Coaxial Flow Contactors as Alternative to Double T-Contactors for Triphasic Slug Flow Generation

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Triphasic gas-liquid-liquid slug flow systems have great application potential in flow chemistry and are normally generated with a double T-junction where the continuous phase and one disperse phase form a two-phase flow and the second disperse phase is added at the second junction. This design is limited to high disperse phase ratios when a regular and uniform flow is desired. The use of coaxial contactors allows overcoming most of these restrictions. The slug generation, stability, and regularity of the generated triphasic flow were experimentally characterized.

Keywords: Capillary microreactor, Flow pattern, Gas-liquid-liquid segmented flow, Multiphase flow, Slug flow

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1 Introduction

Two- and three-phase slug flow systems play a crucial role in the field of microreactor and microextractor designs. While the generation of two-phase slug flows is a well-understood task with a lot of different contactor and junction designs, the generation of triphasic flow is not so well studied. Nevertheless, triphasic systems have been used and tested for a variety of different applications. Rajesh and Buwa, Yue as well as Wang et al. studied the flow regimes in triphasic flows and generated flow maps for stable regions [1–5]. Pressure drop was studied by Ladosz et al. and Yue et al. [5, 6]. The usability for extraction in multiphase flows was studied by Assmann et al., Aoki et al. and Su [7–9]. Homogeneous catalyzed reactions were performed in triphasic flow by Önal et al. and Cech et al. [10, 11]. The use of suspended heterogeneous catalyst has not been reported but seems possible by extending the work of Scheiff et al. [12].

In multiphase microfluidics it is important that the formed flow is regular and stable. This means that the sequence and size of the different phases are constant. In the case of a triphasic flow, there should be only one segment of each phase per repetition unit to allow a good mass transfer between all phases. The size variation of the slugs is normally expressed as the polydispersity index (*PDI*), which is defined as the standard derivation of the slug size divided by the mean slug length (Eq. (1)).

$$PDI_{\mathrm{i}} = \frac{\sqrt{\frac{1}{N-1}\sum\left(L_{\mathrm{i}} - L_{\mathrm{avg,i}}\right)^2}}{L_{\mathrm{avg,i}}} \cdot 100\% \tag{1}$$

In literature, a *PDI* below 5 % is considered as regular flow and can be achieved with most contactor designs for biphasic flows, gas-liquid and liquid-liquid [5].

Double T-junctions and cross junction were used to generate triphasic flow and their performance studied in the literature [1–5]. Double T-junctions were studied in detail by Wang et al. [4, 5] and they discovered some limitations in phase ratio and slug length. The frequency in which the slugs were generated at the junctions is dependent on the velocity in the capillary, which leads to different frequencies in the first and second contactor. The slug formation frequency for the first (Eq. (2)) and second (Eq. (3)) T-junction are listed below. In their experiments, the continuous phase was the organic phase and gas was added in the first contactor, while water was added as the second disperse phase in the second contactor. The frequency in the second contactor is the theoretical frequency that would occur if the water slug formation was not disturbed by the gas slugs. The actual frequency for the water slugs is highly dependent on the gas slug frequency due to slug cutting.

$$f_1 = \frac{\dot{V}_{\mathrm{G}}}{d^2 h\left(0.85\frac{\dot{V}_{\mathrm{G}}}{\dot{V}_{\mathrm{O}}} + 0.88\right)} \tag{2}$$

$$f_2 = \frac{\dot{V}_{\mathrm{W}}}{d^2 h\left(0.85\frac{\dot{V}_{\mathrm{W}}}{\dot{V}_{\mathrm{O}}+\dot{V}_{\mathrm{G}}} + 0.88\right)} \tag{3}$$

A regular flow pattern with only one segment of each phase per repetition unit can only be achieved when the flow rates are in the limit of Eq. (4). In all other cases, the

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slug formation frequency in the second contactor is high enough to generate more than one water slug per repetition unit or cut the existing disperse gas slug from the first contactor in half, also leading to more than one segment per repetition unit. Theoretically, there should be a region where the flow rate of the second disperse phase is so low that the second disperse phase is not cut off by the first phase every time, resulting in an unregular flow. However, this effect is not mentioned in the publication of Wang et al. and may only appear at very low phase ratios of the second disperse phase.

