GAS-LIQUID DROPLET MICROFLUIDICS UNDER CONFINED 3D FLOW-FOCUSBING GEOMETRIES FOR DROPLET GENERATION UNDER THE JETTING REGIME

A Thesis Presented

By

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The purpose of this thesis is to study an alternative method for droplet generation using in-air confined flow focusing microfluidic chips. Conventional methods for droplet generation use oil as the continuous phase in a microfluidic chip to generate highly uniform droplets. Previous studies proved the possibility of using highly inertial gases as the continuous phase to generate liquid droplets. Other studies have generated liquid droplets by dispersing the liquid in an open gaseous environment using non-microfluidic formats.

This work investigates a novel method for droplet generation within the Dripping and Jetting regimes in a confined microchannel. It identifies relationships between the geometry of the microchannel and the flow rates in terms of dimensionless numbers to then relate this to the physical characteristics of the droplets and the jet.

This study uses multilayered 3D microfluidic chips using SU8-photolithography to fabricate the mold to later replicate it in PDMS. Several geometries are fabricated to study the influence of air, liquid and output channels width and height as well as the size of the neck before the output. These parameters are studied against each other to understand their physical meaning over the jet formation and its resulting droplets. The result of this experiment is represented in several flow regime maps which allow to explain the physical requirements for the Jetting to occur and how different parameters affect it.

A study of the passive control of the droplet generation is made. Indicating that is possible to control the droplet size and generation frequency by adjusting the flow rates of the
continuous (gas) and the dispersed (liquid) phases. For the Dripping regime results are obtained with frequencies in the order of 10kHz and droplet sizes between 160 µm and 50 µm. On the other hand, for the Jetting we obtain droplets between 50 µm and below 15 µm at frequencies higher than 100 kHz.

The outcomes of this work are useful in many areas, namely pharmaceutical and food industries where uniform droplets can be generated purely in air and without any extra liquid contaminants. Also, the ability to create these droplets within a confined microchannel allows for additional modules for developing integrated Lab-on-a-chip platforms suitable for material synthesis and nanotechnology.
1 INTRODUCTION

Droplet generation microfluidics has become a predominant tool in many applications. This technology is used in many areas such as biological, pharmaceutical, drug delivery and food industry. These types of applications have generally required a continuous stream of micron-sized droplets with nanoliter scale volumes. Some of the advantages are its ease to isolate elements like solids, cells or microorganisms into droplets, the ability to produce a massive number of analyzable droplets and the possibility to integrate this technology with other lab-on-a-chip solutions to create multi-step systems both for research and industry. Droplet based microfluidics fulfill these requirements.

In these microfluidic systems the droplet is generated by the flow of two immiscible solutions, a continuous phase surrounding an inner phase which breaks into discrete droplets. The breakup mechanisms may vary depending on the breakup source and the flow regime of both phases. This source can be passive or active. The passive droplet generation systems achieve breakup without external sources, meaning that the breakup is due to surface tension, viscous and inertial effects as well as instability propagation. The active method category encompasses any system that uses an external source to add the required energy to generate the breakup into droplets. This external source can be mechanical, pneumatic, electrical or even electroacoustic among other methods [1], [2].
The most commonly used droplet microfluidics set-up is oil-based. This technique uses oil as the continuous phase and an aqueous solution as the discrete phase. In general applications the liquids exhibit relatively high viscosities. As a consequence, together with the small channel dimensions these flows are characterized by a very small Reynolds number, meaning a highly viscous flow where the inertial effects are negligible. This highly viscous flow makes the flow regimes of oil-based droplet generation systems easier to characterize. Oil-based microfluidic droplet generation has been studied extensively, with publication reporting good controllability and monodispersity[3]. Regardless of its advantages, the use of oil has several shortcomings. For example, it requires the use of surfactant to avoid coalescence and increase the shelf life of the droplet, but this makes it hard to recycle the oil. Additionally, separating the existing oil-immersed droplets generally requires additional multichip steps and this may present an additional challenge.

In-air microfluidics appears as an alternative to oil based microfluidics. This technique uses air instead of oil as the continuous phase. In-air microfluidics allows droplet generation in processes that require the interaction with gaseous phases such as chlorination, oxidation and hydrogenation. The gaseous phase allows the system to bypass the use of oil and eliminates additional steps the for cleaning and separation. In general, this method uses less viscous continuous phase fluids and at higher velocities, meaning its Reynolds number is higher. These high Reynolds numbers mean inertial effects cannot be neglected[4].
Previous works in gas-liquid droplet microfluidic generation have identified different flow regimes: Co-flowing, Threading, Plugging, Dripping, Jetting and Multi-satellite droplet formation. For the scope of this paper, the Dripping and Jetting regimes are relevant[5].

In the Dripping regime the droplet breakup occurs due to a pinch-off mechanism when the viscous force pulling the droplet is greater than the surface tension force holding it. This effect is easily characterized by the capillary number. This regime has high droplet size together with a relatively low droplet generation frequency; however, it has a very high monodispersity index. This means all droplets generated are almost the same size.

On the other hand, the Jetting regime generates the droplets by the breakup of a coflowing liquid jet surrounded by a continuous gas phase. The breakup of the liquid jet is due to the instability propagation along its length. This break-up is usually described by the Rayleigh-plateau instability analysis. This analysis studies the breakup of a liquid jet with no interactions from the surrounding media. Other instability analysis has studied the jet breakup using different assumptions. Tomotica analyzes the co-flow of two viscous fluids and Webber analyzes the breakup of a jet surrounded by viscous media.

