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Determination of the entrance length for viscoelastic fluid flows in microchannels

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Abstract

Fluid flows at micro-scale have several scientific and commercial applications. One important example regards scientific interest in biological flows, particularly blood flow, which shows visco-elastic behavior. Knowledge regarding flow properties for visco-elastic fluid in micro-channels is then important to better characterise and more accurately study blood flow dynamics. One flow property worth analysing is the entrance length, which is studied in this thesis. Blood-analogue polymer solutions of varying visco-elastic character are used.

The polymer solutions were prepared and rheologically characterised under steady-shear and elongational flows. These tests provided the viscosity curve that was successfully fitted to a Carreau-Yasuda model, and the determination of the longest relaxation time of the polymer solutions, λ respectively.

The micro-channels used for the flow were also fabricated and characterised through a Scanning Electron Microscope (SEM).

The μ -PIV technique was used to determine the velocity profiles throughout the length of the channel in the range of $2.86 \leq Re \leq 45.71$, $0 \leq Wi \leq 9.4$. To be able to measure the velocity profiles further downstream from the inlet without losing track of the x positioning of the measurement, a methodology using reference points between μ -PIV capture areas is described and used.

Due to the relatively low range of Wi used and corresponding small elastic effects in the flow, no visible center-line velocity over-shoots and no velocity peaks near the channel walls were registered, which allowed the analysis of the entrance length to be based on the evolution of center-line velocity. A fitting function was adjusted to the measurements and used to determine the entrance length.

For measurements of the Newtonian solution, very little experimental error was registered, good repeatability was observed and data obtained regarding both the center-line velocity and the entrance length measurements matched results present in the literature. For measurements of the visco-elastic solution, the evolution of the center-line velocity seems to indicate systemic error, and shows little repeatability. The entrance length could still be calculated, but the obtained values were greatly inconsistent, with the only clear interpretation being flaws in the experimental data, which impeded the determination of the sought-after relation between the dimensionless entrance length, Re and Wi .

Keywords: Entrance length, Visco-elastic fluids, Micro-channels, μ -PIV.

Resumo

Os escoamentos de fluidos em microescala têm várias aplicações científicas e comerciais. Um exemplo importante é o interesse científico em escoamentos biológicos, particularmente o escoamento do sangue, que apresenta um comportamento visco-elástico. O conhecimento de propriedades do escoamento de fluidos visco-elásticos em micro-canais é desta forma relevante para caracterizar adequadamente e estudar com maior precisão a dinâmica do escoamento de sangue. Uma propriedade do escoamento que merece análise é o comprimento de entrada, que é estudado nesta tese. São utilizadas soluções poliméricas análogas ao sangue, com diferentes características visco-elásticas.

As soluções poliméricas foram preparadas e caracterizadas reologicamente em escoamentos de taxa de deformação constante e escoamentos elongacionais. Estes testes permitiram obter a curva de viscosidade, que foi ajustada com sucesso a um modelo Carreau-Yasuda, bem como determinar o maior tempo de relaxação das soluções poliméricas, λ respectivamente.

Os micro-canais utilizados no escoamento foram também fabricados e caracterizados através de um Microscópio Electrónico de Varrimento.

A técnica μ -PIV foi utilizada para determinar os perfis de velocidade em todo o comprimento do canal, na gama de $2,86 \leq Re \leq 45,71$, $0 \leq Wi \leq 9,4$. Para poder medir os perfis de velocidade mais a jusante da entrada sem perder a rastreabilidade da posição x da medição, é utilizada uma metodologia que utiliza pontos de referência entre as diferentes áreas de captura do μ -PIV.

Devido ao intervalo relativamente baixo de Wi utilizado e aos correspondentes pequenos efeitos elásticos no escoamento, não foram registados "overshoots" visíveis de velocidade no centro do canal, nem picos de velocidade perto das paredes do canal, o que permitiu que a análise do comprimento de entrada fosse baseada na evolução da velocidade no centro do canal. Uma função de ajuste foi aplicada aos resultados experimentais, e posteriormente utilizada para determinar o comprimento de entrada.

Nas medições com a solução Newtoniana o erro experimental foi muito baixo, tendo-se verificado uma boa reprodutibilidade, e os dados obtidos tanto em relação à velocidade da linha central como às medições do comprimento de entrada corresponderam aos resultados presentes na literatura. Para as medições da solução visco-elástica, a evolução da velocidade da linha central parece indicar a existência de um erro sistemático, e uma baixa reprodutibilidade. O comprimento de entrada ainda pôde ser calculado, mas foram obtidos valores muito incoerentes, apontando claramente para falhas nos dados experimentais, o que impediu a determinação da pretendida relação entre o comprimento de entrada adimensional, Re e Wi .

Palavras-chave: Comprimento de entrada, fluidos visco-elásticos, micro-canais, μ -PIV.

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Nomenclature

Abbreviations

CCD	Charge-Coupled Device
CEFT	Centro de Estudos de Fenómenos de Transporte
CMOS	Complementary Metal Oxide Semiconductor
DMSO	Dimethyl Sulfoxide
GNF	Generalised Newtonian Fluid
PDMS	PolyDiMethylSiloxane
PIV	Particle Image Velocimetry
SA	Sodium Azide
SDS	Sodium Dodecyl Sulfate
SEM	Scanning Electron Microscope
SRM	Slow Retraction Method
TMCS	TriMethylChloroSilane
XG	Xanthan Gum

Notation

$\Delta \vec{x}$	Difference between position vectors [μm]
Δt	Adjustment to show difference between solutions' break up times [s]
Δt_{pulses}	Time interval between laser pulses [μs]
$\Delta y_{measured}$	y range measured in μ -PIV [mm]
δz	Depth of field in μ -PIV measurements [μm]
$\dot{\gamma}$	Shear rate [s^{-1}]
$\dot{\gamma}_{wall}$	Shear rate at the wall of the channel [s^{-1}]
$\dot{Q}$	Volume flow rate [mL/h]
$\eta(\dot{\gamma})$	Viscosity as a function of the shear rate [mPa.s]

η_0	Viscosity as the shear rate tends to zero [mPa.s]
η_∞	Viscosity as the shear rate tends to infinity [mPa.s]
$\eta_{min}(\dot{\gamma})$	Minimum torque imposed limit for viscosity measurements [mPa.s]
$\eta_{secf}(\dot{\gamma})$	Secondary flow imposed limit for viscosity measurements [mPa.s]
η_{wall}	Fluid viscosity at the wall of the channel [mPa.s]
$\hat{R}$	Specific parameter of parallel-plate measuring systems
λ	Fluid longest relaxation time [ms]
λ_{CY}	Time constant in Carrau-Yasuda model [s]
λ_{laser}	Laser wavelength [nm]
μ	Constant fluid viscosity (Newtonian fluids) [mPa.s]
μ	Varying fluid viscosity (non-Newtonian fluids) [mPa.s]
$\bar{\gamma}$	Average flow shear rate [s^{-1}]
$\bar{\mu}_\infty$	Dimensionless infinite viscosity
$\bar{U}$	Average flow velocity [m/s]
ϕ	Rotational rheometer plate diameter [mm]
ϕ_{in}	Needle inner diameter [mm]
ϕ_{out}	Needle outer diameter [mm]
ρ	Fluid density [kg/m^3]
$\vec{u}$	Velocity vector [m/s]
A	Area of the channel's cross-section [mm^2]
a	Parameter in Carrea-Yasuda model
a_{exp}	Parameter in exponential decay fitting function for center-line velocity
AR	Aspect-ratio of the cross section
b	Auxiliary variable in the branched fitting function for center-line velocity
d	Parameter in the branched fitting function for center-line velocity
D_h	Hydraulic diameter of channel cross-section [mm]
D_0	Extensional rheometer plate diameter [mm]
$D_{min}(t)$	Minimum thread diameter at time t [mm]
El	Elasticity number
H	Channel height [mm]

h	Rotational rheometer plate gap [mm]
h_0	Initial extensional rheometer plate gap [mm]
k	Auxiliary variable in the branched fitting function for center-line velocity
L	General characteristic length of the flow [mm]
L_e	Entrance length [mm]
L_{ep}	Entrance length at which center-line velocity is $p\%$ of its fully-developed value [mm]
m	Parameter in the branched fitting function for center-line velocity
M_0	Rotational rheometer characteristic minimum torque [mN.m]
n	Parameter in different viscosity models
R	Rotational rheometer plate radius [m]
Re	Reynolds number
Re_{MR}, Re_{gen}	Reynolds number generalisations
s	Parameter in the branched fitting function for center-line velocity
T	Temperature [°C]
t	Time [s]
t_{exp}	Parameter in exponential decay fitting function for center-line velocity
U	Fluid velocity [m/s]
$U(y)_0$	Inlet velocity profile [m/s]
U_C	Center-line velocity [m/s]
$U_C^{exp}(x)$	Exponential decay fitting function for center-line velocity [m/s]
$U_C^{fit}(x)$	Branched fitting function for center-line velocity [m/s]
$U_{C,FD}$	Fully developed center-line velocity [m/s]
$U_{C,FD}^{exp}$	Fully developed center-line velocity for exponential decay fitting function [m/s]
$U_{C,FD}^{fit}(x)$	Fully developed center-line velocity for exponential decay fitting function [m/s]
V_{plate}	Extensional rheometer plate velocity [mm/s]
W	Channel width [mm]
Wi	Weissenberg number
y_C	y coordinate at the center-line of the width of the channel [mm]

Chapter 1

Introduction

1.1 Contextualisation

Microfluidics refers to the behavior, precise control, and manipulation of fluids that are geometrically constrained to a small scale (typically sub-millimeter) at which surface forces dominate volumetric forces [23]. At the microscale, fluid flow behaviour may differ from that of conventional macroscale. Flows at this scale have several scientific and commercial applications, which increases the interest and importance of the study of microfluidics [35, Chap.1]. Two examples are found in lab-on-a-chip devices [47, 13] and in the field of biomedicine [44, 18].

The flow behaviour of a viscoelastic fluid at microscale is then particularly relevant as the combination of high deformation rates, small time scales and small characteristic lengths results in an increased importance of viscoelastic effects in comparison to inertial effects. This can be better understood by looking into dimensionless relations.

Dimensional analysis is often used when dealing with experimental data. It consists of analysing flow characteristics in relation to each other through the use of dimensionless numbers. This allows for the experimental results to be generalised and reduces the complexity of experimental models [33, Chap.7].

In the field of fluid dynamics, one key dimensionless number is the Reynolds number (Re). This parameter compares inertial and viscous effects through the relation $Re = \rho \bar{U} L / \mu$, where ρ is the fluid density, $\bar{U}$ its velocity, μ its viscosity and L a characteristic length of the problem (for example in pipe flows, L is usually the pipe diameter) [33, Chap.7]. For non-Newtonian fluids, Re needs to be generalised, as μ is no longer a constant fluid property, and depends of the flow conditions. There are different ways to do this, each with advantages and disadvantages depending on the type of problem at hand. Some ideas include using the viscosity for characteristic shear rates or at the wall, or basing the Reynolds number as a function of pressure drops observed along the channel [41]. This thesis uses Re with the wall viscosity. A more in-depth discussion can be found in Subsection 2.4.2.

More specifically for viscoelastic flow, the Weissenberg number (Wi) gains importance. Whereas Re compares inertial and viscous effects, this number instead compares elastic and viscous effects

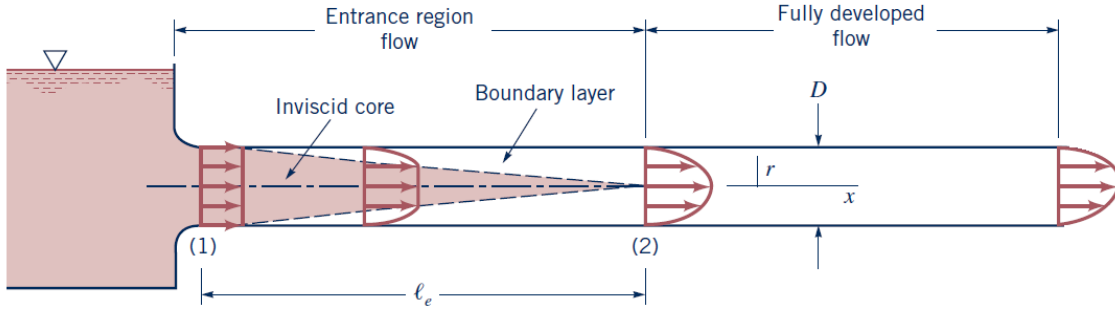


Figure 1.1: Illustration of entrance region in a pipe system. Adapted from [33, chap. 8]

[40]. It can be defined as $Wi = \lambda \bar{\gamma}$, where λ is the fluid relaxation time and $\bar{\gamma}$ is the average shear rate of the flow in the channel. Finally, a relation between elastic and inertial effects is given by the elasticity number $El = Wi/Re$ [17].

