Micro Droplets of non-Newtonian Solutions in Silicone Oil Flow through a Hydrophobic Micro Cross-Junction

B Rostami and G L Morini
DIN – Alma Mater Studiorum Università di Bologna, Laboratorio di Microfluidica, Via del Lazzaretto 15/5, Bologna 40131, Italy

E-mail: behnam.rostami2@unibo.it

Abstract. In this paper the generation of non-Newtonian droplets of aqueous Xanthan gum solution (0.3, 0.5 wt%) in a silicone oil flow through a micro cross-junction is experimentally analyzed. A commercial glass cross-junction microchip with hydrophobic walls has been employed to study the droplet generation mechanism. The cross-section of the channel is stadium-shaped, the width of the junction varies between 195 to 390 μm while the height of the channel is fixed at 190 μm. Tween 20 (2 wt%), as a surfactant, has been added to the dispersed phase to avoid the coalescence of the droplets and to enhance the droplet formation. With the aim to follow the time evolution of the droplets inside the channel a specific experimental setup has been implemented. The post-processing of the recorded images has been carried out by means of an “in-house” Matlab code. The typical flow patterns obtained by imposing different flow rates at the inlets of the cross-junction have been observed. The effect of the continuous and dispersed phase flow rates as well as the concentration of Xanthan gum solution on the main droplet characteristics has been studied in detail.

1. Introduction
Nowadays microdevices are commonly used to generate mono-dispersed droplets. A series of simple microfluidic devices have been proposed for droplet generation like T-junctions [1], co-flow devices [2], flow-focusing devices [3] and microchannel terraces [4]. Among flow-focussing devices micro cross-junctions have received a special attention for the generation of mono-dispersed droplets. In these devices, the continuous flow is introduced by means of two lateral junction branches in order to squeeze and shear off the interface of an immiscible liquid used as the dispersed phase and to introduce it inside the junction through its central microchannel.

Mono-dispersed droplets with low size variation are highly desirable in many technical applications such as DNA and blood analysis [5], chemical reactions [6] and drug discovery [7]. It has been demonstrated that the main governing and controlling parameters which enable the control of the droplet size generated in a micro cross-junction are the properties of the liquids introduced in the junctions, their inlet flow rates and the geometry of the micro-junction.

Although squeezing regime was observed and considered as the main mechanism in droplet formation by Garstecki et al. [8], additional mechanisms, like dripping and jetting can play a role on the droplet generation in these microdevices. In fact, as demonstrated by the experimental observations, all these three mechanisms can be met by varying the continuous phase flow rate Qc at the entrance of the micro-junction for a fixed flow rate of the dispersed phase (Qd). By focusing the attention on the location
of the droplet generation inside the junction, it is possible to distinguish: (i) droplets formed at the cross-junction (DCJ) and (ii) droplets formed downstream of the channel (DDC), by increasing the inlet dispersed phase flow rate \( Q_d \). Most of the works appeared in literature were devoted to the analysis of the droplet generation in cross-junctions characterized by low inlet flow rates for which the squeezing mechanism becomes predominant. It will be demonstrated in this paper that, especially when non-Newtonian fluids are involved, dripping and jetting flow patterns become more and more important with respect to squeezing and they cannot be ignored.

Despite the importance of non-Newtonian fluids in industrial and pharmaceutical fields, the investigation of their behaviour in micro cross-junction during droplet generation can be nowadays considered disregarded. In order to start to fill this gap in this work an aqueous solution of Xanthan gum with low concentration (0.3-0.5 wt\%) has been used as dispersed flow. These aqueous Xanthan gum solutions are typical shear thinning fluids with properties similar to blood plasma. The effects of the elasticity of these solutions as well as the effect of the channel dimension on the droplet generation was studied by Steinhaus et al. [9] by considering a Newtonian fluid as carrier fluid. Hong and Cooper-White [10] utilized Carbopol dispersions in order to study the droplet formation and breakup mechanism in presence of non-Newtonian dispersed flows. By the use of Carbopol dispersion which shows shear thinning and elastic properties, they showed that the droplet size was notably influenced by the viscosity ratio between the dispersed and the continuous phase. Husny and Cooper-White [11] have evidenced the presence of elongated filaments during the droplet formation with non-Newtonian dispersed flows due to the elasticity of the droplets as well as the presence of secondary droplets (satellites) close to the main droplet. They highlighted that the droplet polydispersity and the number of secondary droplets are a function of viscosity ratio. Also Lee et al. [12] studied the effect of viscoelasticity of the dispersed phase on the breakup dynamics. Arratia et al. [13, 14], using polyacrylamide solutions with different molecular weights and different elasticity values, studied in detail the filament thinning and droplet breakup in presence of these non-Newtonian flows. As evidenced by this short literature survey, the researchers have concentrated their attention on the role played by the elasticity and the molecular weight of non-Newtonian fluids on the droplet characteristics but other aspects was not deeply investigated up to now.