Some of the in-air microfluidic methods like the one proposed by Gañan-Calvo shoot a liquid jet into a quiescent fluid (environmental air)[6]. These methods are well characterized and studied and are useful for a variety of applications like polymer 3D printing and coating. But sometimes complex systems require multi stage chips to perform a series of processes, requiring the droplet generation to occur in a confined geometry. Confined in-air droplet microfluidics has been studied far less than oil-based
droplet microfluidics and unconfined in-air droplet microfluidics, mostly because of the physical complexity of the system increases with the gas velocity.

This work presents an in-depth study on the formation and breakup of a liquid jet surrounded by a coflowing gas stream inside a microchannel. We study the effect of individual geometrical parameters like the channel width, height and neck diameter on the droplet generation dynamics. The droplet generation dynamics are measured by jet diameter, droplet size, droplet generation frequency and monodispersity. Furthermore, it characterizes the system’s behavior in terms of dimensionless numbers that relate these variables to both the gas and liquid flow rates. These dimensionless numbers are represented by the weber of the liquid, Reynolds of the gas and a modified Reynolds number that represents the ratio of inertial force of the liquid and viscous force of the gas.

We expect these results will advance confined in-air microfluidic technology by providing a wider understanding of its physics and its consequences to find solution to mitigate its deficiencies and strengthen its advantages.
2 THEORY

A study on the model and theory for droplet micro fluidics is made with the goal of understanding its underlaying phenomena. Using simple physics and developed theory is possible to set a solid background for experimental analysis. The knowledge of this phenomena is key to improve existing droplet generation methods and implement systems for active droplet generation.

For an analytical study of the droplet generation, a Co-flow geometry is suitable to understand the physics. However, for most of our experiments we will be using a flow focusing geometry, which slightly differs from the co-flow geometry. For the Jetting regime most of the analysis is made further in the channel so it is valid for both geometries. The co-flow geometry is a channel where both fluids are flowing at different flow rates in the same direction. For our case the gas is surrounding the liquid. The liquid that flows in the inside and generate the droplets is called the dispersed phase and the fluid (gas) that is flowing surrounding (or encompassing) the liquid is called the continuous phase.

![Figure 2-1: Schematic for dripping within a microchannel](image-url)
2.1 THE DRIPPING REGIME

Although the scope of this work centers on the Jetting regime, it is important to mention the basic aspects of the Dripping regime.

We can characterize this flow regime by 2 dimensionless numbers. The capillary number and the weber number. The capillary number is the ratio of the viscous shear stress and the surface tension force

\[ Ca = \frac{\text{Viscous shear force}}{\text{surface tension force}} = \frac{\mu u}{\sigma} \]

\( \mu, u \) and \( \sigma \) are the viscosity of the fluid, its velocity along the channel and its surface tension.

The Webber number is the ratio between the inertial force and the surface tension.

\[ We = \frac{\text{Inertial Force}}{\text{surface tension force}} = \frac{\rho u^2 D}{\sigma} \]

\( \rho \) and \( D \) are the density and the diameter, respectively.

The ratio of these dimensionless number leads to the Reynolds number:

\[ \frac{We}{Ca} = Re = \frac{\rho u D}{\mu} \]

To describe a co-flow system, we use the capillary number of the continuous phase and the weber number of the disperse phase.
In the Dripping regime the breakup occurs due to a pinch off. This happens when the viscous drag forces overcome the surface tension forces of the fluid. When the droplet is small the surface tension dominates, and it breaks when the droplet is big enough so that the surface tension and viscous shear force (Drag) become comparable in magnitude.

\[ \sigma \cdot D_{\text{tip}} \sim \mu_{\text{out}} \cdot u_{\text{out}} \cdot D_{\text{drop}} \]

Then it is reasonable to assume that the droplet size scales as:

\[ D_{\text{drop}} \sim D_{\text{tip}} \cdot \frac{\sigma}{\mu_{\text{out}} \cdot u_{\text{out}}} = D_{\text{tip}} \cdot \frac{1}{Ca_{\text{out}}} \]

This behavior has been observed in many experimental studies.

As it was shown, the viscous drag is exerted by the continuous phase and the shear stress force come from the continuous phase.
If the ratio of flow rates remains the same but both flow rates increase the droplet size will remain the same but instead its generation frequency will increase until the inertial effects become prevalent[7].

\[ \text{Generation frequency} = f \left( \frac{Q_{\text{in}}}{Q_{\text{out}}} \right) \]

2.2 **The Jetting Regime**

As mentioned earlier, increasing the flowrates of the continuous phase leads to a decrease in droplet size until it is more stable and its diameter is small enough to form a cylindrical jet instead of Dripping small droplets at very high speeds.

![Schematic of a jet break-up](image)

*Figure 2-4: Schematic of a jet break-up*

This cylindrical jet will breakup further downstream to form very small droplets. The breakup of this jet is due to the phenomena that is called the Rayleigh Plateau instability.

2.2.1 **Rayleigh plateau instability**

This instability first described by Plateau and later studied by Rayleigh [8] considers infinitesimal varicose perturbations, which normally come from external factors and
cannot be totally controlled. Their theory evaluates how this perturbations growth until the jet breaks up. This approach neglects the density of the continuous fluids and its viscosity and therefore the existences of friction between both fluids.

The formulation by Rayleigh states that the instability maximums further amplify, and the minimums further decrease until they disappear, and the break-up occurs.