As characteristic lengths decrease, the Re number decreases and the Wi number increases, and therefore El increases. This means that for very small characteristic lengths, as is the case in microfluidics, inertial effects become less relevant and viscoelastic effects are amplified.

This phenomenon is at the basis of scientific interest for microfluidics in the field of rheometry [37, 39]. Viscoelastic microfluidics has also revealed high efficiency in particle separation techniques [52], providing one more example of its potential uses [11, 29].

As mentioned before, one important real life application of microfluidics is in the field of biomedicine. It is worth highlighting that many biological samples have viscoelastic characteristics (e.g. blood), pointing to promising potential of the use of viscoelastic microfluidics in their handling [52].

One important fluid flow characteristic, both in micro and macro-scale is the entrance length (L_e). Once a fluid enters a channel, its velocity profile changes due to viscous effects imposed by the channel walls. This does not happen abruptly: the shape of the velocity profile changes gradually as the flow develops inside the channel, in a region of the flow referred to as the developing region. Nonetheless, at a certain distance from the inlet, the flow eventually becomes fully developed, which means that the velocity profile stops varying along the direction of the flow for the remainder of the channel (Figure 1.1). This distance is called the entrance length or development length [33, chap. 8]. Since the transition into the fully developed velocity profile is smooth, it can be difficult to precisely measure the entrance length after which there are no variations in the velocity profile. For practical purposes, a more common definition for the entrance length is the distance from the inlet of the channel at which the the velocity at the centre-line of the channel reaches 99% of its fully-developed value.

As the velocity profile is evolving along the developing region, it may affect other flow calculations. Examples of this can be found at the micro level. Alfieri et al. [3] found that for a problem involving the use of microchannels for cooling of chip stacks, temperature predictions could be affected up to 40% by ignoring the developing region. In the field of biomedicine, the

importance of developing regions is also discussed by Lighthill [28], which suggests that for some physiological flows, the entrance length may represent a majority of the entire channel length.

One of the recent application of microfluidics is the study of the complex flow dynamics of blood around microbot prototypes able to swim along the human circulatory system. For a proper fluid flow characterisation it is essential to have a good understanding of the entrance length of blood analogue complex fluid inside the microchannel mimicking some geometries of the human circulatory system. This thesis is developed within the framework of the FCT project HEMOSwimmers - Hemodynamic optimization around 3D swimming microbots (PTDC/EME-EME/30764/2017). Laboratory work was done at the Centro de Estudos de Fenómenos de Transporte (CEFT) facilities.

The thesis is divided into four chapters:

- Chapter 1: "Introduction", which mentions the objective of the thesis, explains the importance and pertinence of the problem analysed, discusses relevant work present in literature and describes the approach taken to achieve the set goals;
- Chapter 2: "Materials and Methods", which includes the description of the preparation and rheological characterisation of the working fluids, a description of the fabrication process and characteristics of the used microchannels, and the μ -PIV methodology for analysis of the entrance length;
- Chapter 3: "Results and Discussion" presents the results obtained from the μ -PIV analysis of the entrance length;
- Chapter 4: "Conclusions and Future Work" finally summarises the work conducted, and presents the conclusions that can be drawn from it, as well as discusses how it may impact future work done regarding this issue, and what this should be.

1.2 State of the art

The determination of the entrance length have been investigated experimentally since the first works of Atkinson et al. [5] and Chen [10]. Solutions to calculate the entrance length of a flow aim for a relation between a dimensionless entrance length (typically normalised with a characteristic length of the channel cross section), and dimensionless values relevant to the flow. This section explores and summarises some of the work developed in the determination of entrance lengths for both macro and microscale. It is organised by fluid characteristics, focusing first on Newtonian fluids, then non-Newtonian and specifically viscoelastic fluids.

For Newtonian fluids, the determination of entrance length in fluid flows has been studied at length, with many studies focusing on macroscale pipe flows, due to their practical relevance. It is possible to obtain a relation between the dimensionless entrance length as a function simply of the Reynolds number, Re . At the macroscale, for the laminar flow of a Newtonian fluid, the dimensionless entrance length was approximated to a linear function of Re . However, Durst et al.

[14] found that this may not be the case for low Re numbers ($1 \leq Re \leq 100$), where the dimensionless entrance length stabilises and remains close to constant. They propose a new equation which reflects this tendency and holds validity for the entire Re range within laminar flow.

Work is not as extensive in the field of microfluidics, but there is also a good enough understanding of the entrance length for laminar flows of Newtonian fluids in rectangular channels. Experimental studies have been performed by Ahmad and Hassan [1] for a square aspect ratio, and a Re range varying between 0.5 and 200. Lee et al. [25] also studied the effects of inlet plenums for aspect ratios of 2.75 and 0.4 and found that they significantly reduced the entrance length when compared to macroscale equations present in the literature. This study had Re numbers varying between 1 and 100.

Some numerical studies have also been conducted. Ferreira et al. [16] proposes a non-linear numerical solution varying with both the Reynolds number (valid for $0 \leq Re \leq 2000$ and the aspect-ratio of the cross section ($0.1 \leq AR \leq 1$)).

For non-Newtonian fluids, even at the macroscale the problem changes. Flow characteristics normally depend not only on the Reynolds number but also on the rheological characteristics. Poole and Ridley [41] expand the work of Durst et al. [14] and propose an equation for the entrance length in pipe-flow of Power-Law fluids, depending on an adjusted Reynolds number and on the Power-Law index n . The adjusted Reynolds number used is the one described by Metzner and Reede [31] for pressure drop calculations and varies between 0 and 1000. The work of Poole and Ridley was later expanded by Fernandes et al. [15] for planar channel flows and for fluids following the Sisko model, with an equation that also depends on the dimensionless infinite viscosity $\overline{\mu_\infty}$. This study uses a generalised Reynolds number varying between 0 and 100, defined similarly to the one used by Poole and Ridley for its equations.

In viscoelastic flow, the viscoelastic effects must be considered and other dimensionless numbers to describe these behaviours gain importance along with Re , particularly the Weissenberg number, Wi . Na and Yoo [34] showed that for constant Re , the entrance length increased with Wi , using a finite volume technique. They studied the velocity development for Re varying between 0 and 50, and Wi varying between 0 and 0.72. Alves et al. [4] noted a few benchmark solutions for viscoelastic flow at contraction geometries, namely analysing upstream vorticity and entrance pressure drops. Yapici et al. [51] numerically studied Oldroyd-B and PTT-linear fluid models and plots the entrance length using different criteria. This is done with a fixed very low Re number ($Re = 0.001$) and varying Wi ($0.1 \leq Wi \leq 1$) and with a fixed Wi (0.8 for the PTT-linear model and 0.5 for the Oldroyd-B) and varying Re between 0.001 and 10. With constant Re , the entrance length clearly increased with Wi . For constant Wi , however, the effects of Re were less obvious, and different tendencies were found for different regions within the studied range. The authors attribute this to the complex interactions taking place between the terms of inertia and viscoelasticity. Bertoco et al. [6] also numerically studies the development length of visco-elastic fluid flow, using a gPTT model. They consider creeping flow, $Re = 0.001$ at a Wi range of $0 \leq Wi \leq 1$ and plot solutions also analysing the effect of El . The analysed flow is between parallel plates.

Focusing more specifically on viscoelastic flow in microchannels, Li and Haward [27] studied

the development of viscoelastic flow through a contraction in microchannels. This paper investigated streamlines both upstream and downstream of the contraction that constituted the entrance of the microchannel and found a strong correlation between the flow upstream and downstream. They define Re using wall viscosity. The entrance length is studied for $0.3 \leq Re \leq 11.5$ and $10.8 \leq Wi \leq 256.2$. It should be highlighted that the values of Wi are much larger than that of previously mentioned studies [34, 51]. The results of Li and Haward [27] showed that the entrance length in viscoelastic flow is dependent on the Weissenberg number and much larger than for Newtonian fluids. Along the range of Wi they reported vastly different velocity profiles in the developing region of the flow, with a central dip and peaks in velocity near the channel walls. For these profiles, the velocity profile may change even after the centre-line velocity stabilises in the center dip, and therefore this is not a sufficient condition for classifying the flow as fully developed. The authors were unable to observe a steady velocity profile at the capture area visualised under μ -PIV, and therefore it was not possible to calculate the entrance length. This is likely due to analysis of the flow with high Weissenberg numbers, corresponding to high relaxation times of the fluid, and to a small capture area analysed through the PIV. Nonetheless, the developed work allowed them to find an equation for the entrance length of Newtonian fluid-flows in microchannels (studied in $1.9 \leq Re \leq 37.5$), similar to that of Ahmad and Hassan [1].

A summary of the equations found in the literature and mentioned in this section are presented in Table 1.1.

Table 1.1: Summary of equations in the literature for the entrance length of laminar flow in planar channels.

Authors	Approach	Type of fluid	Scale	Studied Re and Wi ranges	Proposed Equation
Durst et al. [14]	Experimental, Numerical and Analytical	Newtonian fluids	Macroscale	Entire range of laminar viscous flow	$\frac{L_e}{H} = [(0.631)^{1.6} + (0.0442Re)^{1.6}]^{\frac{1}{1.6}}$
Ahmad and Hassan [1]	Experimental	Newtonian Fluids	Microscale	$0.5 \leq Re \leq 200$	$\frac{L_e}{D_h} = \frac{0.63}{0.035Re+1} + 0.0752Re$
Li and Haward [27]*	Experimental	Viscoelastic and Newtonian fluids	Microscale	$0.3 \leq Re \leq 11.5$ $10.8 \leq Wi \leq 256.2$	$\frac{L_e}{D_h} = 0.24 + 0.03Re$
Ferreira et al. [16]	Numerical	Newtonian Fluids	Microscale	$0 \leq Re \leq 2000$	$\frac{L_e}{W} = [C_1^{\frac{C_3}{3}} + (C_2Re)^{\frac{C_3}{3}}]^{\frac{1}{C_3}}$ $C_1 = 0.7AR^{0.25}$ $C_2 = \frac{6.8AR^{3.75}}{1+90AR^{2.8}}$ $C_3 = 1 + AR$
Poole and Ridley [41]	Numerical	Power-Law Fluids	Macroscale Pipes	$0 \leq Re \leq 1000$	$\frac{L_e}{D} = [(0.246n^2 - 0.675n + 1.03)^{1.6} + (0.0567Re_{MR})^{1.6}]^{\frac{1}{1.6}}$
Fernandes et al. [15]	Numerical	Sisko model fluids	Macroscale planar channels	$0 \leq Re \leq 100$	$\frac{L_e}{H} = [h(n)^{1.6} + 0.0444Re_{gen}^{l(n)1/1.6} - m(Re_{gen}, n, \overline{\mu_{\infty}})]^{\frac{1}{1.6}}$ $h(n) = 0.0611n^2 - 0.2352n + 0.808$ $l(n) = -0.259n^2 + 0.1184n + 1.5468$ $m(Re_{gen}, n, \overline{\mu_{\infty}}) = \frac{Re_{gen}}{n(-367871\overline{\mu_{\infty}}^{-3} + 181733\overline{\mu_{\infty}}^{-2} - 23986\overline{\mu_{\infty}} + 1200.2)}$

*The equation presented applies only to Newtonian fluids (studied in $1.9 \leq Re \leq 37.5$).

1.3 Motivation and objectives

As explained in the first section of this chapter, being able to predict the entrance length of viscoelastic fluids flow in microchannels would prove useful in a variety of applications, and give a better understanding of this type of flow. One such application is biological flows, namely blood flow.

Whilst a lot of progress has been made in the determination of the entrance length of the flow of different fluids, a gap in the literature remained, with no equation to calculate it for viscoelastic fluid flows in microchannels.

The main goal of this master thesis was to fill this gap and propose an expression, based on experimental data, for the determination of the entrance length scale for viscoelastic fluid flows in microchannels.

To achieve this, the aim was to expand the work of Li and Haward [27], by working with less elastic fluids and focusing on a smaller Wi range. Whilst this limits the applicability of the results, it should be noted that many biological fluids, and particularly blood, are less elastic than the solution used by Li and Haward [27] and the results obtained in this study should hold interest in this field. In addition, experimentally working with less elastic fluids could allow for comparison and validation of already existing numerical studies ([34, 51, 6]) which also used a smaller Wi range. Besides, the work of Li and Haward [27] did not provided a equation on the dependence of the entry length with Re and Wi .

The decision to work with less elastic fluids was expected to lead to smaller entrance lengths. This should also help overcome the limiting problem experienced by Li and Haward [27], of not being able to observe the fully developed profile within the μ -PIV capture area. However, just lowering the elasticity was unlikely to ensure that this problem does not undermine the work of the thesis. With this in mind, a methodology that makes use of several measurement along the x-axis is developed.

The following objectives were set:

- Preparation and experimental rheological characterisation of viscoelastic fluids;
- Fabrication of microchannels to be used in flow analysis;
- Characterisation of flow dynamics by means of μ -PIV techniques to determine the flow velocity profile along the length of the channels, for different viscoelastic fluids;
- Data analysis to determine the entrance length for various flow rates and viscoelastic fluids.