$$\frac{\dot{V}_{\mathrm{W}}}{\dot{V}_{\mathrm{O}}} \leq \frac{0.88}{0.85 + 0.88\frac{\dot{V}_{\mathrm{O}}}{\dot{V}_{\mathrm{G}}} - 0.85\frac{1}{1+\frac{\dot{V}_{\mathrm{G}}}{\dot{V}_{\mathrm{O}}}}} \quad (4)$$

The region of stable slug flow as measured by Wang et al. is displayed in Fig. 1. By inverting the disperse phases it should also be possible to generate a stable flow in the region marked by the dashed line. It is obvious that the double T-junction is limited to high disperse phase ratios and a stable and regular flow with a phase ratio of one to one for the disperse phases is not achievable. The resulting limitations can not be overcome by improving the current design as the formation frequencies are fixed for a given flow rate and geometry. It is, therefore, necessary to develop a new design to achieve stable flow for even phase ratios.

An alternative design to conventional contactors is the coaxial contactor, where the disperse phase is injected inside the continuous fluid with a needle. The needle position inside the broader region can be changed while the system is in use, allowing an ad hoc adjustment of the running system. The disperse phase forms a bubble at the tip of the needle and is removed from the tip by shear forces. By combining two coaxial contactors to a double coaxial contactor, similar to a double T-junction it is possible to generate triphasic slug flows. The control over the needle position allows the change and adjustment of the slug formation frequencies by changing the geometry in both contactors and, therefore, it should be possible to set both contactors to an equal formation frequency independent from the phase ratio and volume flow to avoid slug cutting.

2 Experimental Setup

Hexanol and M3 silicone oil were used as continuous organic phases and were obtained from Sigma Aldrich with a purity of 95 % or higher and double-distilled water was used as disperse phase. Methylene blue and Sudan red from VWR were used to dye the organic and water phases, respectively, to achieve a better contrast. Technical grade nitrogen from Linde was used as the gaseous dispersed phase. All experiments were carried out in fluorinated ethylene propylene capillary of 1 mm inner diameter within a tolerance range of ± 5 % deviation from Techlab. The coaxial contactors were constructed in the mechanical workshop of the Technical University Dortmund and made out of poly(methyl methacrylate). Due to their wetting properties, hexanol and silicone oil generate a wetting film and form the continuous phase in all experiments. Water and gas constitute the dispersed phases being screened from contact with the capillary wall by the organic films. The biphasic water gas flow after the first coaxial contactor is non-wetting due to the surface conditions of the capillary tube and no liquid film is formed around the gas phase. Two Legato 100 syringe pumps from KD Scientific were used to generate the liquid phase flow. The gas flow was regulated with an El Flow mass flow controller (MFC) from Bronkhorst. The flow rate for each phase was varied between 0.25 mL min^{-1} and 2.5 mL min^{-1}, resulting in a total volume flow between 0.75 and 7.5 mL min^{-1} and an average velocity of 19.9–199.4 mm s^{-1}. The corresponding capillary number *Ca* (Eq. (5)) and Reynolds number *Re* (Eq. (6)) ranges for hexanol and silicone oil are displayed in Tab. 1.

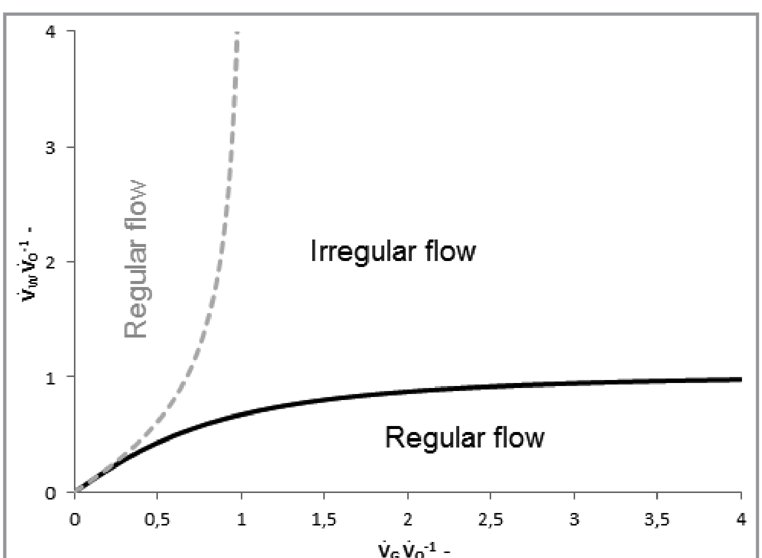

Figure 1. Regular and irregular triphasic slug flow regions for a double T-junction as proposed by Wang et al. [5]. The dotted line is the estimated regular region for inverted disperse phase inlets.