We assume a cylindrical jet with a straight interphase that breaks up into droplets further down the stream, after the perturbations take place.

*Figure 2-5: Schematic of the process of a jet break-up.*
As observed in Figure 2-5 the pressures can be balanced for the point A and B.

\[ R_B < R_A \]

\[ P_B - P_{out} \sim \frac{\sigma}{R_B} \]

\[ P_A - P_{out} \sim \frac{\sigma}{R_A} \]

The fact that \( P_B > P_A \) starts a positive pressure gradient that drives from point B towards point A. Because of this the fluid accumulates at A and the droplets are created.

It is important to understand the mechanics of the instability and how they propagate through the flow. The instability analysis done by Rayleigh allows us to calculate the wavelength of the most unstable perturbation \( \lambda_D \) in terms of the jet diameter \( D_j \), and from the wavelength we can calculate its expected droplet sizes.

\[ \lambda_D = \pi \sqrt{2} D_j = 4.4 D_j \]

This result allows us to characterize the behaviour of the droplet in terms of the jet diameter which is easy to obtain experimentally.

Although this model has some significant simplifications and probably cannot represent the full extent of the physical phenomena in our system, it is a valuable tool as a first approach analysis.
2.2.2 Estimation on the droplet size and frequency

We can assume that both fluid flows have the same velocity and use the ratio of the flow rates and its relation to the Jet diameter and channel size to estimate the droplet size in a confined channel:

\[
\frac{Q_{in}}{Q_{out}} \approx \frac{A_{in}}{A_{out}} = \frac{D_{jet}^2}{D_{channel}^2}
\]

The droplet diameter scales as the square root of the ratio between the continuous phase and the disperse phase flow rates.

\[
D_{drop} \approx f(D_{jet}) \approx D \sqrt{\frac{Q_{in}}{Q_{out}}}
\]

From this result we can infer that increasing the flow rate ratio will increase the droplet diameter and decreasing it will generate smaller droplets.

Although the previous studies done by our group [5] have analyzed the flow map and conditions where the Jetting flow regime occurs, this project focuses on further investigating the physics of the Jetting.
To estimate the frequency $f$, we can use the estimation obtained from the Raleigh-plateau instability analysis assuming that the droplet always breaks in the most unstable wavelength.

$$f = \frac{u_{jet}}{\lambda_D}$$

$$u_{jet} = Q_{in} \cdot \frac{A_{in}}{A_{jet}} = Q_{in} \cdot \frac{D_{in}^2}{D_{jet}^2}$$

And from the flow rate and the frequency it is possible to get an estimation of the droplet volume, and therefore the droplet size.

If we assume that all the droplets are uniform, we can assume that the volume of the droplet is equivalent to a cylinder with the diameter of $D_j$ and the length of $\lambda_D$. It is then
is easy to obtain an expected droplet diameter based on the Rayleigh plateau instability analysis.

\[
\pi (R_j)^2 \times 8.8R_j = \frac{4}{3} \pi (R_d)^3
\]

\[
1.87D_j = D_{drop}
\]

These results are interchangeable with each other and in experimental analysis it is sometimes easier to observe some of the physics directly and later correlate them. For example, sometimes is easier to obtain the drops and then study those drops to know the frequency break up and diameter of the jet.

It is also important to mention that in higher flow rates of the inner fluid, the inertial force overcomes the breakup due to Rayleigh instability, and the drag force is high enough to pull the liquid to the tip of the jet before breaking up. At this point another mode of jetting starts.
This regime results in droplet sizes bigger than the regular Jetting regime, and at the same order of magnitude as the Dripping regime. It presents high frequencies but also a high polydispersity index.

2.3 OTHER APPROACHES:

There are several more elaborated analyses on the stability of a liquid jet. While Tomotika[9] takes into account only the viscosity of the outer fluid, Guillot and Colin[10] and take into account the viscosities, the confinement, and velocity profiles in the system. Nevertheless the former approach is not applicable for a high viscosity ratios of $\mu_l/\mu_g \approx 50$ and the latter method requires a finite element analysis to be solved. For the scope of this thesis they will not be studied here.
3 METHODOLOGY

3.1 GEOMETRIES

The experimental design is based on the comparison of different geometric components which can be tuned: neck height, neck width, outlet channel height and outlet width. The variation of these components allows some secondary comparisons such as aspect ratio, neck area and outlet area.

Figure 3-1: This figure shows some of the basic dimension of the flow focusing geometry used for the studies presented in this thesis.
Figure 3-2: This figure shows a detail of the region of convergence of the flow focusing microchannel.

Figure 3-3 This image shows the dimensions along a longitudinal cut at the center of the channel cutting throughout the neck.