Chapter 2

Materials and methods

In this chapter, the working fluids, and the protocols used in microchannel fabrication and in the development of the experimental tests are described.

Firstly, a description of the Newtonian and non-Newtonian working fluids and their preparation is shown. This is followed by the rheological characterisation under simple shear and elongational flows. The viscosity curves for all fluids were obtained as well as the relaxation time of the non-Newtonian fluids. The PDMS microchannels used were also fabricated in the laboratory. The microfabrication process used is also presented here. Finally, the flow analysis using the μ -PIV technique is explained.

2.1 Fluid preparation

Five viscoelastic solutions, as well as one Newtonian solution were prepared.

The Newtonian sample consisted on an aqueous solution of 52wt.% of dimethyl sulfoxide (DMSO) (CARLO ERBA Reagents). To provide the viscoelastic character, xanthan gum (XG) was added in varying quantities to the remaining solutions. Biocide sodium azide (SA) was also added to the solutions containing XG, in order to prevent bacterial growth. A summary of the composition of each of the prepared fluids is given in Table 2.1. All concentrations were measured by mass, and the solvent used was distilled water.

The solution containing 100 ppm of XG is meant to emulate rheological properties of blood, as described by Campo-Deaño et al. [9]. The solutions containing other quantities of XG were studied in order to obtain a wider range of Wi , as has been done in other articles present in the literature [48, 36].

Table 2.1: Composition of the different working fluids.

Solution Acronym	XG [ppm]	SA [ppm]	DMSO [%]
XG0	-	-	52.0
XG50	50	50	
XG100	100		
XG250	250		
XG500	500		
XG1000	1000		

Initially, the desired quantities of XG and SA for each solution were measured and mixed with distilled water. The mixing process was done with a magnetic mixer, shown in Figure 2.1. The solutions were left mixing for seven days at a low speed, in order to not interfere with the structural integrity of the polymer molecules. At the end of the mixing process, the DMSO was added.

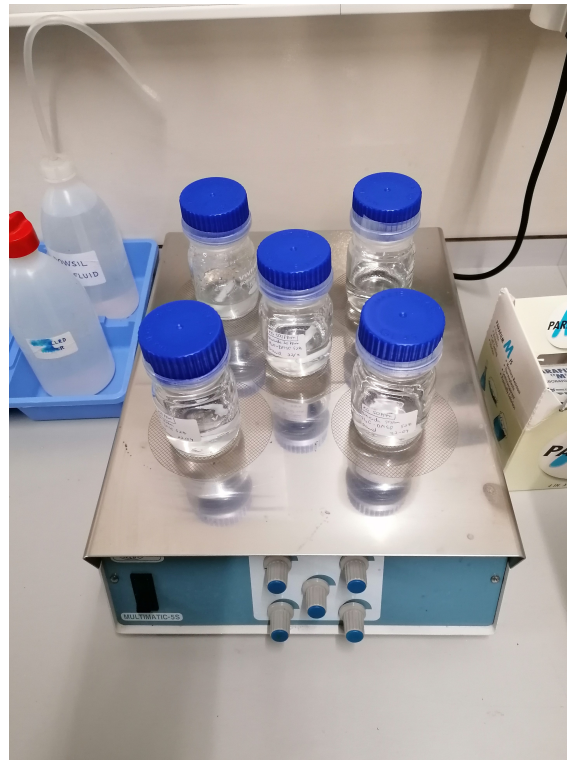


Figure 2.1: Mixing of XG and SA in distilled water using magnetic mixer.

2.2 Rheological characterisation

In order to conduct the dimensionless analysis of the entrance length, it is necessary to know the fluids rheological properties. In particular, the viscosity and relaxation time, as these are parameters used to calculate the Re and Wi numbers.

2.2.1 Shear flow

In non-Newtonian fluids, the shear viscosity changes with the shear rate applied to the fluid. This is why in order to properly characterise this property, a viscosity curve should be used. The viscosity curve was plotted using a rotational rheometer, which measured the shear viscosity for different shear rates.

The steady shear experiments were carried out using a stress-controlled shear rheometer (Anton Paar Physica™ MCR 301; minimum torque 10^{-4} mN.m) equipped with parallel-plate measuring system with diameter $\phi = 49.968$ mm (PP50) and a gap $h = 0.100$ mm, which can be seen in Figure 2.2



Figure 2.2: Rotational Rheometer used for shear flow analysis.

In order to ensure the steady state for each point of shear rate in the viscosity curve, a constant shear rate test was previously conducted.

As the polymer concentration increases, the elasticity of the solution increases too, and more time to achieve the stationary state is needed. Therefore, the constant shear tests were only conducted for XG500 and XG1000. The time needed to safely assume steady flow for XG500 was used also in measures for solutions with a smaller XG concentration.

For both XG500 and XG1000, a constant shear rate of 25 s^{-1} was applied, and it was studied how the measured viscosity changed within the first 30 seconds of the plate's rotation. The measurements were conducted at a controlled temperature of 20°C . Figure 2.3 shows the measured viscosity in intervals of 0.1 seconds for the first 5 seconds.

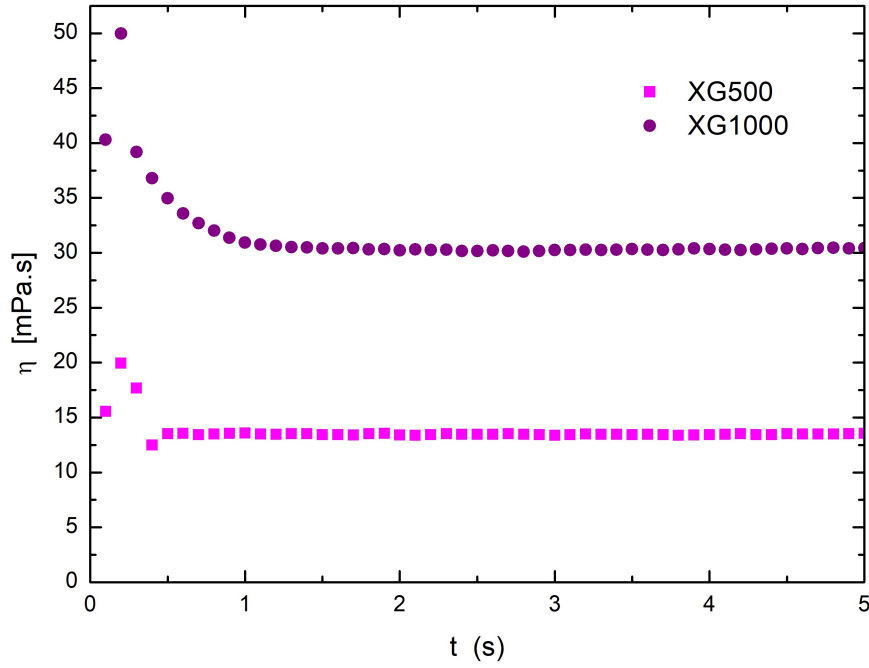


Figure 2.3: Constant shear analysis of XG500 and XG1000 at $\dot{\gamma} = 25 \text{ s}^{-1}$ and $T = 20 \text{ }^{\circ}\text{C}$.

The steady shear viscosity is achieved at $\sim 0.5 \text{ s}$ for the XG500 solution, and at $\sim 1.2 \text{ s}$ for the XG1000 solution. At times lower than those values, the flow between the rheometer plates can not be deemed steady, and results should not be considered. To ensure that this was not the case, viscosity measurements for the flow curve of XG500 and solutions with lower concentrations were taken 1 second after imposing the shear rate. For XG1000 this value was 20 seconds.

Once the required time to achieve the steady state viscosity is decided, the viscosity curves were obtained varying the shear rate from 0.1 to 10000 s^{-1} . This procedure was repeated at least 4 times for each solution to ensure reproducibility. Again, all measurements were conducted at a controlled temperature of $20 \text{ }^{\circ}\text{C}$.

Figure 2.4 shows, for each strain rate, the average viscosity obtained for the studied solutions. Error bars show confidence intervals of 95% for the measurements, obtained through a Student-t distribution.

As expected, the Newtonian solution's viscosity does not change with the shear rate, staying constant throughout the considered interval. In the viscoelastic solutions, the shear-thinning effect is clear, with the viscosity of the solutions decreasing as the shear rate increases.

This behaviour is concurrent with previous studies which used blood analogue polymeric solutions [9, 48]. Dilute polymeric solutions, such as the ones used, are typically shear thinning, with a shear thickening behaviour being observed only in solutions which use polymers with large molecular masses and highly viscous solvents [43].

This characteristic of polymeric solutions is correlated with changes in polymer shape and interactions caused by the application of a shear rate. At rest, polymers are typically entangled

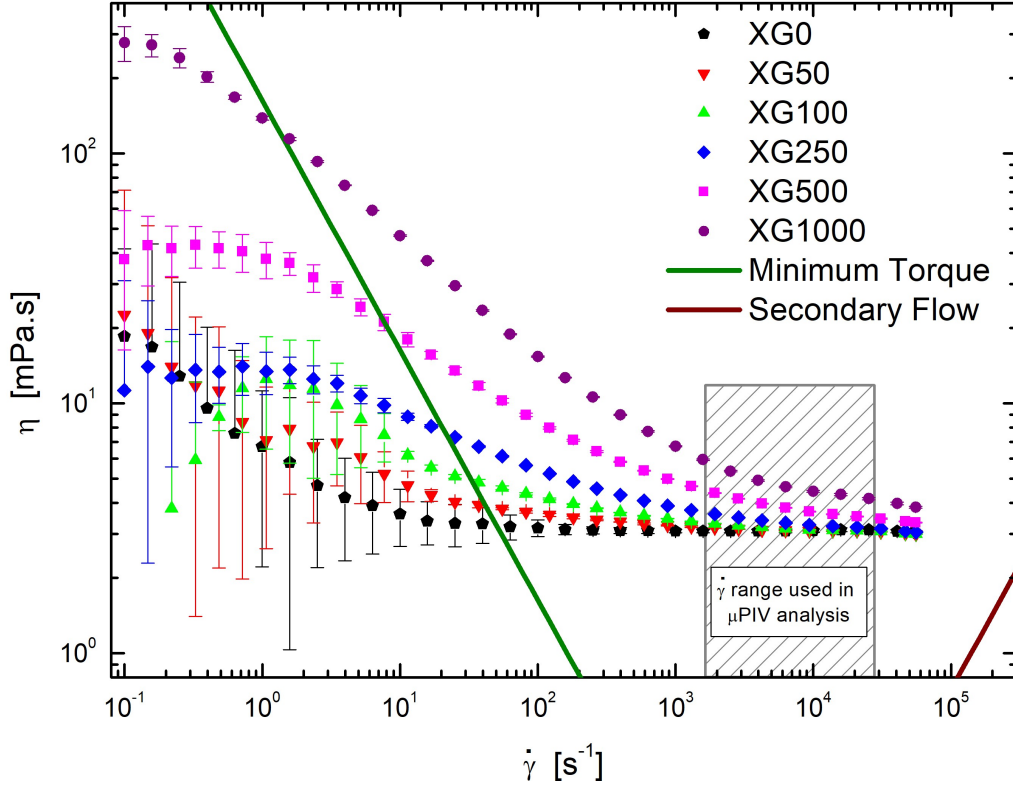


Figure 2.4: Flow curves of the studied solutions at $T = 20$ °C.

and randomly oriented. When a shear rate is applied, it leads to the polymers aligning and showing less resistance to the flow: the viscosity decreases. This phenomenon was studied and explained in more detail in the work of Xu et al. [50].

The minimum torque line in Figure 2.4 was obtained from the characteristic minimum torque of the rheometer, to which a correction factor of 40 was applied. The line plotted was then obtained through the equation:

$$\eta_{min}(\dot{\gamma}) = \frac{2(40 \times M_0)}{\pi R^3 \dot{\gamma}} \quad (2.1)$$

where M_0 is the minimum torque of the rheometer and R is the plate radius. It is clear that below the minimum torque line, the difference between repetitions is a lot higher, which is reflected in the bigger error bars. Points below the minimum torque line were therefore neglected.

The secondary flow line was obtained from the equation:

$$\eta_{secf}(\dot{\gamma}) = \frac{h^3 \rho \dot{\gamma}}{12 \hat{R} R} \times 10^3 \quad (2.2)$$

where h is the plate gap, ρ is the fluid density, obtained from the work of Rodrigues et al. [48],

$\hat{R}$ is equal to 0.5 and is a specific parameter of parallel-plate measuring systems, and finally R is the plate radius. None of the measured points fell under the secondary flow line, with the limiting factor of the measurements being the maximum rotation velocity of the rheometer.