$$Ca_{\mathrm{i}} = \frac{u_{\mathrm{s,i}}\eta_{\mathrm{o}}}{\gamma_{\mathrm{i,o}}} \quad (5)$$

$$Re = \frac{u_{\mathrm{avg}}d\rho_{\mathrm{o}}}{\eta_{\mathrm{o}}} \quad (6)$$

To determine the slug length and *PDI* of the flow pictures were taken with a D3300 camera from Nikon. The exposure time was set to 1/2000 s resulting in an maximal image blur of 0.1 mm in flow direction for the maximal

Table 1. Range of *Ca* and *Re* for silicone oil and hexanol as organic phase.

	Minmum	Maximum
$Ca_{G,hexanol}$	0.0029	0.0289
$Ca_{L,hexanol}$	0.011	0.106
$Re_{hexanol}$	3.6	36.3
$Ca_{G,silicone}$	0.0028	0.028
$Ca_{L,silicone}$	0.0021	0.021
$Re_{silicone}$	6.3	63.1

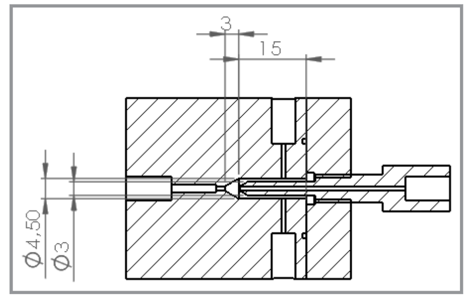

Figure 2. Dimension of the coaxial contactor used in the experiments. All capillaries are connected to the contactor with ¼" 28 UNF standard connections.

average velocity of 199.4 mm s^{-1}. A minimal resolution of 20 pixels per millimeter was used. The slugs were identified by their color as described below, the first 40 % of the color change from the base level outside the slug to the level inside the slug normally occurs within 3 pixels or ± 0.15 mm. For high velocities, the image blur increases the length of the color change to 5 pixels. The bubble surface is set to the middle of the color change region. Therefore, the combined accuracy of the length detection was less than ± 0.125 mm for high velocities and normally less than ± 0.075 mm. At least 30 repetition units per image were used to calculate the mean slug length and *PDI* for each phase. To determine the *PDI* derivation between different images three to five pictures for every experiment where compared. The PDI difference between the single pictures was less than 2 % for all images and less than 1 % in average. The medium *PDI* derived from all pictures was used for further comparison. The slug length and *PDI* were calculated with an in-house developed Matlab® program from a reference length in the pictures. The slug length is measured in the center of the capillary. A LED panel was mounted behind the capillary to reduce shadows and improve the image processing by providing uniform color intensities. The picture is separated into the red, blue and green color channel. The green values are used to determine the reference length from two green circles with a distance of 20 cm. The red and blue color channel are used to identify the organic and water slugs by a transportation to binary images, with white pixels representing the corresponding phase. The Canny algorithm in Matlab® is used to identify the edges of the capillary and the center of the capillary is determined by averaging the two capillary edges. The amount of succeeding white pixels is counted to determine the length of each slug along the center of the capillary. The gas slug length can be obtained by adding up the red and blue binary image and subsequent inversion of the image. The obtained slug length are saved for later evaluation and displayed on the original image to ensure an adequate detection.

The dimensions of the coaxial contactors constructed in the workshop are shown in Fig. 2 and the complete experimental setup is displayed in Fig. 3. As shown two coaxial contactors are placed after each other. The first contactor is used to combine both disperse phases to biphasic slug flow, while the second contactor introduces the continuous phase as the third phase to the system.