<table>
<thead>
<tr>
<th>Label</th>
<th>Description</th>
<th>Constant or variable</th>
<th>Length or range</th>
</tr>
</thead>
<tbody>
<tr>
<td>$L_{ai}$</td>
<td>Air inlets length</td>
<td>Constant</td>
<td>4000μm</td>
</tr>
<tr>
<td>$L_{li}$</td>
<td>Liquid inlet length</td>
<td>Constant</td>
<td>3000μm</td>
</tr>
<tr>
<td>$L$</td>
<td>Outlet length</td>
<td>Constant</td>
<td>5500μm</td>
</tr>
<tr>
<td>$L_n$</td>
<td>Length of the neck</td>
<td>Constant</td>
<td>20μm</td>
</tr>
<tr>
<td>$L_{nt}$</td>
<td>Length of the neck including the outlet region</td>
<td>Constant</td>
<td>100μm</td>
</tr>
<tr>
<td>$W_{ai}$</td>
<td>Air inlet width</td>
<td>Constant</td>
<td>80μm</td>
</tr>
<tr>
<td>$W_l$</td>
<td>Liquid inlet width</td>
<td>Constant</td>
<td>20μm</td>
</tr>
<tr>
<td>$W_n$</td>
<td>Width of the neck</td>
<td>Variable</td>
<td>25-90μm</td>
</tr>
<tr>
<td>$W$</td>
<td>Outlet width</td>
<td>Variable</td>
<td>50-190μm</td>
</tr>
<tr>
<td>$H_1$</td>
<td>Height of the liquid inlet and neck</td>
<td>Variable</td>
<td>76-140μm</td>
</tr>
</tbody>
</table>
The neck width and channel width are defined in the mask using computer assisted design software. The neck height and outlet height are defined in the microfabrication as will be explained in the next section.

It is important to mention the liquid inlet height is the same as the neck height because they are created in the same layer. This gives additional complexity for the experiment geometries because it means that only geometries with the same first layer will be equivalent systems.

<table>
<thead>
<tr>
<th></th>
<th>Height of the outlet and air inlets</th>
<th>Variable</th>
<th>50-349μm</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Layer 1</strong></td>
<td>Height of the first layer of the mold</td>
<td>Variable</td>
<td>25-70μm</td>
</tr>
<tr>
<td><strong>Layer 2</strong></td>
<td>Height of the second layer of the mold</td>
<td>Variable</td>
<td>8-80μm</td>
</tr>
</tbody>
</table>

Table 1 This table gives a brief overview on the characteristics of the geometrical parameters seen in the previous figures.

3.2 Fabrication

Previous studies used 2D geometries where the geometry is a planar extrusion with constant height and all features are defined in the planar sketch. The mold was fabricated using SU8 photolithography [11], [12] which was then replicated using PDMS. This chip was then attached to a glass slide that was sometimes PDMS coated.

The fabrication of the 3D microfluidic chips is based on the creation of a multilayered silicon wafer mold using SU8 photoresist[13]. This requires creating two layers of SU8 with different heights. The first layer has the full geometry which includes the complete
features of the channel. The second layer removes the neck and the liquid inlet features. This means that these features have only the height of the first layer, allowing the design of 3D features along the channel. It is necessary to print two different masks, one corresponding to each layer.

![Diagram](image)

*Figure 3-4: Simplified schematic of how the different layers form the halve of the geometry of SU8 on the silicon wafer.*

![Image](image)

*Figure 3-5: Example of the mask used for the SU8 photolithography process. The image of the left is the first layer with all the features. The image on the left is the second layer which omits the liquid inlet and neck section to make a 3D geometry.*
Table 2 Description of the steps involved in the creation of a multilayered mold on a silicon wafer using SU-8 photolithography for the creation of 3D flow focusing geometries.

Fabricating the mold requires two different SU-8 photolithography processes. The steps follow coating, soft bake, exposure and post-exposure bake for the first layer. The second layer coating is deposited on top of the first layer and it follows the same steps, except for additional alignment before exposure.

One example for the creation of a 100µm outlet channel with a first layer of 40µm and a second layer of 10µm.
### Table 3 Example of a specific photolithography process for the creation of a mold with a first layer of 40μm and a second layer of 10μm

<table>
<thead>
<tr>
<th>Layer</th>
<th>Steps</th>
</tr>
</thead>
</table>
| **1st layer 40μm** | - Coating: 40 μm SU8-50 10 seconds @500 rpm + 30 seconds @ 3000 rpm  
- Softbake: 5 min @ 65° + 15 min @ 95°  
- Exposure: 40 seconds with the first layer mask on top  
- Post exposure bake: 5min @65° and 10 min @95° |
| **2nd Layer 10μm** | - Coating:10 um SU8-10 10 seconds @500 rpm + 30 seconds @ 2000 rpm  
- Softbake: 3 min @ 65° + 5 min @ 95°  
- Alignment: An aligner is used to line up the features of the second layer mask with the 1st layer on the wafer.  
- Exposure: 27 seconds with the mask hold on the aligner  
- Post exposure bake: 5 min @65° and 15 min @95°  
- Development: The wafer is submerge in an SU8 developer solution around 8 minutes to develop both layers at the same time |

The mold becomes the negative of the channel, which is later replicated from the mold using PDMS.

Every mold has around ten geometries that have different planar section features but the same individual height in each of its two layers. To create the microfluidic chips, two PDMS replicas must be created for the mold. Both replicas are cut. The upper replica is also punched with holes for the tubing connections.

It is necessary to clean both replicas thoroughly with IPA and DI water to prepare them for plasma bonding[14]. Plasma bonding is a process using plasma to remove the hydrocarbon groups. This promotes the adhesion of SI-O-SI covalent bonds between the PDMS surfaces. After the plasma treatment, a drop of water is set between the replicas,
which are stacked to form a complete microchannel. The alignment process is done using a microscope. This step is crucial because it aligns the geometries to create the flow focusing microchannel.