The viscosity considered for the μ -PIV analysis was obtained from this data: For XG0, a Newtonian solution, a constant viscosity was assumed. Its value was calculated by initially applying Chauvenet's criterion [49] to pre-select data (from the 5 repetitions, considering the points above the minimum torque line) and averaging the results. For the viscoelastic solutions containing XG, the plotted points above the minimum torque line were used to fit a Carreau-Yasuda model [32]:

$$\eta(\dot{\gamma}) = \eta_{\infty} + (\eta_0 - \eta_{\infty})[1 + (\dot{\gamma}\lambda_{CY})^a]^{\frac{n-1}{a}} \quad (2.3)$$

where $\eta(\dot{\gamma})$ is the fitting function which gives the viscosity of the solution for a given shear rate $\dot{\gamma}$; η_{∞} is the viscosity as the shear rate tends to infinity, and a plateau is formed in the viscosity curve; η_0 is the viscosity as the shear rate tends to zero, and a plateau is formed in the viscosity curve; λ_{CY} is a time constant that determines at what shear rate there is a transition from the zero-shear viscosity η_0 into the power-law relation; a governs the shape of said transition, and finally n governs the slope of the power-law section of the viscosity curve. The parameters used for each solution were calculated using Microsoft Excel™ Solver Add-in to calculate the parameters by minimising the relative squared error between measurements and model. The obtained values ensured an average relative error of 0.58% and a maximum relative error of 2.11%, taking into account all the measured points above the minimum torque line.

Table 2.2 shows, for each solution, the obtained parameters for the Carreau-Yasuda models, as well as the maximum relative error between the averaged experimental data for each point and the model. For XG0 the maximum relative error is in comparison with the calculated constant viscosity value.

Table 2.2: Parameters used in the Carreau-Yasuda modelling of the solutions.

Solution	Obtained Carreau-Yasuda Parameters					Max. relative error
	η_0 [mPa.s]	a [-]	n [-]	λ_{CY} [s]	η_{∞} [mPa.s]	
XG0	-	-	-	-	3.08	1.20%
XG50	6.49	0.996	0.605	0.714	2.97	1.13%
XG100	6.48	1.38	0.566	0.0973	2.94	0.94%
XG250	10.4	1.50	0.558	0.116	2.92	0.95%
XG500	24.1	2.68	0.516	0.169	3.10	0.65%
XG1000	166	1.65	0.430	0.996	3.57	2.11%

The range of $\dot{\gamma}$ used in μ -PIV analysis marked in Figure 2.4 was calculated based on the average velocity of the flow through the channel. Calculations are explained more precisely in Subsection 2.4.2. It is worth noting that in this range, the shear-thinning effects are neglectable for XG50 and XG100, and very small for XG250: their viscosity is close to its η_{∞} value, and almost

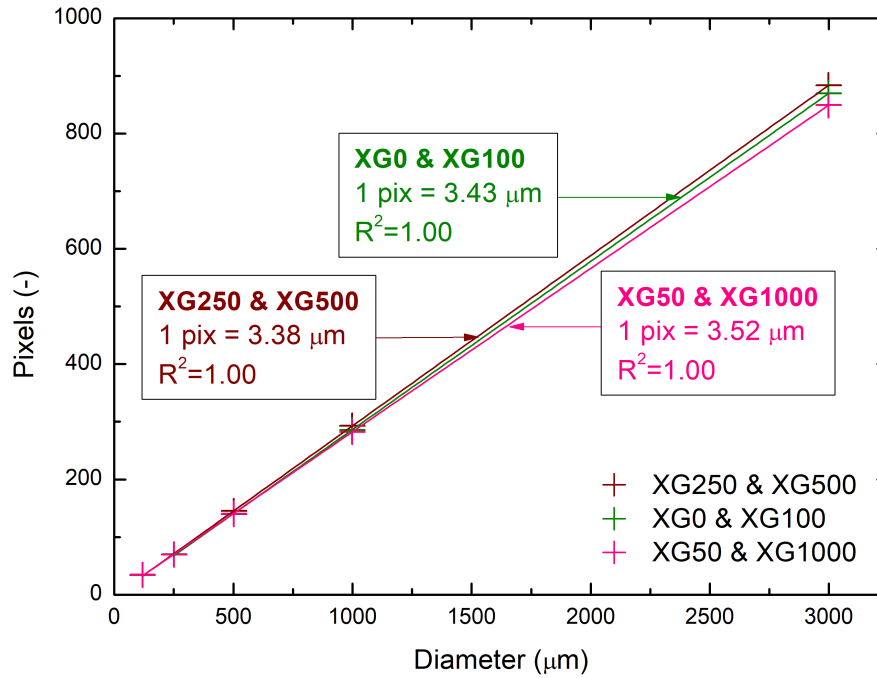


Figure 2.5: Calibration curves and micron/pixel ratios for the different solutions.

overlaps with the viscosity of the Newtonian solution. For XG500 and XG1000, the shear-thinning effect is still noticeable, albeit smaller than for lower values of $\dot{\gamma}$. The same can be said for the difference in the viscosity values of these solutions and the other solutions used.

2.2.2 Elongational flow

For the characterisation of the elongational flow and the relaxation times, a Capillary Breakup Extensional Rheometer (Thermo Scientific™ HAAKE™ CaBER™ 1) was used, together with a high-speed CMOS camera (Photron FASTCAM Mini UX100), equipped with a set of optical lenses (Optem Zoom 70 XL, Qioptiq, Fairport, NY, USA) to capture the fluid filament thinning silhouettes evolution over time. The camera operated at 20000 frames per second (fps), recording pictures of 1280×248 px².

The micron/pixel ratio was determined by taking pictures of standard filament diameters under the same recording conditions used for the experiment. This is the same calibration procedure used by Nunes et al. [36]. The exact camera positioning was not the same in all measurements, which results in a slightly different micron/pixel ratio between sets of measurements. Figure 2.5 shows the calibration curves obtained for each set of experiments, indicating the analysed solutions corresponding to each calibration curve.

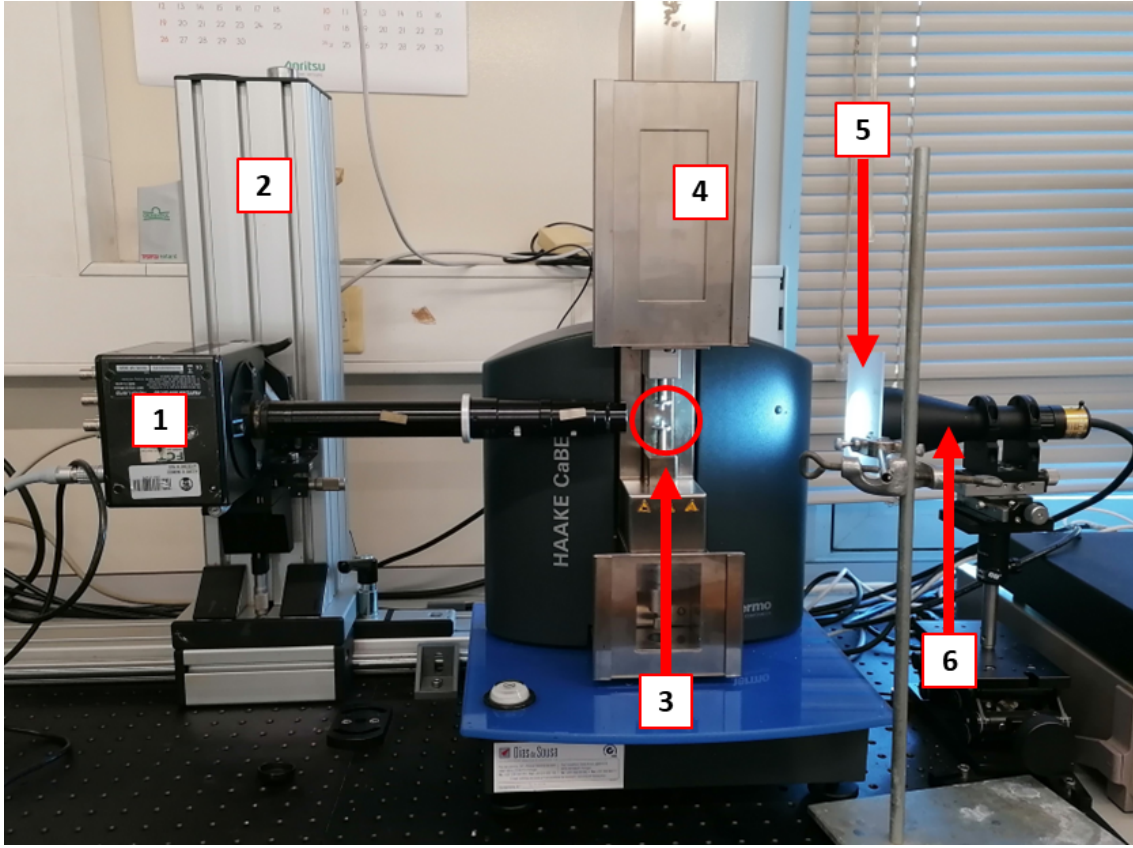


Figure 2.6: Extensional rheometer experimental set-up: 1- High-speed camera; 2- Sliding adjustable camera support; 3- Rheometer plates and solution sample; 4- Extensional rheometer; 5- Glass; 6- Light source.

To facilitate the recording process of the high contrast images, a 52 mm Telecentric Backlight Illuminator was used, connected to a metal halide light source (LeicaEL6000, Leica Microsystems, Wetzlar, Germany). Furthermore, to help regulate the lighting conditions, a frosted glass was put in front of the light-source when necessary. The entire experimental set-up is shown in Figure 2.6.

All samples were analysed using plates with a diameter of $D_0 = 4$ mm and an initial gap of $h_0 = 1.99$ mm. To minimise inertial effects, the slow retraction method (SRM) [8] was used. With the solution between them, the plates were separated at very low velocities ($V_{plate} = 0.10$ mm/s), and the camera recorded the evolution of the filament thinning until rupture. Since the used rheometer did not allow for temperature control, the experiment was conducted at room temperature of around 24 °C.

Figure 2.7 shows, for each solution, examples of some of the recorded images, for the indicated time instances. These include pictures immediately before and immediately after the filament breaking.

In Figure 2.7, it can be observed the filament thinning process recorded by means of the high-speed camera. As has been reported in the literature [48, 36], it is clear that there is an increase in

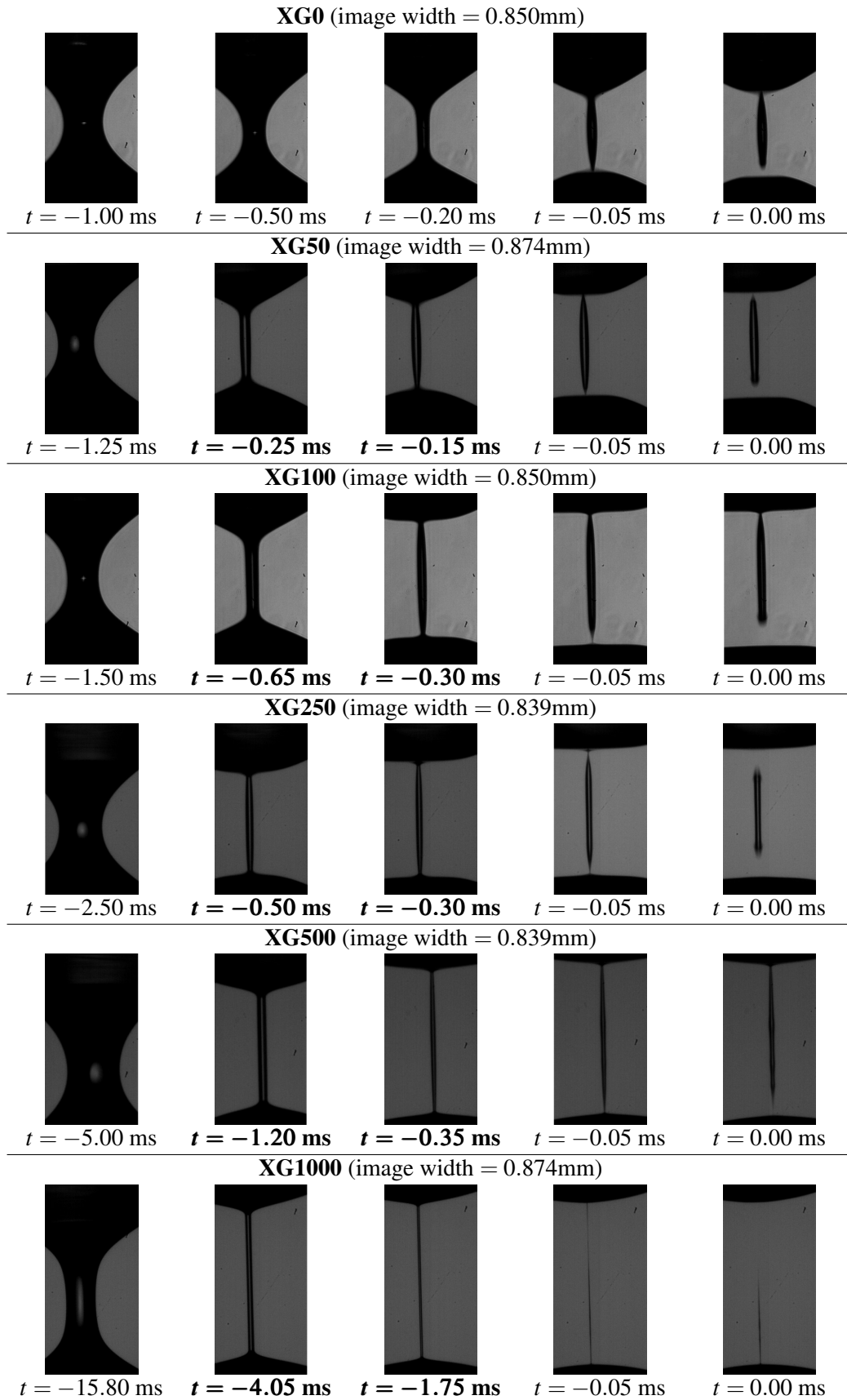


Figure 2.7: Filament thinning process for each solution at room temperature. Picture scale is indicated for each solution. Time scale is adjusted for rupture time of each solution.

elastic behaviour with the increase of XG concentration in the samples. The Newtonian solution (XG0) presents a clear *pinch-off* behaviour, as should be expected [8].