3 Results

In double T-junction normally one of the disperse phases and the continuous phase are brought into contact in the first contactor, as described by Wang et al. [5], and the second disperse phase is added in the second contactor, leading to the problems described above. Changing the order of flow has no beneficial effect in a double T-junction as the formation frequency in the second contactor would lead to a cutting of the disperse phases by the continuous phase in most cases. In the coaxial contactor it is possible to adjust the frequency of both contactors, which allows to combine both disperse phases in a first contactor, with the disperse liquid in the outer region and the gas flow in the needle and introduce the wetting continuous phase in the second coaxial contactor from the outer region to supplement the formation of a wetting film. The use of the conventional flow order is not recommended because the second disperse phase has to be introduced through the outer area of the second contactor, which is contradicting the natural structure of the triphasic flow, leading to a highly irregular flow.

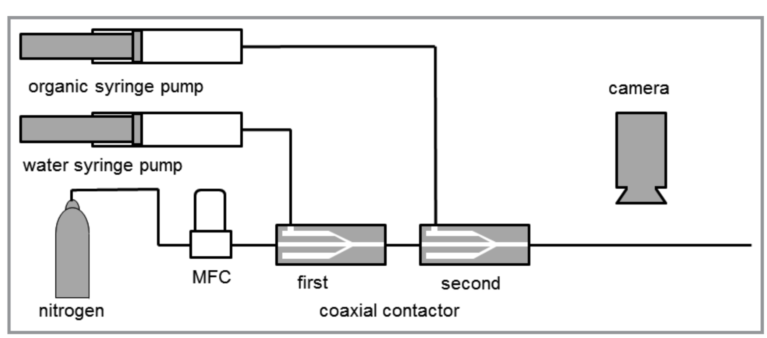

Figure 3. Experimental setup consisting of two coaxial contactors and optical slug detection.

No stable flow could be generated with this configuration, therefore, all experiments were carried out with the setup described above.

The first experimental goal was to prove that the slug length in this double coaxial contactor system can be controlled by the needle distance, which allows controlling the slug frequency in later experiments. Therefore, the needle distance was changed step by step in the first contactor while all volume flows were kept constant. Depending on the size of the slugs formed in the first contactor the gap in the second contactor was adjusted to avoid slug cutting or phase separation. This approach allowed a study of the first contactor without negative interference of the second contactor. In a second experiment, the position of the first contactor was kept constant to study the influence of the needle distance in the second contactor on the overall flow. The results for the first contactor are displayed in Fig. 4.

The behavior of the system can be clearly divided into three different regions: region one with no measured slug length and no flow at all, due to the small gap in the contactor the pressure drop was too high for the syringe pump. The second region is the region where the slug length is directly controlled by the needle position. As expected a greater gap volume leads to a larger slug if no slug cutting occurs, which only happens under certain conditions at high phase ratios. The reason why slug cutting can occur is explained later in greater detail. The experimental results suggest that the slug length is linearly dependent from the needle distance. The slug length for all three phases can be controlled by the first contactor. The length of the disperse phases is directly controlled by the needle position in the first contactor. For a regular slug flow with only one segment of each phase the size of the organic segment is then determined by the volume flows and length of the disperse phases. Eq. (7) gives a rough estimate of the resulting length, for spherical caps and a neglected volume of the continuous film. It is also worth noticing that the size difference between the phases stays constant. The slug length in the third region is independent of the needle position. In this region, the gap volume is so big that the phases separate inside the contactor volume and a parallel flow enters the capillary, which forms a slug flow inside the capillary completely independent from the contactor.

$$L_{\mathrm{O}} = \frac{2}{3}r + \frac{\dot{V}_{\mathrm{O}}}{\dot{V}_{\mathrm{G}} + \dot{V}_{\mathrm{W}}}\left(L_{\mathrm{G}} + L_{\mathrm{W}}\right) \quad (7)$$

For the second contactor, no influence of the needle position on the slug length could be determined except slug cutting if the needle distance was small and large disperse slugs were formed in the first contactor. In this case, the formation frequency in the second contactor is lower than the formation frequency in the first one and, therefore, more than one organic segment has to be formed in each repetition unit. If the needle distance is too big, phase separation occurs as explained for the first contactor and a highly irregular slug flow is formed inside the capillary. In the conducted experiments it was always possible to find a needle position for the second contactor, which resulted in a stable flow when a stable slug flow with equal disperse phase flow ratio was generated in the first contactor.