![Unaligned replicas facing each other after the plasma process.](image)

*Figure 3-6: An example of the aligning process in the microscope at 5x. The image on the left shows the original unaligned replicas facing each other after the plasma process. The image on the right shows the final 3D microchannel after the alignment of the replicas.*

![Symmetry Axis](image)

*Figure 3-7: This schematic represents a cut along the longitudinal axis for the final 3D flow focusing geometry. It shows the symmetry both related to the replicas and the layer of the mold.*
Despite its increased fabrication difficulty, the 3D chip geometry allows for variations in height as well as variations in the X and Y axis. With this geometry it is also possible to create PDMS on PDMS chips, giving the chip complete symmetry on the longitudinal axis, with complete PDMS surroundings.

This fabrication methodology allows the creation of several geometries aiming to create a parametrical study of the geometries. The original design assumed the fabrication process was very precise and therefore it was focused on square geometries. Nevertheless, some additional fabrication difficulties appeared creating an important difference between the expected thickness of the geometries and the ones obtained as illustrated in Figure 3-8.

![Figure 3-8: Figure of the cross-sectional area the red dashed line represents the obtained cross-section and the blue line represents the expected cross-section.](image-url)
After a qualitative analysis of the possible causes of the difference between the expected and obtained geometries. Most the evidence point to the age of the SU-8 photoresist used in the fabrication. Most of this photoresist age more than 10 years and their used before date is at least 5 years past due. Other possibility is the difference between the adhesion in SU-8 over SU-8 and SU-8 and silicon this difference might generated different layer thicknesses for the coating. Further studies should be made to debug this issue for future experiments. For the case of this thesis the experiment correlation was modified and adapted to the new geometries. These new experiments go from a focus on squared geometries to rectangular geometries and square geometries but with less detail than originally planned.

![Figure 3-9: An example of the expected geometries and the obtained geometries, the case on top is more severe than the case below.](image)

### 3.3 Experimental Set-up

The experimental set-up consists of a pressure controller followed by a mass flow meter. The pressure controller creates a pressure induced flow which is then reported by the
mass flow meter. The gas line is then connected to each gas inlet. The mass flow meter measures a volume flow rate in sccm (standard cubic centimeters per minute) which then is adjusted considering the compressibility (adiabatic compressible flow in ducts). This becomes the flow rate at the jetting entrance in cm^3/min.

The liquid flow rate is set up using a syringe pump that injects a constant DI water flow rate. This line is connected to the liquid inlet and it directly provides the values in μl/min.

Figure 3-10 schematic representing the experimental set-up. It shows some of the main elements together with its interactions.

The PDMS microfluidic chip is connected to a reverse microscope with a light source above it. In this experiment the magnification ranges from 4x up to 15x. The reverse microscope is then coupled to a Photron highspeed camera which can achieve frame rates of 150000 FPS. This is captured using the Photron software to record the droplet generation phenomena inside the microchannel.
The recorded videos are divided into frames and analyzed using ImageJ software to obtain the measurements in pixels to then convert them to units of length. This step measures the following metrics: droplet diameter, jet diameter and jet breakup length.

It is important to mention the limitations of the experimental set up. The maximum controllable pressure is 20 PSI and the maximum measurable gas flowrate is 500 SCCM. Other important limitations are optical. As mentioned, the high-speed camera maximum recording frame rate is 150000 fps and sometimes the droplet generation frequency is considerably higher than that. Also, the light source power limits the maximum magnification used at high shutter speeds. The high shutter speeds are not only required for high sampling rates but also for obtaining an acceptable contrast.
4 EXPERIMENTS

4.1 INTRODUCTION TO FLOW MAPS

Oil-based systems are characterized using the capillary number $Ca = \frac{\mu U}{\sigma}$, which describes a ratio between the viscous force and the surface tension force of two immiscible fluids[15]. The capillary number for liquid-gas systems loses importance in the dynamics due to the extremely low values for gas viscosity. This means the capillary number does not provide a good description of the physical phenomena involved in these types of systems. Furthermore, in the Jetting regimes the inertial forces gain importance because this regime occurs at very high speeds.

As mentioned earlier, the Reynolds number represents the ratio between inertial and viscous forces. It also has a known transition between laminar and turbulent flow around $Re_{trans} \approx 2300$. Therefore we use the Reynolds number to describe the flow conditions of the air in the system. To parametrize the liquid break process, we use the Webber $We = \frac{\rho U^2 D_h}{\sigma}$, number which represents the ratio between inertial forces and surface tension forces in a fluid.

The systems studied are liquid-gas systems where air is used as the gaseous phase and distilled water as the liquid phase. It is important to mention that the gas flow regimes interact in some mildly compressible regimes due to the high air velocities involved in this study. These effects are taken into account by modeling the gas flow as adiabatic compressible flow in ducts.
write a brief explanation of the physics and the assumptions.

The flow maps of this paper are characterized by the Weber of the liquid $We_{liq}$ in the $y$ axis and the Reynolds of the gas $Re_{gas}$ in the $x$ axis. The experiment was made by injecting a constant stream of liquid with an injection pump in the range of 0-700 µm, together with a gas flow induced by a pressure controller. The gas flow is then measured by a mass flow meter in the range of 0 to 500 SCCM. Both of these fluids are connected to a flow focusing geometry.

![Figure 4-1: Location of where the dimensionless numbers are calculated at the geometries.](image)

These stem outputs of $We_{liq}$ 0-9 and $Re_{gas}$ 0-4500 depending on the geometries of the liquid inlet and the output channel respectively.