In the case of the non-Newtonian samples, the plate movement creates capillary pressure that acts in a squeezing action. The polymer solutions are initially unstretched and the capillary pressure is balanced only by inertial acceleration in the fluid column. This is the initial regime of the elongational flow, called potential flow regime, and a typical profile can be seen in the first picture shown for each solution in Figure 2.7. Once the rheometer plates are far enough away and high enough deformation rates are imposed, the polymer molecules stretch and unravel, and the resulting entropic stresses balance the capillary pressure's squeezing action: the flow enters the elasto-capillary balance regime. Here the fluid forms a thin thread that becomes thinner exponentially overtime, in a relation which involves the relaxation time of the solution [8]:

$$D_{min}(t) = Ce^{\frac{-t}{\lambda}} \quad (2.4)$$

where $D_{min}(t)$ is the minimum diameter observed at a time t and λ is the fluid longest relaxation time. The elasto-capillary balance regime is represented in the second and third pictures of each solution shown in Figure 2.7.

The collected images were analysed through a MatLabTM image-analysis script, as in previous works developed at CEFT [36, 42], which plotted the minimum diameter registered in each picture through time. For the viscoelastic solutions, an exponential regression was applied to the elasto-capillary balance regime section of these plots. Matching the parameters obtained from the regression with Equation 2.4 allowed for the determination of the relaxation times.

This process was repeated six times per solution, obtaining a relaxation time for each repetition. At least three repetitions were considered for statistical analysis. For each viscoelastic solution, the obtained relaxation times were averaged and 95% confidence intervals were calculated with a Student-t distribution.

Figure 2.8 shows the obtained plots (one per solution) of the evolution of minimum diameter (normalised for plate diameter D_0) throughout time. To better reflect the difference between break-up times, the time scale of each solution is adjusted by adding the difference between its break-up time and the time where the pinch-off occurs for the Newtonian solution. The adjustments in diameter normalisation and time scale force all flow plots to coincide in their initial potential flow phase. Figure 2.8a) shows the plots of every solution, and Figure 2.8b) is an enhancement to show with more clarity the plots pertaining to solutions with lower elasticity.

The exponential regressions, and the average relaxation times of the corresponding solutions are also marked, along with the calculated uncertainty. The obtained relaxation times are also shown in Table 2.3. As the graph is plotted in a log-lin scale, it is clear that an exponential regression fits the plotted curves in the chosen areas and that these points correspond to the elasto-capillary regime. It should be noted that the regression was applied between the points corresponding to the pictures with the time indicated in bold in Figure 2.7 (the second and third pictures of

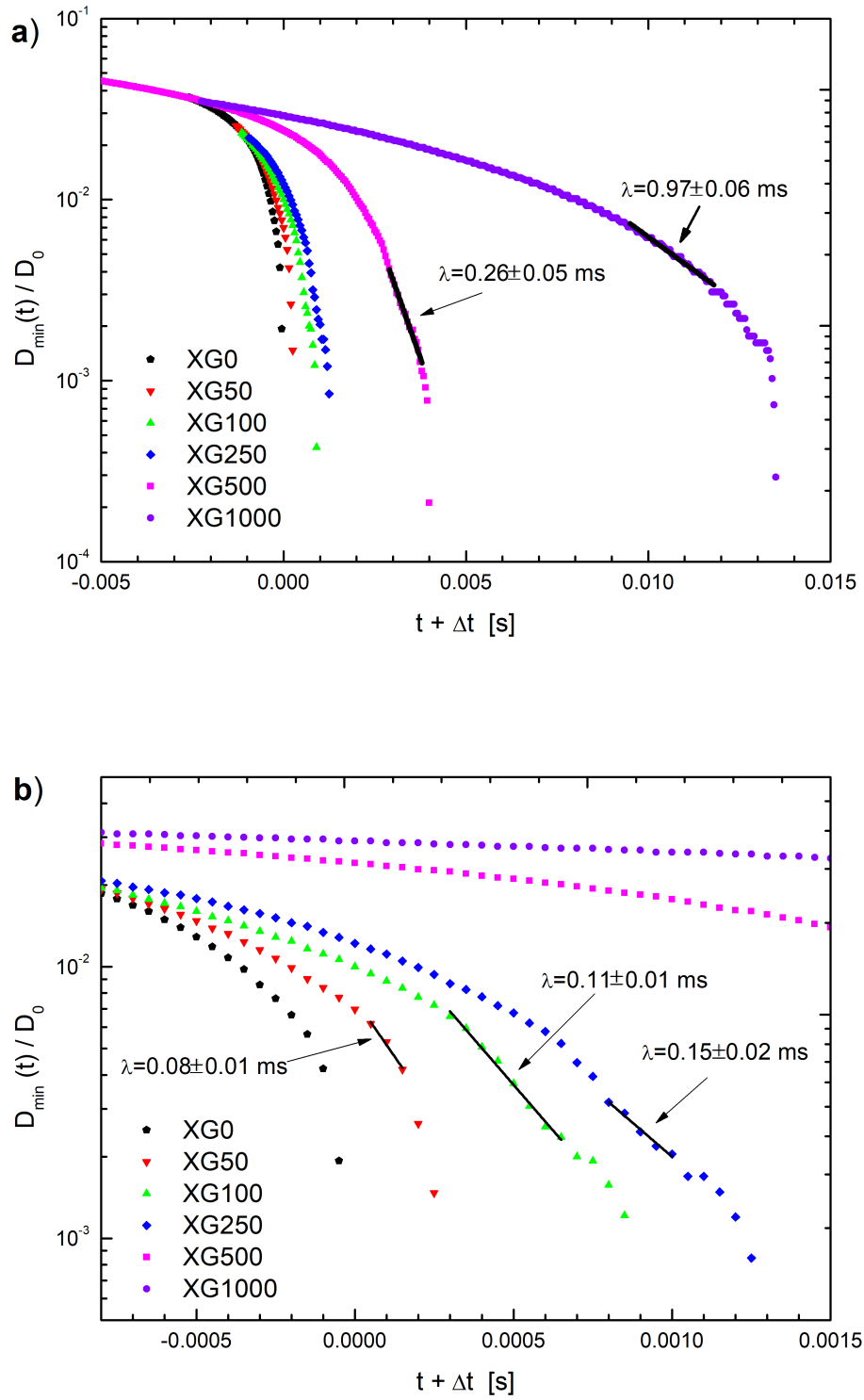


Figure 2.8: Normalised minimum diameter throughout the filament thinning process at room temperature. Time scale is adjusted to show difference between solutions' break-up times ($\Delta t = t_{\text{break-up}}_{\text{solution}} - t_{\text{pinch-off}}_{\text{XG0}}$).

Table 2.3: Obtained longest relaxation times of each solution.

Solution	λ [ms]
XG50	0.08
XG100	0.11
XG250	0.15
XG500	0.26
XG1000	0.97

the solutions containing XG). The pictures further show that, in the analysed areas of the plots, the flow is in its elasto-capillary balance regime. The calculated relaxation times seem coherent with the behaviour shown in the photos obtained by the high-speed camera seen in those pictures.

These values are, however, lower than those found in the literature [48], and the overall elastic behaviour of the solutions is also less evident than that reported by other authors [36]. The possible influence of temperature in the obtained results was studied, but the obtained variations were not enough to explain the discrepancies with the literature. One possible explanation for this difference is a different batch of solutes than that used by other authors. Nevertheless, the results are consistent between solutions and repetitions, and the calculated parameters reflect the behaviour observed experimentally. The calculated relaxation times values described this particular group of solutions, and were therefore the values used in further calculations.

It was noted that XG50 showed very little difference in behaviour to the Newtonian solution. Whilst some shear-thinning effect was seen in the viscosity, it was negligible in the $\dot{\gamma}$ range to be analysed in μ -PIV (see Figure 2.4). What is more, its relaxation time was also close to null, showing a very similar behaviour in the conducted elongational flow analysis (see Figures 2.7 and 2.8). It was therefore decided not to conduct μ -PIV analysis for the XG50 solution.

2.3 Microchannels

The microchannels used for the flow of the solutions were fabricated in the microfluidic laboratory. This section describes the fabrication process, and the characteristics of the microchannels.

The microchannels were fabricated based on master moulds of SU-8 coated silicon substrates and using two-part Sylgard® 184 PolyDiMethylSiloxane (PDMS) mixed to a mass ratio of 10:1. The moulds, shown in Figure 2.9 acted as a core to imprint the geometry of the microchannels in the PDMS mixture. Figure 2.10 shows a schematic of the master mould geometry (not to scale) which highlights the channel where fluid flow was analysed, and the intersection where an outlet was created.

In order to later facilitate the demoulding process of the PDMS, the mould underwent a silanisation treatment with TriMethylChloroSilane (TMCS) for 50 minutes. For safety reasons, due to the characteristics of the TMCS, this process, which can be seen in Figure 2.11, took place in a hood.

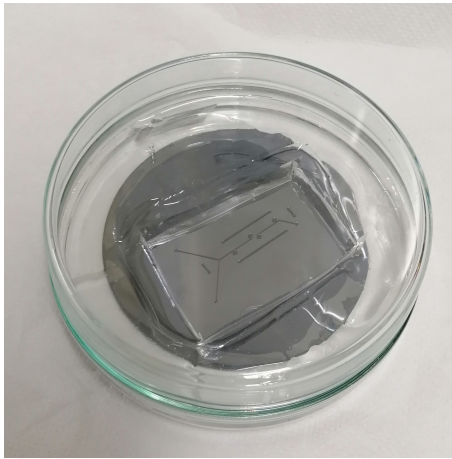


Figure 2.9: Master mould of SU-8 coated silicon substrates.

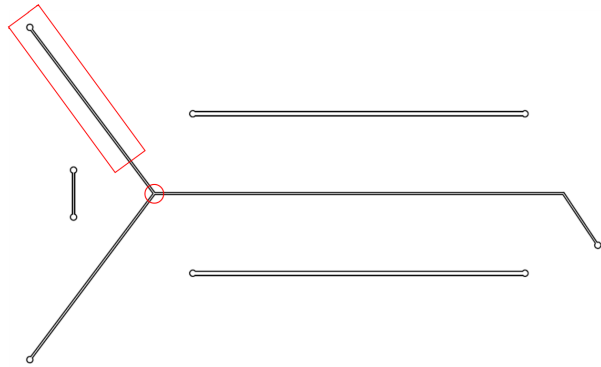


Figure 2.10: Schematic of master mould geometry.

To eliminate air bubbles near or inside the microchannels, that could influence the flow, the PDMS mixture degassed under vacuum in a desiccator, before being poured onto the moulds. Figure 2.12 shows this process.

After degassing took place and the silanisation process was concluded, the mixture was poured into the mould and then cured in an oven at 80 °C for 30 minutes.

After leaving the oven, the PDMS layers were cut and peeled off from the moulds, already with the imprint of the microchannels geometry. The peeled layers were then punched to create entrances and exits taps for setting the flow in each channel.

To punch the PDMS layers, a needle with an outer diameter of $\phi_{out} = 1.83\text{mm}$ was used, which ensured that needles with dimensions $\phi_{out} = 1.65\text{mm}$, $\phi_{in} = 1.36\text{mm}$ could be used in the set-up for flow analysis. The needles used can be seen in Figure 2.13. A 90° curved needle was used in the set-up for flow analysis to ensure that flow visualisation is not disturbed. The needle used to punch the PDMS has a bigger outer diameter than the one used in the experimental set-up due to

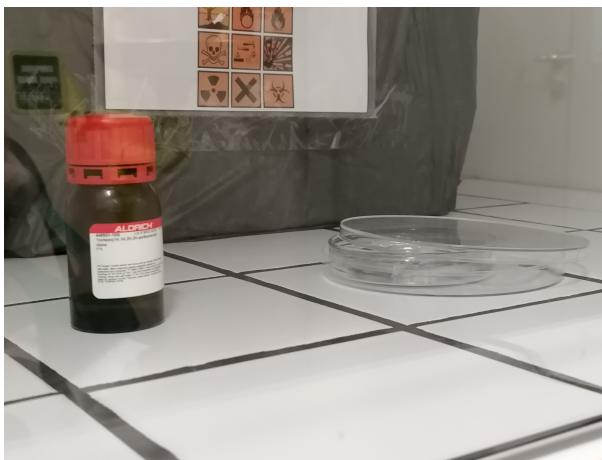


Figure 2.11: Silanisation process using TMCS.