The influence of the overall volume flow on the slug length was tested in the second setup. The volume flow was increased while the phase ratios and needle positions were kept constant. The results as shown in Fig. 5 indicate that the slug length of all three phases is independent of the overall volume flow. The effect of a changed phase ratio on the slug length is shown in Fig. 6. The length of the continuous phase is not displayed but can be determined from the disperse phases by Eq. (5). It can be seen that the gas phase introduced through the needle has a linear reaction to a changed phase ratio. The water phase changes in a corresponding fashion due to the overall flow rate and formation frequency. Therefore, the slug length of the water and gas phase can be calculated with Eq. (8) and (9). The factors α and β are determined by contactor geometry, surface tension and viscosity of the phase not introduced through the needle.

$$L_{\mathrm{W}} = \frac{L_{\mathrm{G}} + \frac{2}{3}\left(1 - \frac{\dot{V}_{\mathrm{G}}}{\dot{V}_{\mathrm{W}}}\right)}{\frac{\dot{V}_{\mathrm{G}}}{\dot{V}_{\mathrm{W}}}} \quad (8)$$

$$L_{\mathrm{G}} = \alpha + \beta\frac{\dot{V}_{\mathrm{G}}}{\dot{V}_{\mathrm{W}}} \quad (9)$$

To conclude this first section it was shown that the double coaxial contactor design is

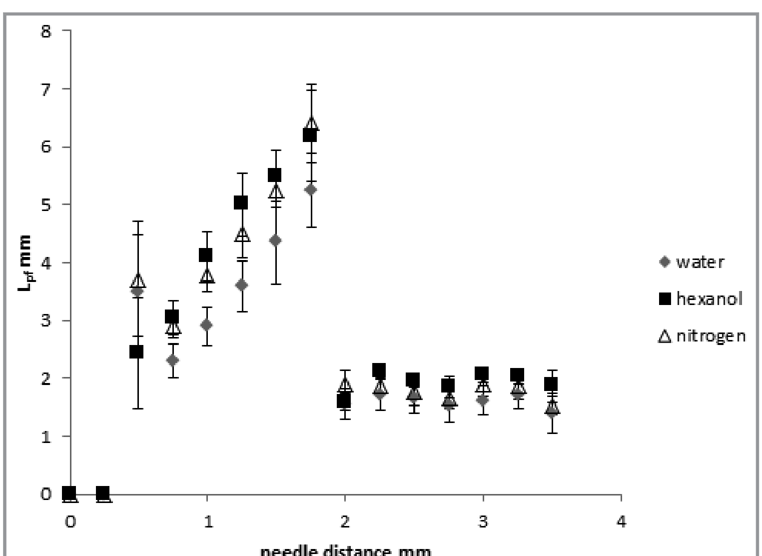

Figure 4. Resulting slug length for different needle positions and resulting gap length for the first coaxial contactor with 0.8 mL min^{-1} nitrogen, 0.7 mL min^{-1} water and 0.7 mL min^{-1} hexanol flow. The error bars indicate the *PDI* length distribution for each point.

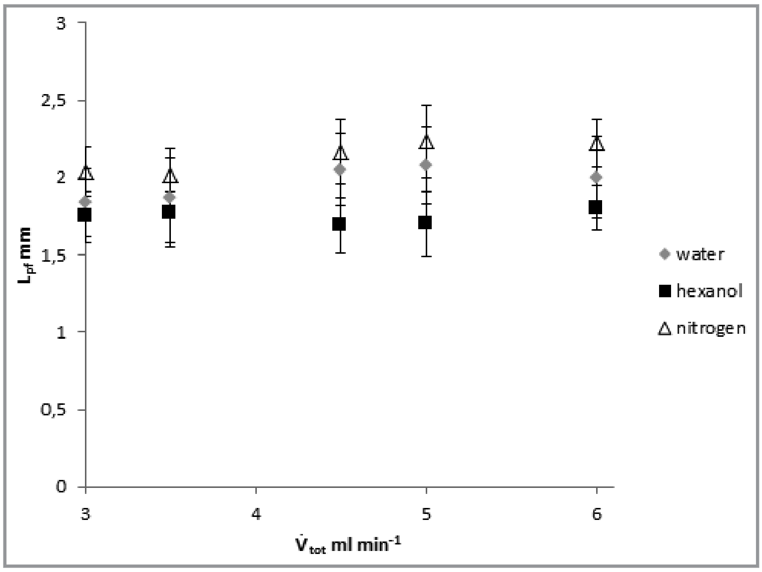

Figure 5. Slug length for constant phase ratos of one with different total volumetric flowrate. The error bars indicate the *PDI* length distribution for the measurements.