This experiment was realized in several 3D chips with different geometries. These flow maps give an overview of the regimes and how they behave in terms of $We_{liq}$ and $Re_{gas}$. This enables direct observation of how some characteristics of the geometry affect the regions where certain regimes are present. The difference and similarities where these regions are present gives an opportunity to study the influence of different geometrical
parameters in the formation of a stable and monodisperse Jetting regime. The region
where this paper focuses.

4.2 Flow Map Regimes Overview

Previous studies have presented mapping for the 2D microfluidic chips [4] focusing in all
the regions. Due to the characteristics of the geometries achieved by the 3D chips is
possible to extent the regions of study due to the enhanced stability at both higher and
lower $We_{liq}$ and $Re_{gas}$. Although this paper reports several regions such as Dripping,
Jetting and Co-flow. It focuses mostly in the Jetting regime and its variations. This means
that the flow-map studies the full scope of the Jetting regions but only study the
boundaries and regions close to the Jetting regime in for other flow regimes.

4.2.1 The Dripping Regime

In the Dripping regime the breakup is generated by the shear due to the viscous forces
exerted over the discrete phase (water) by the continuous phase (air). The regime
presents a growth of the droplet until the viscous shear exceeds the surface tension forces
holding the liquid together.

The region for dripping to happen is at both low $We_{liq}$ and $Re_{gas}$. This regime has low
breakup frequencies and high droplet sizes. In which the droplet size is related to the
microchannel sizes. This regime presents very high monodispersity meaning that the
droplet size remains mostly constant at a constant $We_{liq}$ and $Re_{gas}$. This allows this
regime to offer a precise control to create droplets in the order of 100 μm. Increasing the
$Re_{gas}$ will start decreasing the droplet size.
When the flow rate of the gas increases the $Re_{gas}$ increase consequently. The droplet size becomes less than the capillary length of the tip making a jet the stable regime.

4.2.2 The Jetting Regime

When the jet is formed it develops until it forms a constant crossectional jet. This developed jet goes to breaks in to droplets further downstream. Depending on the characteristics of the flow these droplets might be monodisperse and polydisperse.

In the optimal segments of the Jetting regime the droplets formed are in the order of 10μm and its generation frequencies are in the order of 100 kHz.
The Jetting regime arises a transition when the flow rate of the disperse phase increases at high $We_{liq}$. At this state the higher order terms and the drag become important due to the diameter of the jet and the importance of the inertial terms. The breakup mechanism transitions to the absolute instability to convective instability and the inertia of the jet is the main momentum supplier for the jet. Due to the viscous drag the regime show a jet that is increasing in diameter until a droplet breaks of it.

Figure 4-4 Example of the Jetting regime at high Webber numbers for the liquid.

4.2.3 Oscillating Jetting

When the inertial effects start increasing at high $Re_{gas}$ the stability of the liquid jet is affected by the turbulent components existing on the flow. The breakup length of the jet starts oscillating forward and backward and some secondary droplet breakups start appearing more often such as satellite droplets, this creates a regime with very high generation frequencies where the stability of the system and monodispersity of the droplets is compromised.
4.2.4 Unstable Jetting

When the turbulence effects drive completely the gas flow the liquid jet starts oscillating and shooting a constant stream of very small droplets at its tip at very high generation rates. Still in a confined geometry generally these droplets hit the wall and that makes hard to obtain the metrics of them such as droplet generation frequency and diameter.
4.2.5 Jet attached to the wall

In small geometries the jet does not have space to flow and therefore it hit and attaches to the wall. Then it exits at the other end of the microchannel without never breaking. It tends to move with time changing its path slightly but always flowing to the microchannel exit without breaking.
4.2.6 Flooded

This region is present at low $Re_{gas}$. It occurs when the air flow is not capable of clearing the liquid on the microchannel. Once the air flow increases, the flow regime will transition to Plug flow and Dripping at low $We_{liq}$ and Jetting at higher values.

![Figure 4-8: Flooded microchannel](image)

---

<table>
<thead>
<tr>
<th>$Re_{g}$</th>
<th>Outlet</th>
<th>$We_{l}$</th>
<th>Neck</th>
</tr>
</thead>
<tbody>
<tr>
<td>90</td>
<td>100x118</td>
<td>0.477</td>
<td>90x80</td>
</tr>
</tbody>
</table>

4.2.7 Spray

This regime is present at low $We_{liq}$ and high $Re_{gas}$. In this regime the high gas inertia completely drives the flow. The low mass of the fluid causes it to disintegrate in many small formations in a non-structured pattern.

![Figure 4-9: Spraying microchannel](image)

---

<table>
<thead>
<tr>
<th>$Re_{g}$</th>
<th>Outlet</th>
<th>$We_{l}$</th>
<th>Neck</th>
</tr>
</thead>
<tbody>
<tr>
<td>344</td>
<td>50x48</td>
<td>0.68</td>
<td>NA</td>
</tr>
</tbody>
</table>
4.2.8 Multi-Satellite formation

When the Reynolds of the gas is moderate but the Webber of the liquid is very low, the liquid attempts to form a jet which breaks into several small satellite formations. This cycle of formation and break-up occurs continuously in this flow regime.

![Multi-satellite formation](image)

<table>
<thead>
<tr>
<th>Reg</th>
<th>73</th>
<th>Outlet</th>
<th>50x48</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wei</td>
<td>0.08</td>
<td>Neck</td>
<td>NA</td>
</tr>
</tbody>
</table>

*Figure 4-10: Multi-satellite formation flow regime.*

4.2.9 Plug Flow

This regime is present at low values for both $Re_{gas}$ and $We_{liq}$. It occurs when the liquid fills the microchannel and the air starts moving the plugs of liquid across. Every plug is approximately the same volume for given set of $Re_{gas}$ and $We_{liq}$.