Figure 2.12: PDMS mixture degassing.

the PDMS slightly contracting after being punched.

The needle-punching of the PDMS was done manually. The channel mould included a small circular circular area upstream of the inlet, and the needle was manually pushed through it, as shown in Figure 2.14.

At this stage, the layers were cleaned using compressed air to remove loose PDMS waste formed during the cutting and punching process.

Finally, the layers were plasma-bonded [46] to glass microscope slides to close off the channels in the face which had been in contact with the SU-8 mould. The PDMS-glass bond occurred using a low pressure plasma-bonding technique, using the device shown in Figure 2.15. Using this technique to close off the microchannels contributes to preventing tracer particles used during μ -PIV to accumulate excessively in the walls of the channel, which could create problems in analysing flow patterns.



Figure 2.13: Needles used in the set-up for flow analysis using μ -PIV (left) and for punching (right).

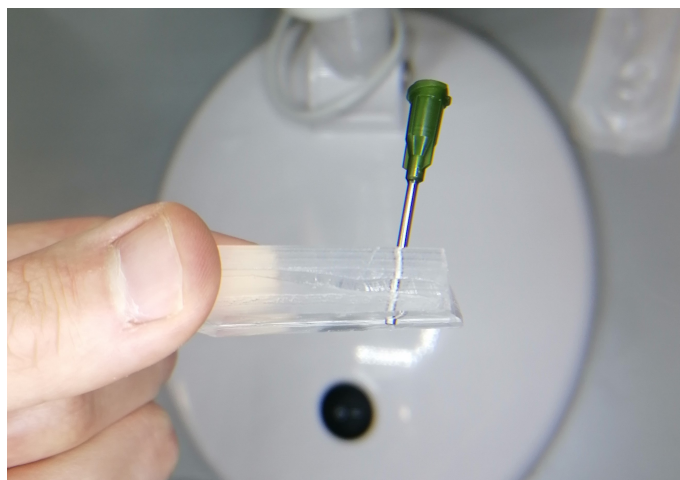


Figure 2.14: Punching of the PDMS layers to create inlets and outlets for the flow.



Figure 2.15: Device used for plasma-bonding the PDMS layers and the glass microscopic slides.

To better understand the fluid flow, it is important to properly characterise the geometry of the microchannels, given by the mould. With that in mind, one sample of the imprinted PDMS, cut but not punched, was analysed under a Scanning Electron Microscope (SEM). This allowed for a detailed observation of its top view and cross section.

The top view of the channels can be seen in Figure 2.16. Figure 2.16a) shows the entrance to the microchannels, including the inlet and the circular area upstream of the inlet. Figure 2.16b) shows a further downstream section of the channel, at a more enhanced scale.

The channel cross section can be seen in Figure 2.17. Figures 2.17 a) and b) show the channel cross section in its entirety, from different angles. Figures 2.17 c) and d) show the wall geometry on each side of the microchannel with more detail.

It is clear from Figure 2.17 that the channels present some deviations from the expected rectangular cross section. This can be attributed to defects of the SU8 silicon substracts mould.

The SEM pictures were analysed with the help of MatLab™ image-analysis tools, to measure

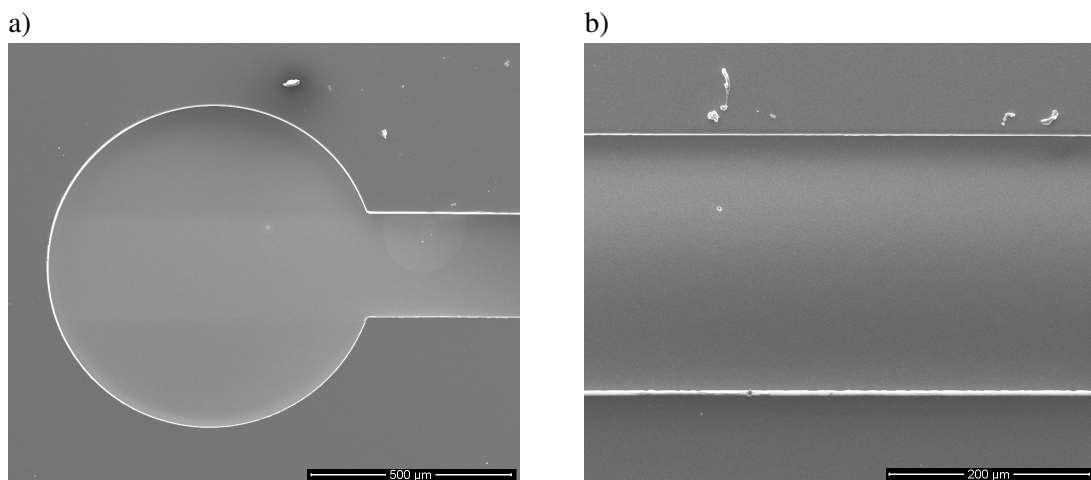


Figure 2.16: SEM images of PDMS imprinted by microchannel mould - top view.

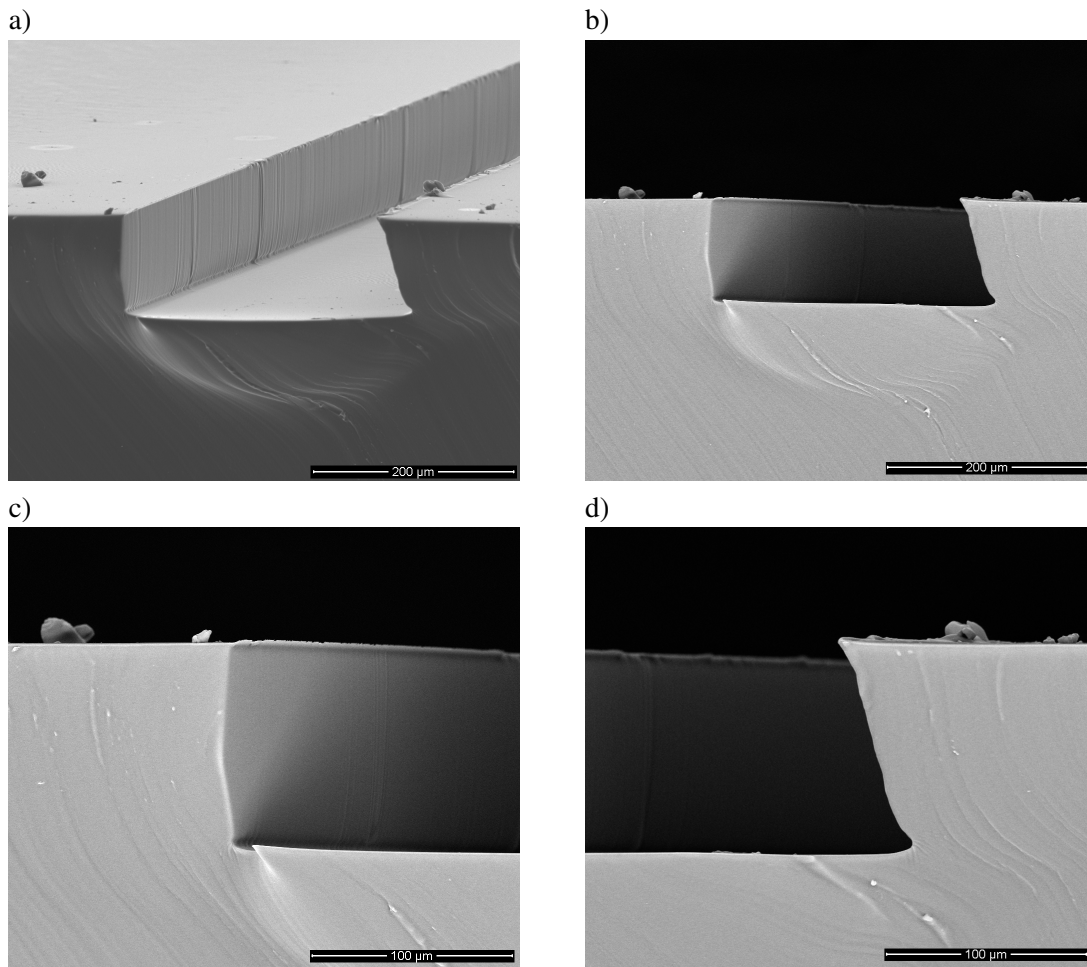


Figure 2.17: SEM images of PDMS imprinted by microchannel mould - cross section.

the dimensions of the microchannels. Smaller defects were neglected, such as the slight curvature of the wall depicted on Figure 2.17c) or the small dip in the corner between that wall and floor of the channel. However, the trapezoidal shape of the cross-section, made evident by the inclination of the wall depicted on Figure 2.17d) was considered and measured.

A schematic representation of the PDMS structure of the microchannels is shown in Figure 2.18, which shows the considered measured dimensions.

Having measured the dimensions of the channel, approximating the trapezoidal shape to a rectangular one with the same area ($H = 0.105$ mm, $W = 0.272$ mm) seemed reasonable (the error in the dimensions of the basis of the trapezoid is less than 7%). This is reinforced by the methodology used for the μ -PIV measurements. At the centre plane of the microchannels, where the measurements were conducted, 0.272 mm is the expected width of the channel, even considering the trapezoidal shape, with variations caused by the measurements' depth of field being very small. The width of the channel does not have particular relevance for flow calculations, and in

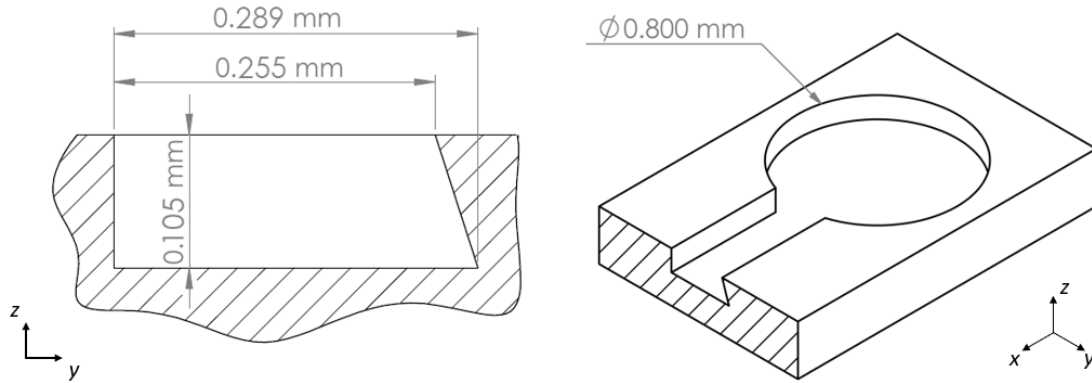


Figure 2.18: Measured dimensions of the PDMS microchannel.

most instances where it is used, using a mean of the length of both basis of the trapezoid would still be an appropriate solution (e.g. the calculation of the cross section area). This approximation was seldom used and should not correspond to any considerable errors in the obtained results.

It should be noted that the diameter of the needle through which the solutions will flow when entering the PDMS structure ($\phi_{in} = 1.36$ mm) is much larger than the dimensions of the microchannel cross-section, and larger than the diameter of the circular area upstream of the inlet. It can then assumed that the needle acts like a large enough reservoir for the fluid flow through the microchannel. Effects of the circular area upstream of the inlet in flow "pre-development" may depend on the exact positioning of the needle punching relative to the channel inlet, which varies from channel to channel since the punching was done manually. Still, differences in pre-development are expected to be negligible, due to the circular shape of diameter larger than the microchannels cross section dimensions.

Lastly, it is also worth noting that in all the SEM images shown in Figures 2.16 and 2.17, it is possible to observe some loose PDMS waste formed during the cutting of the layers and their removal from the mould. These bits of waste are small enough to not represent any significant perturbations in the flow analysis, but still noticeable from the top view, even at smaller magnifications, as in Figure 2.16a).

2.4 Flow characterisation

2.4.1 μ -PIV experimental set-up

Particle image velocimetry is a technique to quantify the velocity field based on images of tracer particles suspended in a fluid flow. Ideally, these particles are homogeneously distributed and their presence does not influence the fluid properties.

In PIV measurements, a laser beam goes through a semi-cylindrical lenses, creating a laser sheet that illuminates the particle field. The laser is activated twice, with the two pulses being separated by a given time interval Δt_{pulses} and two pictures (one per laser pulse) are obtained.