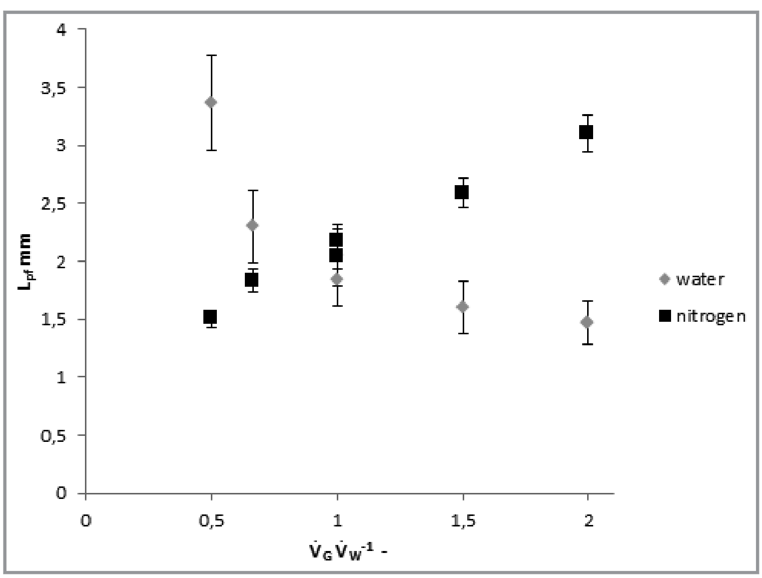

Figure 6. Influence of the gas to water phase ratio under constant total disperse flow rate of 3 mL min^{-1}. *PDI* length distribution is indicatet with error bars.

capable of generating stable triphasic flows, examples for different flow conditions are displayed in Fig. 7. The slug length can be controlled with a linear coupling by the needle distance and is independent of the volume flow. The effect of the disperse phase ratio is linear for the phase that is introduced with the needle and the length of other phases can be predicted based on the gas phase length. This allows calculating the slug length of a given system in a predictive manner. This is an important feature and allows the predictive calculation of the triphasic pressure drop and slug velocity [6, 13].

When the disperse phase ratio is not equal to one, slug cutting can occur. In this case, the longer slug is cut into smaller slugs (Fig. 7d). It is worth noticing that the number and size of slugs that a large slug is cut into stays constant for each flow condition. The cut slug flow is, therefore, regular except the point that more than one segment of each phase is present in one repetition phase.

Fig. 8 shows the range for stable slug flow and the regions where slug cutting occurs. A point was marked as unstable if in at least one experiment no stable flow could be achieved. The border between both regimes can be approximated by a phase ratio greater than three or less than one third. Slug cutting is only observed if the longer phase is the first phase in the repetition unit, the second disperse slug remains a single slug even if the phase ratio is exceeding the limits for the first disperse phase. The order of slugs in the triphasic flow is determined by the slug velocities of both disperse phases. The faster phase closes up to the slower phase in the first millimeters after the second contactor and the formed double slug has the velocity of the slower disperse phase [13]. Therefore, if the first phase is cleaved the newly formed slugs stay separated. If the second slug is cleaved the faster slugs close up to the slower slug and reunite to one slug. Therefore, slug cutting occurs in every case if the phase ratio is high enough, but the cut slugs are only stable if the faster phase is cut. On the other hand, if a high phase ratio is needed and a regular slug flow is needed the phase with the high phase ratio should be the slower phase and the chemical system should be chosen according to this fact.

The *PDI* of the generated triphasic slug flow is normally in the range of 5 to 10 % and can reach values up to 20 % for the water and organic phase. On the other hand, gas *PDI* stays around 5 %. This is not sufficient for regular slug flow, due to the fact that a high *PDI* has a negative influence on the separation efficiency or the selectivity for reactive systems. A more detailed study shows that the *PDI* is dependent on the gas flow rate as shown in Fig. 9. An effect of a nearby experiment that was connected to the same gas source was also observed, leading to the conclusion that the gas flow from the MFC was not constant during an experiment. This leads to a fluctuation in the gas slug formation frequency while the gas slug size remained almost constant, resulting in regular gas flow with low *PDI* but fluctuations in the water and subsequently organic slug length.