![Plug flow in the microchannel](image)

<table>
<thead>
<tr>
<th>Reg</th>
<th>218</th>
<th>Outlet</th>
<th>190x349</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wei</td>
<td>0.46</td>
<td>Neck</td>
<td>NA</td>
</tr>
</tbody>
</table>

*Figure 4-11: Plug flow in the microchannel*
4.3 **Flow Maps Experimental Results**

As mentioned in previous sections, several experiments were designed to study the influence of the neck, outlet area, and aspect ratio for droplet generation purposes. The quantity of experiments was handicapped by the variance of the geometries. Consequently, three experiments were performed. Two of which study the influence of the neck in small and large geometries. The third geometry is focused on the influence of the area at similar aspect ratios. Other experiments were performed but still incomplete by the time of the making of this thesis.

The flow maps are a marking of the regions where different flow regimes are present in terms of the Reynolds of the gas and Weber of the liquid. These maps allow to study the shift of the boundaries of the flow regimes and the influences of the variation of isolated parameters.

4.3.1 **Cross Sectional Area Effects**

This experiment focuses on rectangular geometries with similar aspect ratios and decreasing areas. These geometries have the same liquid inlets. The area decrease is close to 2.5 from geometry to geometry.
Figure 4-12: Geometries with similar aspect ratios for the decreasing area experiment. The geometries are referred from left to right as: A(190um x 349um), B(100um x 245um) and C(50um x 195um).

Figure 4-13: Flow map for geometry A
Figure 4-14: Flow map for geometry B

Figure 4-15: Flow map for geometry C
As easy observable, a decrease in the area initially enhances the Jetting region significantly. Although a decrease in the area also mean that the jet needs to be more centered and stable because of the possibility of hitting and getting attached to the channel walls. As seen in the smallest geometry the Jetting region gets limited by smallest dimension of the microchannel. This decreases the performance of the Jetting region both in terms of higher liquid flow rates (high weber numbers) and high gas flow rates (high Reynolds numbers).

The reason for this might be that with higher inertial components both for the liquid and the air, the jet position is less stable and with a strong confinement this means that is easy for the jet to find the wall. Once the jet hits the wall it stays attached to the wall.

4.3.2 Neck Effects in Rectangular Cross Sections

This experiment was performed with the goal of studying the influence of the neck in large geometries the original geometry is a large flow focusing microchannel with no neck. The other two geometries have a neck as seen in the geometries section.
Figure 4-16: Geometries with similar aspect ratios for the decreasing area experiment. The geometries are referred from left to right as: A(190um x 349um; Neck: NA), B(190um x 349um; Neck: 90um x 126um) and C(190um x 349um; Neck: 70um x 126um).

Figure 4-17: Flow Map for geometry A
Figure 4-18: Flow Map for geometry B

Figure 4-19: Flow Map for geometry C
For the following case we can observe the neck’s impact is moderate for the second geometry in terms of enhancing the Jetting region in comparison with the first geometry. Nevertheless, there is an appreciable increase of the Jetting region for the smallest neck.

For both cases with neck there is an appearance of an oscillating jet region, this region starts showing some oscillating behavior due to the inertial term. The position of the jet oscillates but it is still bounded the same applies for the breakup length which starts to oscillate periodically in the longitudinal direction. Regardless of that this regime still gives usable small droplets at very high generation frequencies. In contrast with the no neck geometry where there is no transition to the completely unstable Jet which is a non-usable scenario due to its chaotic nature.

4.3.3 Neck Effects in Square Cross Sections

This experiment differs from the previous one because it focuses on small quasi-squared geometries. Similarly, the neck influence is studied.

Figure 4-20: Small square geometries. The geometries are referred from left to right as: A(100um x 118um; Neck: NA), B(100um x 118um; Neck: 90um x 80 um) and C(130um x 118um; Neck: 70um x 80um).
Figure 4-21: Flow Map for geometry A

Figure 4-22: Flow Map for geometry B
In this experiment the first flow focusing geometry with no neck exhibits a behavior where obtaining Jetting is not possible and most of the chip scenarios are non-favorable possibly due to the small size of the microchannels. Once the neck is added the microchannel develops Jetting regime areas. This might be due to the neck acting as a high acceleration region which centers the jet before it releases in the outlet channels. The region is further enhanced for even smaller neck allowing a wider range of droplet generation frequencies and size to be obtained.

In comparison with the large rectangular geometries the neck influence is substantial for small square geometries and it is easier to observe and analyze its role.
4.4 ADDITIONAL FLOW MAPS DATA ANALYSIS

Other experiments where designed and performed for these geometries. Although by time of the making of this thesis they’re not performed for all the available data points marked on the flow maps.

This section is meant to showcase this additional obtainable metrics from the obtained videos with its correspondent flow map datapoints and how is possible to calculate additional droplet generation related metrics with video analysis.

Each experiment data point has a video associated these videos allow to obtain different measurement in terms of pixels. The preliminary results shown in this thesis are just a rough approximation using a limited number of frames and droplets an calculating its size manually using the ImageJ software to obtain the size of the droplets in 4 random frames and using an average of them.

The following figures are a preliminary approximation of the planned studies for the analysis of droplet generation physics.