In this paper an experimental setup has been designed in order to analyze the generation of non-Newtonian droplets in a silicone oil continuous flow within a commercial glass micro cross-junction. Aqueous Xanthan gum solutions with two different concentrations (0.3 and 0.5 wt\%) have been considered as dispersed phase. Tween 20 (2 wt\%) has been added to the non-Newtonian solution to avoid the coalescence of the droplets and to enhance their formation. A wide range of volumetric flow rates at the inlet for both continuous and the dispersed phase have been tested in order to be able to detect different flow patterns including squeezing, dripping and jetting regimes. More in detail, the continuous phase (silicone oil) flow rate \( (Q_c) \) varies from 0.3 to 10 ml/h; the range of dispersed phase (Xanthan gum solution) flow rate \( (Q_d) \) has been varied from 0.01 to 2.1 ml/h. The non-dimensional droplet diameter has been defined and plotted as a function of flow rate ratio and the Capillary number to study the effect of the main physical governing parameters on the droplet features. In addition, the effect of Xanthan gum concentration in aqueous solution has been analyzed.

2. Experimental setup

The experimental apparatus used in this work for the analysis of the characteristics of the droplets generated in a glass micro cross-junction is shown in figure 1.

An inverted microscope (1, Nikon Eclipse TE2000-U) with a double system of illumination by means of a Mercury lamp (2a, Nikon C-SHGI) from the bottom and a COB LED lamp (2b, 100W, 9000 Lumens) from the top is the main part of the test rig. An air immersion lens with a numerical aperture \( N A = 0.25 \) and magnification \( M = 10x \) (3, Nikon CFIDS 10X) is selected in order to visualize the droplets. A high-speed camera (4, Olympus I-speed II) connected to the inverted microscope is used to acquire the droplet images; a LCD monitor (5) and Olympus I-speed II software (6) makes possible a run-time visualization of the flow within the micro-device. The volumetric flow rate of silicone oil (continuous
phase) and of the Xanthan gum solution (dispersed phase) are controlled by two independent syringe pumps \((7, \text{ Cole-Parmer Version Hills} \text{ and } 8, \text{ Harvard Apparatus PHD4400 Programmable})\) and introduced at the three inlets of the junction \((11)\) using two gastight Hamilton syringes \((9 \text{ and } 10)\). The relative uncertainty of the imposed volumetric flow rate for continuous and dispersed phase is equal to ± 0.5% and ± 0.35%, respectively, based on the accuracy of the syringe pumps.

![Figure 1. The experimental setup](image)

The generation of non-Newtonian emulsions has been obtained by using a commercial glass micro cross-junction \((\text{Dolomite Microfluidics Co.})\). The stadium shape of the cross-section, the channel width variation through the junction and the hydrophobic inner walls are the main features of this junction. The cross-junction (figure 2a) is characterized by a cross-section restriction at the junction in which the width of the channel varies from \(W_\omega = 390 \mu m\) far from the junction to \(W_j = 195 \mu m\) at the junction (figures 2b and 2c). The restriction ratio defined as the ratio of these two parameters \((R = W_j/W_\omega)\) is equal to 0.5. The height of the channel \((H)\) is uniform and equal to 190 μm. The aspect ratio, i.e., the ratio between the channel height and width \(H/W\), is equal to \(A_j = 0.97\) at the junction and \(A_\omega = 0.49\) out of it. In order to avoid the coalescence of the emulsions in an organic phase, a hydrophobic coating is required.

