward trend of $d_{32}$ is notable at 15 and 25 mm, with the $d_{32}$ being the smallest at 25 mm. For the probe positions at 40 and 55 mm, on the other hand, the change in $d_{32}$ is negligible. An increase in stirring speed leads to no notable reduction in the overall difference in $d_{32}$ between the various probe positions. As it can be seen in Figure 8, the influence of the stirring speed on the droplet sizes is higher with the stirrer positioned at 1/3 of the liquid level.

The axial stirrers PBup and PBdown are compared in Figure 9. Unlike the radial stirrers, an increase in the stirrer speed reduces the relative differences in $d_{32}$ between the various probe positions and therefore increases the homogeneity of the dispersion. In comparison, the PBup resulted in the closest distribution of $d_{32}$ of all tested stirrers at 2000 min$^{-1}$. With the PBup, a downward trend for $d_{32}$ is measured above the stirrer, while an upward trend is measured at and below the stirrer. For the PBdown, a downward trend is measured at 15 and 55 mm and an upward trend at 25 and 40 mm. At 2750 min$^{-1}$, a high degree of homogeneity of the dispersion is achieved using PBdown.

With the present data on various droplet sizes for varying stirring speeds, it is possible to derive a correlation for the prediction of $d_{32}$ based on $We$ following eq 4. Using the information on power numbers, $C_i$ in eq 4 can be substituted by $C_i N_e^{-\alpha_i}$ to describe the influence of stirrer geometry. This leads, by minimization of the squared errors, to the correlation shown in Figure 10. Herein, the experimental values of RT, PBup, and PBdown are considered at a fixed borescope immersion depth. Following the findings of Figures 8 and 9 for the average trends of $d_{32}$, this depth was set for the RT to be at stirrer height, for PBup to above, and for PBdown to below the stirrer. It is found that for the studied system, $\frac{d_{32}}{D} \sim We^{-0.375}$ describes the data well. Thus, the studied lean water-in-1-octene system is found to be dominated by coalescence.

The difference in droplet sizes for the radial and axial stirrers is found to be small compared to their difference in $N_e$, resulting in $\frac{d_{32}}{D} \sim N_e^{-1/16}$. However, as evident from Figures 8 and 9, the measured droplet sizes within the reactor are inhomogeneous and thus depend significantly on the

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**Table 5. $d_{32}$ and $d_{90}$ and Number of Recognized Droplets with Varying Measuring Gap Width Using RTgas at 2000 min$^{-1}$ and $\phi_d = 5\%$ Using Setup A**

| Gap Width (mm) | $d_{32}$ [μm] | $d_{90}$ [μm] | Recognized Droplets |
|---------------|---------------|---------------|---------------------|
| 1.5           | 444.92        | 241.61        | 425                 |
| 2             | 438.19        | 366.18        | 739                 |
| 3             | 567.77        | 586.71        | 1144                |
| 4             | vision obstructed by bubbles | in Supporting Information | |

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**Figure 7. Illustration of the borescope positions in the reactor.**
immersion depth of the borescope. More advanced models are required to predict the distribution of droplet sizes along the reactor axis. In Figures S19–S24 in the Supporting Information, the DSDs for the RT and PBup for varying stirring speeds and borescope immersion depths are compared for further reference and future validation studies.

When synthesis gas pressure is applied to the system using the RT, the difference in $d_{32}$ across the height of the reactor is decreased up until 20 bar (see Figure 11a). Furthermore, in comparison with the experiment with argon at atmospheric pressure (0 bar in Figure 11a), the measured $d_{32}$ is decreased. It is argued that this can be linked to the dissolved gases influencing the physical properties such as the viscosity and density of the continuous phase, promoting a more homogeneous dispersion of the aqueous phase. Furthermore, the increased pressure benefits the measurement itself as the present gas bubbles are of increased sphericity, enhancing the image quality opposed to large unshapely bubbles.

In Figure 11b, the influence of an increased temperature is shown. The $d_{32}$ at elevated temperature is decreased, which is explained by a change in physical properties such as a lowered density and viscosity. The $d_{32}$ at varying heights is affected in a similar matter. Unlike pressure, an increase in temperature up to 80 °C does not therefore benefit the homogeneity of the dispersion.

It is thus concluded that while the axial stirrers offer an increased homogeneity of $d_{32}$ along the reactor axis with increasing stirring speeds, the radial flow pattern is better
suited to monitor the trend of $d_{32}$ at varying stirring speeds if it is monitored at one point in the reactor only. This further holds true when pressure and temperature are varied as well. With the RT positioned at 1/3 of the liquid level, the $d_{32}$ shows a stronger influence on the stirring speed compared to the other studied geometries. In addition, the immersion depth at (25 mm) or above (40 mm) the RT closely follow the trend of the average $d_{32}$ along the reactor axis, making these the preferred probe positions.