The first two studies show a jet diameter analysis based in the physics of the system the force interactions between the liquid jet and the air are expected to be between the inertial forces of the air and the inertial force of the fluid. Therefor we are proposing a modified Reynolds number to estimate the jet diameter in terms of these forces $Re_{mod} = \frac{\rho l V^2 D_h}{\mu V h} = \frac{\text{Inertial force of the fluid}}{\text{viscous force of the fluid}}$. In the Y axis the jet diameter is being scaled against the
channel diameter as a dimensionless number for the first experiment and against neck diameter for the second experiment.

The fit is not perfect mainly because there might be a necessity for statistical data of the data points. Also, more studies must be done to find a better scaling both for the x axis and the y axis.

Figure 4-24: Analysis on the jet diameter against a modified Reynolds for the large rectangular geometries in terms of the outlet channel.
Figure 4.25: Analysis on the jet diameter against a modified Reynolds for the large rectangular geometries in terms of the neck.

\[ Re_{mod} = \frac{\rho l V_l^2 D_h}{\mu V_g} \]

Figure 4.26: Analysis of the droplet diameter in terms of the jet diameter also the yellow line represents the break-up based on the Rayleigh jet break-up mechanism for comparison purposes.

The goal is to make this analysis with a significant higher population both for statistical analysis of the data points and more datapoint and experiments.
4.5 Video analysis

Within the time of this thesis a more accurate measurement methodology has been developed. A series of algorithms have been developed in MATLAB to measure several droplet generation metrics. These metrics are jet diameter, droplet diameter and jet break up length. The algorithms coded required the video to be exported as series of .png images, one for each frame of the video. When the frames are selected a mask is created (same mask for every frame) and the video analysis performed. Every one of the metrics mentioned has a different algorithm and mask region related for its calculation.

In general, after the creation of the mask all algorithms follow the following preprocessing steps.

<table>
<thead>
<tr>
<th>Algorithm</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Define Threshold</td>
<td>Define a threshold that defines which intensity represents 0 and 1 in the grayscale image. This is defined using a matlab integrated function but it can also be done manually.</td>
</tr>
<tr>
<td>Binarize</td>
<td>This step creates a binary matrix from the grayscale image.</td>
</tr>
<tr>
<td>Complement</td>
<td>This step exchanges the 0's and 1's in the matrix. The obtained image will be an image where the desired measure (jet or droplet) will be a matrix segment of ones (white) where the remaining parts of the matrix are zeros (black).</td>
</tr>
<tr>
<td>Fill and filter</td>
<td>This step is made to homogenize the regions where glare is present creating fake dark regions in the matrix.</td>
</tr>
</tbody>
</table>

Table 4: Basic image preparation using the MATLAB image processing toolbox

The specific algorithms vary for each metric and depend on how the calculation is performed.
4.6 JET DIAMETER

With an image where the jet is represented as a black stripe of a given thickness. A code is written which measures the height of the stripe column by column. For every image the average of height is calculated, and this is done for every frame. This gives us an average jet diameter in time and space.

The jet diameter algorithm works for all the regimes where the jets exist and does not have blur related issues because the jet is semi-stationary along time. The only major limitation is the contrast quality in the source video, but for most of the videos this is not an issue.

4.7 DROPLET DIAMETER

This algorithm uses a MATLAB built-in function which allows to find circles within the matrix. This function has a sensibility input parameter which can select how strict has to be the circle defining criteria. Meaning that is easy to tune it for the droplets to be recognized. Using these algorithms, the radius of the droplets is obtained and then put into an array for statistical analysis.

This algorithm can recognize all the droplet sizes. Its main shortcoming is the appearance of highly deformed droplets at high Webber numbers which sometimes haven’t achieve a circular shape in the sampling region. There is also a setup to define the contrast of the circle which allows to overcome any glare issue with the droplets.
4.8 Jet Break-up Length

This algorithm is on an earlier development stage and has several exceptions like diagonal or crooked jets. It works with straight jets and it calculates row by row the distance until where the jet is present. Finally, it takes the longest distance as the break-up length. This distance is calculated along time and can be not only average but also mapped to know the jet break-up length position in time.
5 CONCLUSIONS

State of the art oil-aqueous systems can achieve 100 microns droplets at generation frequencies of around 10 kHz. With air-water systems we achieved droplet sizes below 10 microns and droplet generation frequencies higher than 100 kHz. This system can be implemented in large scale to produce micron sized droplets with ease due to its long stability. These particles present higher cleanliness and require less post processing which is desirable for the pharmaceutical industry.

It is also important to mention that this technology bypasses the use of oil completely, reducing the environmental footprint of the system. By avoiding the use of oil, we significantly diminish the diffusion rates within the microchannel. This have the potential to positively impact applications such as biological macromolecule characterization and material synthesis.

The flow maps realized give an overview of the effect of feature variation in the region of the Jetting regime. For example, in the case of the neck it has a significant effect in small squared geometries but moderate in large rectangular geometries. The effect of the confinement is desired until a threshold where it stops the developing of the jet due to over-constraining in the microchannel. These experiments give an initial understanding that can be studied further with the use of additional metrics.

The study of the physical phenomena involved in the Jetting although complex is required to make liquid-gas systems a viable alternative. This thesis showcases some preliminary analysis which can be further enhanced and exploited using statistical analysis for the
experiments already made. Computational tools using the MATLAB image processing toolboxes were develop for measuring some of the important physical characteristics for droplet generation within the Jetting regime.
6 References