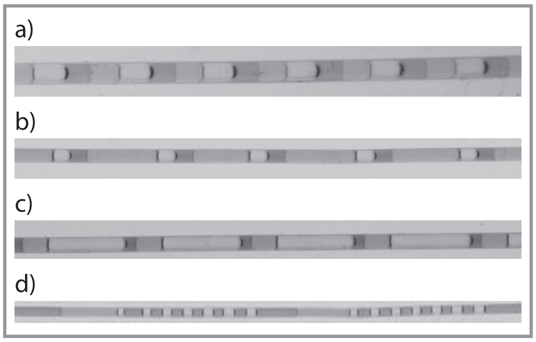

Figure 7. Flow direction from right to left in all pictures. Gas slugs are white, lighter gray is the water phase and dark gray is the continuous hexanol phase. Gas is always in front of the water phase due to the lower slug velocity of gas compared to water in hexanol. a) Triphasic slug flow with 1 mL min^{-1} for all phases; b) stable flow with high water percentage; c) stable triphasic flow with high gas content; d) slug cutting for very high gas phase ratio.

To determine the true potential of the double coaxial contactor a stable gas source was needed. An electrolytic cell was used as an alternative gas source to generate a stable gas flow with no outer interference and fluctuations. The experiments shown in Fig. 8 were repeated and the result is shown in Fig. 10. The displayed stable and regular region had a *PDI* under 5 % and no slug cutting in all experiments. This stable and regular region is limited by a dispersed phase ratio between 0.5 and 2 and a dispersed to organic phase ratio of 0.4 up to 2.5.

The limitation in the dispersed to organic phase ratio results from the small size of either the organic or dispersed section for very high or low phase ratios. In this case, minor fluctuations and the measurement error of the slug length becomes more prominent. The *PDI* was below 10 % or in the range generated by the length measurement error, if it was higher than 10 %, for all experiments without slug cutting. Slug cutting occurred in the same region as displayed

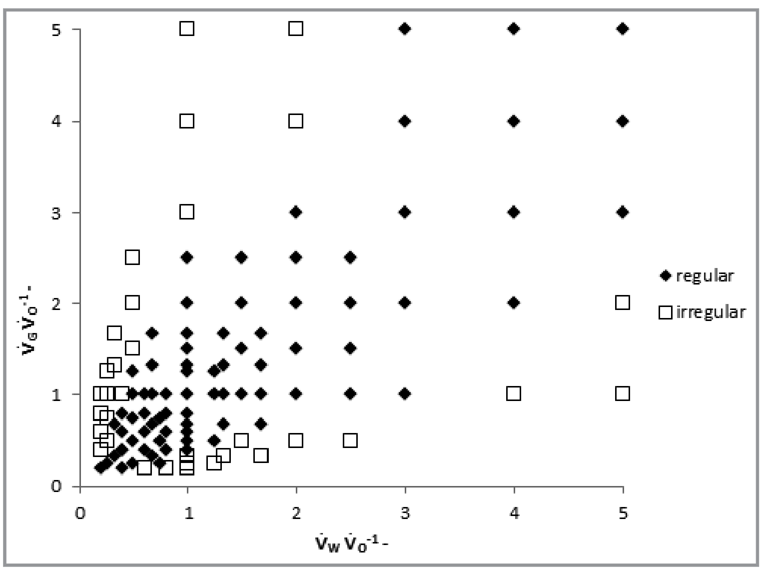

Figure 8. Stable region of triphasic slug flow generated with a double coaxial contactor and MFC as gas source. Slug cutting occurs outside the stable region if the larger slug is the slower disperse phase. Measured with flow rates between 0.5 and 2.5 mL min^{-1} and silicone oil and hexanol as organic phases.