![Figure 2. Micro cross-junction geometry; the distribution of the fluids (a); cross-section far from the junction (b) and at the junction (c)](image)

The main physical parameters involved in the droplet generation in the micro cross-junction are the volumetric flow rate \(Q\), density \(\rho\), viscosity \(\mu\) and the interfacial tension \(\sigma\) of the continuous and dispersed phase. By using the Buckingham-Pi theorem it is possible to demonstrate that a complete set of non-dimensional parameters for the analysis of the droplet generation in a cross-junction is given by the flow rate ratio \(\alpha = Q_d/Q_c\), the dimensionless numbers Capillary number \((Ca = \mu u/\sigma)\) where \(u\) (= \(Q/A\)) is the fluid velocity, Reynolds number \((Re = \rho u W/\mu)\) and by the channel aspect ratio \((A_\omega \text{ or } A_j)\).

The geometry of the channel shown in figure 2 clarifies in which way the continuous and the dispersed phase are introduced in the junction.
The silicone oil (Sigma Aldrich, viscosity 20 cSt, density 0.95 g/ml @ 25 °C) has been employed as the continuous phase in the tests. Tween 20 (Sigma Aldrich, density 1.095 g/ml @ 25 °C) is used as surfactant added to the Xanthan gum solution in small concentrations (2 wt%). Tween 20 (miscible in water) is a non-ionic detergent widely used in biochemical applications. The Hydrophilic-Lipophilic Balance (HLB) value of this surfactant is equal to HLB = 16.7 and it can be proficiently used for O/W emulsions.

The density of continuous and dispersed phases has been measured by using a precise analytical balance (RADWAG AS 220.R2). The measured values of density for the fluids used in this work are reported in Table 1.

The viscosity of silicone oil is measured using a Cannon-Fenske Viscometers and it is shown in Table 1. It is evident that the viscosity of silicone oil measured in the Lab is very close to the viscosity value declared by the manufacturer (19 cP @ 25 °C).

A Brookfield DV-II + viscometer with RV spindles has been used for the characterisation of the non-Newtonian solutions. The aqueous solutions of Xanthan gum exhibited a shear thinning property for various shear rates. The power-law model \( \mu = r \gamma^{n-1} \) can be used for the characterization of the non-Newtonian solution used in these tests where \( \mu \) is the viscosity index (cP s\(^{-1}\)), \( \gamma \) the shear rate (s\(^{-1}\)) and \( n \) the flow index of the fluid. The values of the consistency index and of the flow index associated to the Xanthan gum solutions is reported in table 1 with the correlation coefficient (\( R^2 \)).

Surface tension of the working fluids with air has been measured by means of a force tensiometer (KSV Sigma 700). Each value of the surface/interfacial tension is the average value of a ten-time repeated experiment.

| Table 1. Physical properties of the working fluids with Tween 20 (2 wt%) |
|---------------------------------|---------|---------|---------|
| Fluid                          | Density (g/ml) | Viscosity (cP) | Surface tension (mN/m) |
|                                 | r        | n        | R\(^2\)   |                     |
| Silicone oil                   | 0.9480 ± 5.24 E-4 | 18.07 ± 0.078 | 10.00 - | 19.30 ± 0.005 |
| 0.3 wt% Xanthan gum            | 1.0041 ± 0.00010 | 2562.5 | 0.27 | 0.999 | 26.44 ± 0.31 |
| 0.5 wt% Xanthan gum            | 0.9993 ± 0.00339 | 10538 | 0.16 | 1.000 | 27.95 ± 0.12 |

The interfacial tension between silicone oil and Xanthan gum solutions has been measured by using the same force tensiometer and the values are presented in table 2.

| Table 2. Interfacial tension of the silicone oil with Xanthan gum solutions with Tween 20 (2 wt%) |
|-------------------------------------------------|-----------------|
| Solution                                        | Interfacial tension (mN/m) |
| 0.3 wt% Xanthan gum                             | 7.4246 ± 0.38   |
| 0.5 wt% Xanthan gum                             | 6.4518 ± 0.14   |