**CONCLUSIONS AND OUTLOOK**

In lean GLL systems with water as the dispersed phase, the field of view of a borescopic image acquisition is found to be clouded by adhering aqueous droplets. These droplets are successfully prevented using a polymeric protective disc made of either PC or preferentially FEP. The latter is found to withstand water and 1-octene at 100 °C, enabling its use in future reaction systems. The image acquisition can further be enhanced using a custom-built reflection module using sand-blasted steel. The resulting reflection of the light rays results in images of higher contrast and sharpness over the fully diffuse reflection offered by PTFE.

The study of gas-inducing modifications of stirrers was made possible by 3D-printing. In agitated GLL systems, gas induction reduces the efficiency of mixing which is notable by a sharp decrease in the power number of the stirrer and an increase in droplet sizes. When using gas-inducing stirrers, longer measurement times, an increased number of images, and a fine-tuned algorithm are required in comparison with standard stirrers and nongassing stirrers should be preferred when possible. This is due to gas-inducing stirrers promoting adhering droplets and gas bubbles obstructing the vision on droplets. Nevertheless, the measurement technique reliably provides DSDs in which gas bubbles are successfully neglected. Increased pressure benefits the measurement, as the sphericity of the gas bubbles is increased and the vision on the droplets is thereby improved. Furthermore, the pressure is found to increase the homogeneity of the dispersion.

It was found that axial stirring, especially upward stirring, promotes the homogeneity at higher stirring speeds. The dispersion was found to be the most homogeneous at 2000 min⁻¹, after which it decreases again. This trend, however, results in the fact that either an upward or a downward trend of the Sauter mean diameter is measurable at increasing stirring speeds depending on the position of the photo-optical probe. With radial stirrers, on the other hand, a downward trend of the Sauter mean diameter is measurable at all probe positions. Here, no increase in the homogeneity of the dispersion is notable. The RT provides the smallest Sauter mean diameters of all studied stirrers. It is concluded that the use of an upward stirring axial stirrer is preferable when the interfacial area is to be measured at high stirring speeds and when only one probe position is studied. The radial stirrer, on the other hand, is preferable when the trend of the interfacial area with varying stirring speeds or when large interfacial areas are of interest. For radial stirring, the most beneficial position of the probe is either close or slightly above the stirrer.

The use of high-pressure borescopy is found feasible for the study of droplet sizes in lean water-in-oil GLL systems, as long as adhering droplets can be prevented, that is, by the use of a polymeric disc, and the number of gas bubbles on the images is sufficiently low. Evaluation of the influence of the interfacial area on lean GLL reaction systems such as the industrially highly relevant aqueous biphasic hydroformylation is thus possible with this measurement technique.

**ASSOCIATED CONTENT**

*Supporting Information*

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acs.iecr.1c03707.

Technical drawings of studied stirrer geometries; technical drawing of reactor body and cap; piping and instrumentation diagram; comparison of polymeric discs after contact with 1-octene and water at 100 °C; technical drawing of a custom-built reflection module used in setup B; dead torque of the stirring system over stirring speed including one exemplary measurement (Rushton disc turbine) for reference; evaluation of automated image analysis versus manual analysis; droplet size distributions with varying measuring gap height using RTgas; exemplary image in which the vision on droplets is obstructed by bubbles; and droplet size distributions for RT and PBup for varying borescope immersion depth and stirring speed (PDF).
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SYMBOLS

Latin
\( A_d \) total surface area of dispersed droplets, \( m^2 \)
\( a_{1L} \) specific liquid—liquid interfacial area, \( m^{-1} \)
\( D \) diameter of stirrer, \( m \)
\( d_{32} \) Sauter mean diameter, \( m \)
\( d_{90} \) characteristic diameter of DSD, \( m \)
\( n \) rotational speed, \( s^{-1} \)
\( Ne \) Newton number \( Ne = \frac{p}{\rho \nu^3} \)
\( P \) power input, \( W \)
\( Re \) Reynolds number \( Re = \frac{\rho n D^2}{\eta} \)
\( V_d \) total volume of dispersed droplets, \( m^3 \)
\( V_{\text{total}} \) total liquid volume, \( m^3 \)
\( We \) Weber number \( We = \frac{p n^2 D^2}{\sigma} \)

Greek
\( \varepsilon \) energy dissipation rate, \( W m^{-3} \)
\( \eta \) dynamic viscosity, \( Pa s \)
\( \rho \) density, \( kg m^{-3} \)
\( \rho_c \) density of continuous phase, \( kg m^{-3} \)
\( \sigma \) interfacial tension, \( N m^{-1} \)
\( \tau \) torque, \( N m \)
\( \phi_d \) dispersed phase volume fraction, —

Abbreviations
DSD droplet size distribution
FEP fluorinated ethylene propylene
PB four-blade 45° pitched blade impeller
PBdown downward pumping PB
PBup upward pumping PB
PBupgas gas-inducing upward pumping PB
PC polycarbonate
PEEK polyether ether ketone
PLA poly lactide
PTFE polytetrafluoroethylene
RT six-blade Rushton disc turbine
RTgas gas-inducing RT
RTgasL gas-inducing RT with enlarged diameter
RTgasM mirrored design of RTgas
RTpitched RT with pitched tips

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