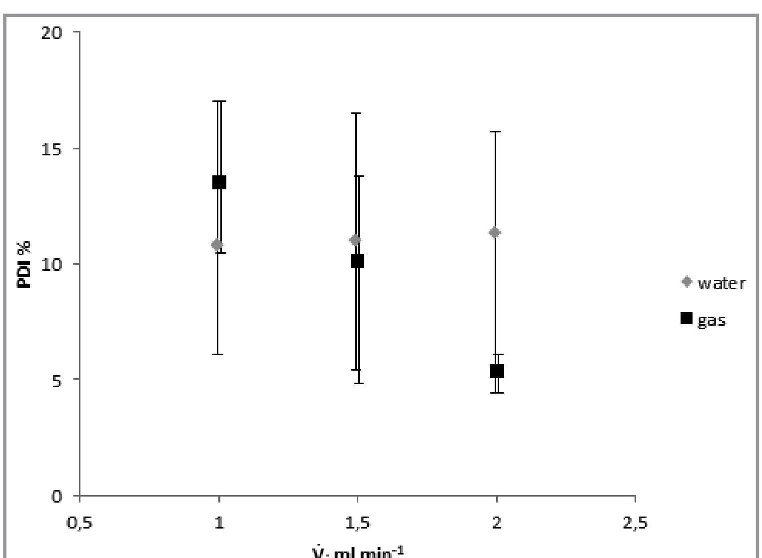

Figure 9. Influence of the gas and water flow rate on the water *PDI* in all measurements for the given flow rate of the named phase. The maximum and minimum *PDI* values are indicated with error bars and the dot indicates the medium value of all experiments.

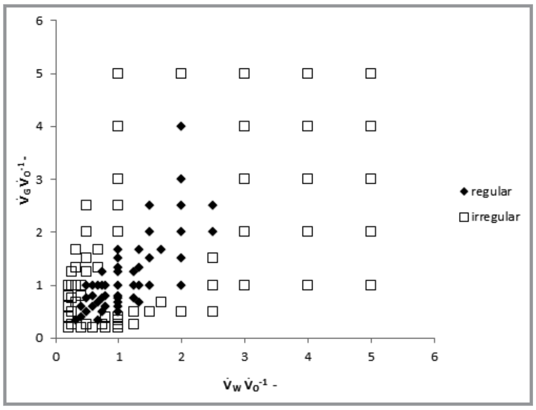

Figure 10. Stable and regular slug flow with a PDI of less than 5 %. Gas flow is generated with an external electrolytic cell. The flow rates of each three phases were varied between 0.5 and 2.5 mL min^{-1}.

in Fig. 8, resulting in a broader region where the double coaxial contactor can be used if a *PDI* of less than 10 % is sufficient. The average *PDI* for equal volume flows of each phase is 4.2 %for the gas phase, 3.6 % for the water phase and 2.3 % for the organic phase. For the same conditions the average *PDI*s in the double T-junction are 14.3, 11.3 and 12.5 % showing the better performance of the double coaxial contactor in this region.

A general benefit of the double coaxial contactor is the independence of the slug length from the other parameters of the flow. This enables the adjustment of the specific surface area in the slug flow to the optimal value for the performed reaction or extraction, without interference with the flow rates. The phase ratios where a stable and regular slug flow can be achieved in a double coaxial contactor and a double T-junction complement one another in a way that either a double T-junction or a double coaxial contactor can generate a stable and regular slug flow for almost every possible phase ratio combination.

5 Summary

The double coaxial is a new contactor design for triphasic slug flow that can fill the gap of the existing double T-junctions. It was possible to generate a stable and regular slug flow for low disperse phase ratios. The length of the generated slugs can be controlled by the needle distance and is independent of the volumetric flow as long as the phase ratios are kept constant. This independence allows the adjustment of slug length during an experiment and greatly increases the flexibility of the double coaxial contactor. Slug cutting occurs when the disperse phase ratio exceeds a certain level, but can be avoided if the cleaved phase is the slower dispersed phase. Double coaxial contactors are a good addition to the existing double T- junctions as they perform best with low disperse phase ratios while double T-junctions are limited to higher phase ratios.

Symbols used

Ca	[–]	capillary number
d	[m]	diameter
F	[s^{-1}]	frequency
L	[m]	length
N	[–]	number of slugs
PDI	[–]	poydispersity index
Re	[–]	Reynolds number
$\dot{V}$	[m^{3}s]	volume flow

Greek letters

α	[m]	fit coefficient
β	[m]	fit coefficient
γ	[N m^{-1}]	interface tension
η	[Pa s]	viscosity
ρ	[kg m^{-3}]	density

Sub- and Superscripts

avg	average
G	gas
i	phase
O	organic
s	slug
tot	total
W	water

Abbreviations

PDI	polydispersity index
MFC	mass flow controler

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