3. Results

As explained before, by means of the two syringe pumps the flow rate of silicone oil and Xanthan gum solutions have been varied at the inlets of the cross-junction in order to investigate the effect of the flow rate ratio on the characteristics of the droplets at the outlet of the junction. In order to study the geometrical characteristics of the droplets, the acquired images have been post-processed by using a Matlab code by means of which the geometry of each droplet (diameter, length, position of the edge with respect to the centre of the junction) is reconstructed [16].

3.1. Flow pattern at low flow rates of the continuous phase (< 2 ml/h)

By varying the volumetric flow rate of the continuous phase (silicone oil) from 0.5 to 2 ml/h a series of visualisations has been recorded for values of the dispersed phase flow rate ranging between 0.01 and 2.1 ml/h.
Figure 3. The typical models of dispersed phase flow 0.5 wt% Xanthan gum solution where \( Q_c = 1.5 \) ml/h and \( Q_d = 0.015, 0.2 \) and 1.65 ml/h, respectively; DCJ (a), DDC (b) and PF (c)

The main mechanism of the droplet formation for low values of \( Q_c \) (< 2 ml/h) is squeezing.

In figure 3 the typical flow patterns observed by varying \( Q_d \) from 0.015 ml/h to 1.65 ml/h for a fixed value of \( Q_c \) equal to 1.5 ml/h are shown. It is evident that droplets are generated close to the centre of the junction when low values of the flow rate ratio (\( \alpha \)) are imposed (see figure 3a); in this case the detachment point of the neck connecting the droplet to the source of the dispersed phase takes place close to the centre of the junction. In this case the main mechanism responsible of the droplet generation is squeezing which means that the pressure drop force is the dominant one; this flow pattern will be called DCJ (Droplet generated at the Cross Junction) in this paper. By increasing \( \alpha \) the detachment point shifts downstream along the outlet of the junction and an elongated filament links the droplet to the core of the dispersed phase (see figure 3b); this flow pattern is called here DDC (Droplet generated Downstream of the Channel). Finally, for very high values of the flow rate ratio (\( \alpha \)) a stratified parallel flow (called PF here) is observed and the droplet generation is stopped (see figure 3c). Both DCJ and DDC regimes are linked to the squeezing mechanism.

Figure 4. Flow pattern maps as a function of flow rate ratio \( \alpha \) and the Capillary number linked to the continuous phase (\( Ca_c \)) for a 0.3 wt% Xanthan gum solution (a) and 0.5 wt% Xanthan gum solution (b)

In figure 4 the correlation among these three typical flow regimes (DCJ, DDC and PF), the flow rate ratio (\( \alpha \)) and the Capillary number based on the continuous phase (\( Ca_c \)) is shown by means of a 2D flow pattern map obtained for two different concentrations of Xanthan gum solution (0.3 and 0.5 wt%). By observing figures 4a and 4b, it is evident how, by fixing the value of \( Ca_c \), an increase of \( \alpha \) leads to the transition from DCJ to DDC to PF regime. On the contrary, an increase in \( Ca_c \) for a fixed value of \( \alpha \) is not able to put in evidence any transition. This confirms that both DCJ and DDC regimes are squeezing regimes in which \( \alpha \) plays an important role. However, it is also evident how for low values of \( Ca_c \), the extension of the DCJ region increases which means that the droplet can be regularly generated at the
centre of the junction for a larger range of flow rate ratios. This result is in agreement with the observations of Liu and Zhang [17] that in a micro cross-junction at low values of the continuous Capillary number \((0.002 \leq Ca_c \leq 0.008)\) observed numerically, by using Lattice Boltzmann method, the same evolution of the droplet regimes from DCJ to DDC to PF when the flow rate ratio was increased.