Image processing software can track the particle movement between the two pictures. To enable the software to better track the tracer particles, the recorded images are divided into interrogation areas [22]. As Δt_{pulses} is typically very small, the difference between the position of the particles, divided by the time between pulses, can be approximated to the time derivative of the position vector, which is the velocity vector:

$$\frac{\Delta \vec{x}}{\Delta t_{pulses}} \approx \frac{d\vec{x}}{dt} = \vec{u} \quad (2.5)$$

For stationary flow, repeating this process for other pairs of images yields a mean velocity field.

Nonetheless, it is broadly used in the literature, as it is able to identify the velocity direction, which can be used to better understand flow streamlines and turbulence [2, 19]. Besides that it can easily characterise the velocity at different points of the channel, and obtain instantaneous velocity profiles [24, 21], as is being done in this thesis, at a microscale.

Use of this technique for microscopic systems has been reported since the introductory works of Santiago et al. [45] and has become wide-spread for flow analysis in microchannels [7], again with many examples of its use in the literature [38, 26]. A key difference between PIV and μ -PIV is the illumination, which in PIV is done through a light sheet, but through a volume illumination in μ -PIV. Besides that, due to the small size of the tracer particles, these may interact with fluid particles and create Brownian motion [7].

For the purposes of this thesis, the studied solutions were prepared for the μ -PIV analysis by being mixed with monodisperse fluorescent tracer particles (Molecular Probes™ FluoSpheres®; F8825 Nile red carboxylate-modified microspheres) of $2.0 \mu\text{m}$ at a concentration of $\sim 0.2 \text{ wt\%}$. To prevent the tracer particles from adhering to the channel and accumulating excessively in its walls, sodium dodecyl sulfate (SDS) was also added, at a concentration of $\sim 0.1 \text{ wt\%}$. Addition of this particles has negligible impacts in the solutions' rheological properties [48].

The experimental setup for μ -PIV flow characterisation can be seen in Figure 2.19.

The optical setup used to capture the images for μ -PIV consisted of an inverted microscope (Leica Microsystems GmbH; DMI5000 M) equipped with a $\times 20$ magnification objective lens (Leica Microsystems GmbH; HCX PL Fluotar with a numerical aperture of $\text{NA} = 0.4$) and a charge-coupled device (CCD) camera (Dantec Dynamics; FlowSense 4M MkII with a pixel depth of 12-bit per pixel). At full frame, the camera recorded pictures with a resolution of $2048 \times 2048 \text{ px}$. For the applied magnification ($\times 20$, correspondent to a scale factor of 0.05), $1 \text{ px} = 0.37 \mu\text{m}$. Each capture area has dimensions of $2048 \times 0.37 \mu\text{m} \approx 0.76 \text{ mm}$. The utilised laser was a double-pulsed, $\lambda_{laser} = 532 \text{ nm}$ wavelength, Nd:YAG laser system. The camera operated in double frame mode, synchronised with the laser pulses such that the pictures were taken at the moment of illumination, such that correlation between positions could be studied.

100 picture pairs were considered for each measurement. The correlation between them was calculated using a cross-correlation PIV algorithm (Dantec Dynamics; DynamicStudio v2.30). In-

terrogation areas were defined with 128 px in the x -direction (direction of the flow) and 16 px in the y -direction (orthogonal to the direction flow in the horizontal plane). The interrogation areas had a 50% overlap with neighbouring interrogation areas. This accounted for detailed measurements of the velocity profiles (y resolution of $2.96 \mu\text{m}$) at different x positions (x resolution of $23.68 \mu\text{m}$). The imposed Δt_{pulses} depended of the flow rate and was calculated as the time interval expected for a particle moving at twice the average velocity of the flow to move through one sixth of the interrogation areas. This lead to Δt_{pulses} in the range of $7.73 \mu\text{s} \leq \Delta t_{\text{pulses}} \leq 129.93 \mu\text{s}$.

PIV measurements were conducted in the mid focal plane of the z direction of the microchannels. To find the mid focal plane, the focal planes of the top and bottom of the microchannels were discovered, by finding the first particle to be fully in focus when moving the focal plane in the z direction towards the center of the channel. The mid focal plane was considered to be in between these two planes, and therefore a mean was applied to find it. It should be noted that for the experimental set-up used, the depth of field of the measurements corresponds to $\delta z \approx 30.7 \mu\text{m}$, calculated from the equation presented in [30]. This corresponds to $\sim 29\%$ of the total channel depth.

The fluid flow through the microchannels was induced and controlled by a neMESYS low pressure syringe pump of 14:1 gear ratio (CETONI GmbH). A Hamilton® Gastight 2.5 mL syringe was used. When choosing the syringe, it was ensured that it could operate in the applied flow rate range, without pulsation. Solutions flowed from the syringe through a connecting chemically-resistant Tygon® tube into the inlet needle ($\phi_{\text{in}} = 1.36 \text{ mm}$, see Section 2.3). The solutions then

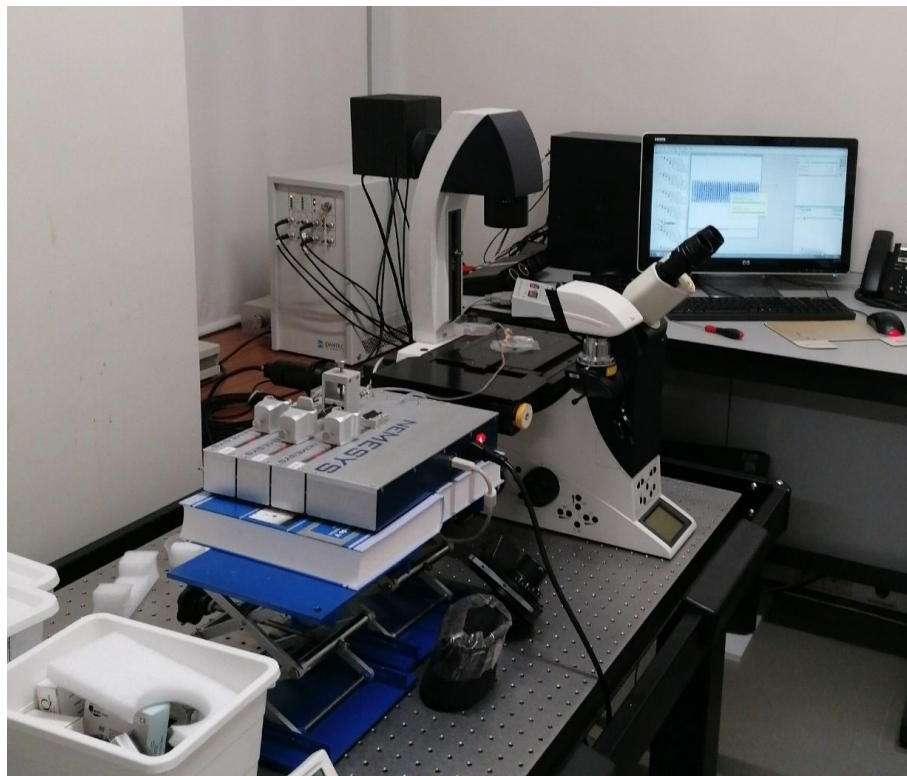


Figure 2.19: Experimental set-up for μ -PIV measurements.

made its way through the channel, exiting through the outlet needle, which was identical to the inlet one, and through another connecting tube which was left open to the atmosphere. After going through the entire course, the solution was collected into a Petri dish, from which it was disposed of. The course of the fluid is highlighted in Figure 2.20.

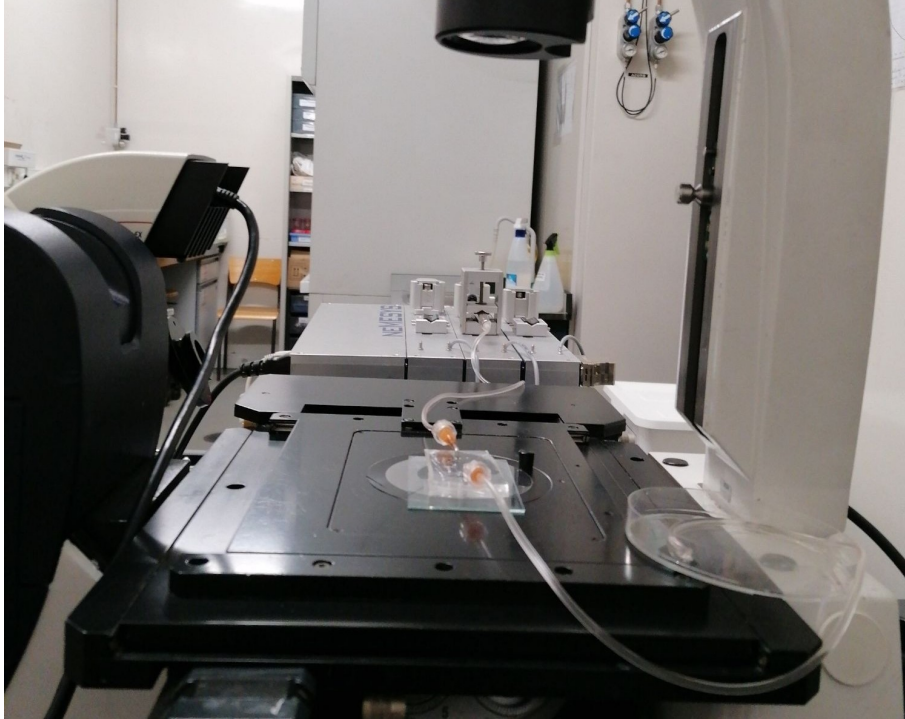


Figure 2.20: Close up on the course of the fluid flow.

Finally, it is worth noting that for each measurement, after activation of the syringe pump and initiation of the fluid flow, some time was given for the flow to develop into stationary state before proceeding with the μ -PIV measurements.

2.4.2 Dimensionless numbers used

Different measurements were defined by their Reynolds and Weissenberg numbers. The Elasticity number was also considered. A brief theoretical explanation regarding these numbers is given in Section 1.1.

There, it is mentioned that for non-Newtonian fluids, the Reynolds number ($Re = \rho \bar{U} L / \mu$) needs to be generalised, as μ is no longer a constant value. Instead, the viscosity is given by $\eta(\dot{\gamma})$, which means it varies depending on the shear rate, and therefore depending on the position of where the flow is analysed.

One possible way to define this is by considering $\eta_{wall} = \eta(\dot{\gamma}_{wall})$, as at the wall, the shear rate is at its highest. This was the approximation used by Li and Haward [27], and will be the one used for the purposes of this thesis. It was decided to follow this generalisation of Re so that

comparisons between dimensionless ranges used in this and the work of Li and Haward [27] were appropriate. They approximate:

$$\dot{\gamma}_{wall} \approx \frac{6(\bar{U} \times 10^{-3})}{H} = \frac{6(\dot{Q} \times \frac{10^3}{3600})}{AH} \quad (2.6)$$

where $\dot{\gamma}_{wall}$ is the shear rate at the wall perpendicular to the channels smallest dimension (in this case, H , the channel's height), $\bar{U}$ is the average imposed velocity of the flow, $\dot{Q}$ is the volume flow rate and A is the area of the channel's cross-section. As A and H are well defined, this equation allows for the calculation of $\dot{\gamma}_{wall}(\dot{Q})$, and therefore it was the one used in the determination of the $\dot{\gamma}$ range used in μ -PIV analysis, shown in Figure 2.4.

Through the use of the Carreau-Yasuda model defined for each fluid (Equation 2.3 and Table 2.2), a relation of $\eta(\dot{\gamma})$ already exists for each fluid. This means that obtaining $\dot{\gamma}_{wall}$ allows for the calculation of η_{wall} , and in turn the calculation of the Re number, which adjusted for the nomenclature used in this paper can be written as:

$$Re = \frac{\rho \bar{U} W}{\eta_{wall}} = \frac{\rho(\frac{\dot{Q}}{3600}) W}{A \eta_{wall}} \quad (2.7)$$

where ρ is the fluid density, obtained from the work of Rodrigues et al. [48] and W is the channel width.

As all other parameters are well defined, for each solution Equation 2.7 yields $Re(\eta_{wall}, \dot{Q})$, Equation 2.3 yields $\eta_{wall}(\dot{\gamma}_{wall})$ and Equation 2.6 yields $\dot{\gamma}_{wall}(\dot{Q})$, which means an implicit relation $Re(\dot{Q})$ is obtained: for each solution the Reynolds number can be defined through the control of the flow rate.

As seen in section 1.1, $Wi = \lambda \bar{\gamma}$, where λ is the fluid longest relaxation time, and $\bar{\gamma}$ is the shear rate. Here, the average shear rate is used:

$$\bar{\gamma} = \frac{2(\bar{U} \times 10^{-3})}{H} = \frac{2(\dot{Q} \times \frac{10^3}{3600})}{AH} \quad (2.8)$$

Similarly, and less complex, than what is discussed for Re , as A and H are defined, this equation yields $\bar{\gamma}(\dot{Q})$. Seeing as $Wi(\lambda, \bar{\gamma})$ and λ is a determined fluid property, a relation $Wi(\dot{Q})$ is obtained for each solution.