By comparing figure 4a with figure 4b, it seems that the concentration of Xanthan gum from 0.3 wt% to 0.5 wt% weakly influences the flow map. However, in order to have a clearer understanding about the effect of Xanthan gum concentration on the transition from one regime to another one, more experimental runs are needed with different values of concentrations. The dimensionless diameter of the droplets \(D^*\), defined as the ratio of the square root of the multiplication of horizontal \((D_h)\) and vertical \((D_v)\) diameter of the droplet and the height of the channel \(D^* = \sqrt{D_hD_v/H}\), is shown in figure 5 as a function of the flow rate ratio \(\alpha\); \(Q_c\) varies from 0.5 to 2 ml/h while the range of variation of \(Q_d\) is from 0.01 to 2.1 ml/h.

It is clear that an increase in \(\alpha\) determines an increase of the dimensionless droplet diameter. Moving from DCJ to DDC flow patterns, the slope shown in figure 5 tends to change; the data quoted in figure 5 underline that the dimensionless diameter of the droplets is more sensible to a variation of the flow rate ratio in DDC regime with respect to the DCJ regime. Once more, the Xanthan gum concentration seems to have a weak effect on the droplet dimensions by comparing open and closed symbols in figure 5.

3.2. Flow pattern at high flow rates of the continuous phase (>2 ml/h)
When the flow rate of the continuous phase becomes larger than 2 ml/h, the squeezing mechanism tends to become less important in the droplet generation and dripping and jetting regimes become predominant. When the flow rate of the continuous phase is low, due to the low shear forces the control of the droplet formation and breakup is linked to the combined effect of interfacial tensions and pressure drop forces (squeezing regime) and the typical interface is represented in figure 6a. In the case presented in figure 6a the central channel of the cross-junction, where the dispersed phase (Xanthan gum solution) flows, is almost blocked at the cross-junction. The increase in \(Q_c\) \((Ca_c)\) leads the shear forces to play a more important role in governing the mechanism of the droplet formation. Dripping regime is obtained when shear forces and the interfacial tensions are of a similar order of magnitude; in this case, in presence of a non-Newtonian flow a filament is originated between the dispersed core at the junction inlet and the droplet.

![Figure 5. Dimensionless droplet diameter as a function of the flow rate ratio for two different concentrations of Xant...](image-url)
If the flow rate of the continuous phase is further increased, the filament becomes longer and longer and the droplet dimensions tend to be blocked by the reduced flow of dispersed phase flowing within the filament, as evidenced by the comparison between figure 6b and figure 6c (Jetting regime). It is interesting to highlight that, in presence of non-Newtonian fluids like Xanthan gum solutions, a very long and slender filament can be observed before the droplet breakup which is due to the high viscosity of the dispersed phase. This filament is transformed in a satellite droplet after the breakup. The dimensions of the satellite droplets are correlated to the length of the filament before the breakup.

Figure 7 confirms that the dimensionless droplet diameter decreases when the flow rate of the continuous phase is increased (which means decreasing of flow rate ratio α). The results shown in figure 7 are obtained by varying the flow rate of the dispersed phase from 0.01 to 2.1 ml/h and the flow rate of the continuous phase from 0.3 to 10 ml/h. These results are in agreement with the literature [17,18]. Garstecki et al. [18] presented the dimensionless length of the aqueous droplets within a micro T-junction. They observed that at high values of flow rate ratio (α> 1) the size of the droplet increases linearly with α. On the contrary, the droplet length tends to reduce its dependence on the flow rate ratio for α < 1. Here the same trend is observed in figure 7 but, due to the properties of the non-Newtonian dispersed phase, the change of slope is located at α = 0.1. The results of figure 7 highlight that in order to obtain larger droplets the micro-junction has to work with large flow rate ratio (i.e. at low flow rates of the continuous phase or at large flow rates of the dispersed phase). In addition, the dimensionless droplet diameter becomes more sensitive to the variation of the flow rate ratio for α > 0.1 where the squeezing mechanism becomes predominant. In addition, if the flow rate of the continuous phase is increased (for a fixed flow rate value of the dispersed phase), the time needed for the droplet formation decreases and a weak dependence of the droplet dimensions on Qd is observed.

![Figure 6. Figure showing Squeezing (a), Dripping (b) and Jetting (c) regimes where Qc = 0.3, 3 and 8 ml/h and Qd = 0.1 ml/h, respectively for 0.2 wt% Xanthan gum solution.](image)

![Figure 7. Dimensionless droplet diameter as a function of the flow rate ratio (α) for different Xanthan gum concentrations.](image)
In figure 8 the role of the Capillary number linked to the continuous phase on the droplet dimensions is highlighted. By increasing the Capillary number $Ca$, the mechanism of the droplet formation changes from squeezing to dripping to jetting with a reduction of the droplet dimensions. At low Capillary numbers $Ca \leq 0.01$ the squeezing regime is observed (see figure 6a). When $Ca$ is increased, dripping and jetting regimes start, where a trade-off between shear forces and interfacial tensions controls the droplet formation. The large viscosity of the Xanthan gum solution justifies the filament formation evidenced in figures 6b and 6c and, as a consequence, the reduced dimensions of the droplets. In dripping and jetting regimes shear forces are predominant and the droplet dimensions are more dependent on $Ca$ than on $\alpha$ as evidenced by the comparison of figures 7 and 8 for $D^* < 1$.

![Figure 8](image)

**Figure 8.** Dimensionless droplet diameter as a function of the Capillary number of the continuous phase

Figure 9 shows the effect of the Xanthan gum concentration on the droplet dimensions. Although the rheological properties of the dispersed phase solutions are different, the droplet diameter variation is not very large for a fixed values of the flow rate ratio. As a rule of thumb, the higher the Xanthan gum concentration is, the larger droplets are generated. Higher values of Xanthan gum concentration mean higher viscosity values.

It’s worth mentioning that these results are in disagreement with the case in which Newtonian droplets are formed.

![Figure 9](image)

**Figure 9.** Effect of Xanthan gum concentration on droplet dimensionless diameter for various flow rates while open symbols stand for 0.3 wt% and closed symbol represent 0.5 wt%
In Newtonian fluids larger droplets with less viscous solutions are generated due to the higher viscous pressure in the thread which results in longer filaments [19, 20]. On the other hand, an increase of the dispersed phase viscosity is responsible of a larger shear force which reduces the droplet dimensions [19]. However, more experimental data obtained with different concentrations of Xanthan gum are needed in order to be able to put a spotlight on the rheological effects of the non-Newtonian dispersed phase on the droplet size.

4. Conclusion
In this work the generation of non-Newtonian droplets of aqueous Xanthan gum solutions in silicone oil by using a commercial glass micro cross-junction has been experimentally analyzed. A surfactant (Tween 20) has been added to the non-Newtonian solutions in order to improve the stabilization of the emulsions. The geometrical characteristics of the droplets were analyzed by means of a specific code developed in Matlab. When \( Q_p \) is lower than 2 ml/h, the squeezing mechanism is responsible of the droplet generation. Two different flow patterns can be observed linked to the squeezing mechanism: DCJ (droplet formed at the cross-junction) and DDC (droplet formed downstream of the channel). For large values of the flow rate ratio (\( \alpha > 2 \)) the droplet generation is ceased and a stratified parallel flow (PF) is observed. By increasing the flow rate associated to the continuous phase (\( Q_c > 2 \) ml/h), the squeezing mechanism tends to be replaced by dripping and jetting. In dripping and jetting regimes an elongated microthread connecting the droplet to the core of the dispersed flow at the inlet of the junction is observed which is responsible for the formation of a smaller satellite droplet. The experimental data confirm that the dimensionless droplet diameter in dripping and jetting regimes depends weakly on the flow rate ratio (\( \alpha \)) and they are a function of the Capillary number. On the contrary, in squeezing regime Capillary number loses its importance and \( \alpha \) influences significantly the dimensions of the droplets. In fact, in squeezing regime since the junction cross-section is almost blocked, the droplet size depends on channel size and \( Q_p \) only. If the Xanthan gum concentration is increased, an increase in the viscosity of the dispersed phase is introduced and an increase in the droplet size is evidenced at the same Capillary number (\( Ca_{\alpha} \)) and flow rate ratio \( \alpha \).

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