A Re range of $2.87 \leq Re \leq 45.71$ was used for the Newtonian solution, with $Wi = El = 0$. This corresponds to flow rates between 3.123 mL/h and 49.763 mL/h.

For the viscoelastic solutions, a Re range of $2.86 \leq Re \leq 39$ was used, corresponding to a Wi range of $0.07 \leq Wi \leq 9.4$ and Elasticity numbers $0.02 \leq El \leq 0.33$. The flow rates used were in the range $3.488 \text{ mL/h} \leq \dot{Q} \leq 52.463 \text{ mL/h}$. These values were established in alignment with the initially set goals of studying flows with small Wi numbers (see Subsection 1.3).

Since registered relaxation times were lower than expected, this corresponded, however, to higher Reynolds numbers than what was initially desired.

2.4.3 Extension of analysed channel length

One of the problems that Li and Haward [27] faced was in the limitations of the capture area used for μ -PIV. This thesis proposes a slight alteration in methodology that attempts to extend the x range of μ -PIV measurements without decreasing microscope magnification and measurement resolution.

With a set position of the microchannel in the microscope, the area which the CCD camera is able to record (capture area) is defined. An initial capture area set at the inlet of the microchannel was then established for every solution. However, when it was dimmed beneficial, the microchannel was moved after completing the initial set of measurements, such that the capture area was further downstream. This established a set of capture areas, and doing measurements in each of them extended the x range in which the velocity profiles were analysed.

Downstream measurements would not be useful if the x position was not properly quantified at the position of measurement. To be able to do this, two reference points were established in each capture area, with each reference point being in turn used in two capture areas, to establish its position in the transition from one capture area to another. The first point of the first capture area should be in a position that can be characterised (e.g. the edge of a wall at the channel's inlet). Since the used software is able to measure the relative x and y positions between two points, the position of the remaining reference points can be inductively calculated, such that the positions of each capture area can be determined.

The reference points used could be anything that is noticeable in the microscopic view of the microchannels and is not at risk of moving. Typically, what was used when performing measurements for this thesis was remainders of PDMS debris created in the microchannel fabrication process, as can be seen in Figures 2.16 and 2.17.

Figure 2.21 shows an example of the application of this process, with Figure 2.21a) showing the first capture area, and Figure 2.21b) showing the immediately further downstream capture area.

In Figure 2.21a), point "Origo" marks the initial point, at the edge of a channel wall in the channel inlet. Point "A" marks a big piece of PDMS debris which can easily be identified. From this capture area, it was possible to establish the coordinates of this point in relation to the channel inlet. The same piece of PDMS debris was then set as point "Origo" in the second capture area, seen in Figure 2.21b). Coordinates of the new Point "A", defined for this capture area, could then be established in relation to point "Origo", and therefore in relation to the inlet of the channel. For both pictures shown, point B is established only to define the scale factor and can be ignored.

This process was repeated along the microchannel, depending on necessity. Solutions with higher concentration of XG and higher visco-elastic character were expected to have longer entrance lengths and so more measurements were conducted.

For the viscoelastic solutions, one measurement (consisting of 100 picture pairs) was done per flow rate, per solution, per capture area. This method for extension of the analysed channel

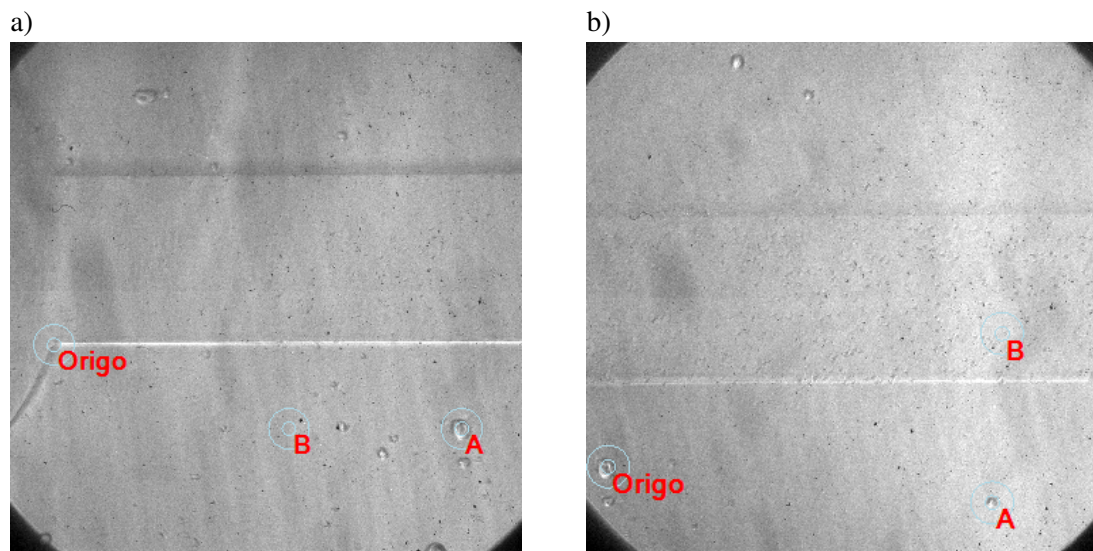


Figure 2.21: Example of application of reference points intended for x range extension.

length was used to a degree that intended to guarantee that some measurements would already show fully developed flow.

For the Newtonian solution, three different sets of measurements were done. All three were repeated at two capture areas: one at the inlet and one far enough away from the inlet where measurements were meant to evaluate the fully developed flow. The method described in this section was not applied to the Newtonian solution measurements since results from the literature [1, 27] indicated that the entrance length should be well within the capture area containing the channel inlet.

All measurements were conducted at room temperature, which was controlled through air-conditioning to be around 22 °C.

Chapter 3

Results and discussion

Data from the PIV software was collected. Velocity in the y -direction was negligible in all measurements, as should be expected in the uni-axial flow, and only the velocity in the x -direction, U was considered. The μ -PIV technique used allowed for the characterisation of velocity profiles throughout the length of the channel. The collected data was run through a MatLab™ script to be organised into $U(x, y)$.

Due to the trapezoidal shape of the channel cross-section (see Section 2.3), it was not always possible to clearly see the exact y position of the channel walls. This information would have been mostly necessary to determine the centre-line of the width of the channel, y_c . The centre-line of the channel width is an important concept and used throughout this section, particularly because of the need to determine the centre-line velocity U_c .

Because of this, an alternative for its determination is given. Seeing as the observed profiles showed symmetry (discussed in more detail in Section 3.1), for a given velocity profile at a defined x , the velocity U at a point with a small value of y was evaluated by a MatLab™ script, which then found the point near the opposing wall with the closest U value, and obtained the coordinate y for this point. y_c was considered to be the mean between the y values of these two points. To avoid random error, the centre-line velocity for the velocity profile at x , was calculated by averaging the velocity evaluated at y_c and at the 5 points nearest to it in both directions (11 points total).

3.1 Inlet velocity profiles

Initially, the inlet velocity profiles $U(y)_0$ were evaluated, to understand the effect of the circular area upstream of the inlet (see Section 2.3) in flow "pre-development" and its potential impact on the results.

Velocity profiles at the measured point closest to the channel inlet ($x \approx 0$), and normalised by their respective centre-line velocity $U_{C,0}$ are shown in Figure 3.1. Profiles of each solution are shown for low and high Re (also indicated) measurements in Figure 3.1 a) and b), respectively.

The profiles shown exhibit symmetry, similar to that reported by [27], with no visible "vena contracta" effects, as shown in [1]. Pre-development is expected to be more accentuated for smaller

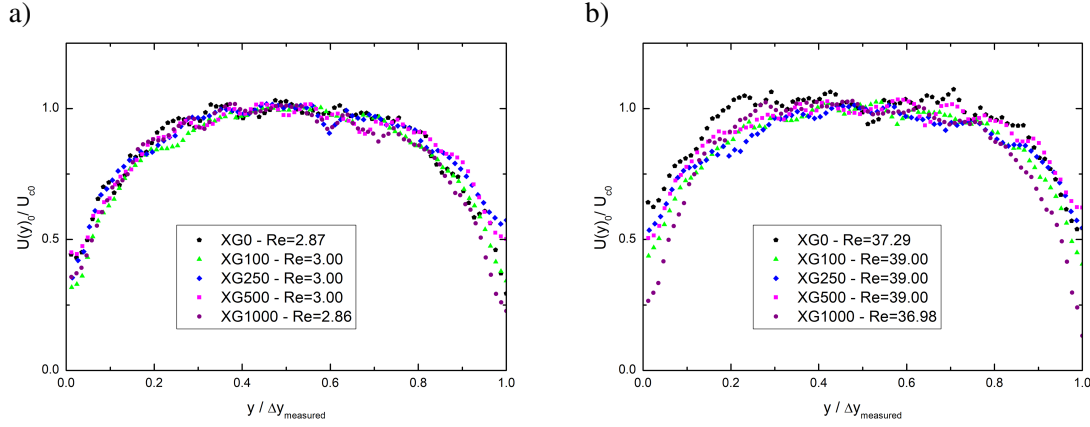


Figure 3.1: Normalised inlet velocity profiles (at $x \approx 0$). a) shows measurements with lower Re numbers than b).

values of Re , which correspond to smaller entrance lengths, and this is evident comparing the inlet velocity profiles for the different Re number, with those of high Re appearing considerably less developed and with a flatter (closer to uniform) velocity profile. Whilst some pre-development does take place - in either of the shown cases the velocity profile is uniform - both still show a somewhat flat shape. Ahmad and Hassan [1] state that a uniform inlet velocity profile is very rarely achieved in practice, especially in microscale experiments. These results were deemed reasonable with no expected considerable impact on the entrance length measurements.

Importantly, pre-development is approximately the same between solutions, with the shape of the inlet velocity profiles being very similar. As measurements of different solutions used different channels¹, this indicates distance from the manually punched entrance tap to the inlet, which varied from channel to channel, did not influence the results.

3.2 Centre-line velocity evolution

3.2.1 Considerations for entrance length calculations

Whilst Li and Haward [27] note that analysis of centre-line velocity may not be enough to determine the entrance length of highly visco-elastic fluid flow, the lower Wi numbers used in these experiments seem to dictate that this is not the case for the collected data. Notoriously, while developing, the velocity profile does not change shape considerably, as had been reported by Li and Haward [27] for high Wi - there are no velocity peaks near the channel walls, nor notorious over-shoots in the centre-line velocity. With that said, evaluation of the centre-line velocity evolution through the channel length $U_C(x)$ should be sufficient for the determination of the entrance length.

¹Different channels from the same SU-mold, as described in Section 2.3. For simplicity sake, since only one channel mold was used, this will henceforth be referred to as different channels.

As mentioned in Section 1.1, a common definition of the entrance length is the distance from the channel's inlet at which the velocity at the centre-line reaches 99% of its fully developed value $U_{C,FD}$. However, there is existing work in the literature of less restrictive definitions, which study the entrance length for which the centre-line velocity reaches 98% of its fully developed value [6]. This eases the high resolution necessities of used methods, both for numerical work and for experimental work.

This thesis will generically define L_{ep} as the entrance length for which the centre-line velocity reaches $p\%$ of its fully-developed value. This section initially discusses how to determine the point at which this condition is met, based on the centre-line velocity evolution data.

Following the definition of the entrance length as the distance from the inlet at which the velocity reaches $p\%$ of its fully-developed value, the simplest way to calculate it would be by direct intersection of a $U_C(x)$ plot with a horizontal line at value $p\% \times U_{C,FD}$.

To use this method, the value of $U_{C,FD}$ must be previously obtained. This can be done with an analytical or numerical solution, or experimentally measured at a point where the flow is fully developed. It should be noted that for visco-elastic fluid flow, a fully developed, stationary state is not dependent on past-times, and therefore its fully developed velocity profile should resemble that of a Generalised Newtonian Fluid (GNF) without memory effects.

If applicable, this method is the most accurate, as the entrance length is obtained exactly in how it is defined. It is the one used in examples of the literature, particularly regarding numerical analysis [51, 15]. However, in experimental studies, experimental or random error associated with the measurements can inhibit its application.

Such was the case here, especially for the measurements of the visco-elastic solutions, as will be further discussed in Subsection 3.2.3. To remove experimental error, the plots were adjusted to fitting functions. These are explained in detail in Subsection 3.2.2.

3.2.2 Newtonian solution

As mentioned at the end of Chapter 2, three sets of measurements were done for the Newtonian solution, each using multiple values of varying $\dot{Q}$ analysed at two capture areas, one at the inlet and one at some point far enough from the inlet that fully developed flow is expected (x/H roughly estimated to be ~ 30 -40).

As an example, the centre-line velocity measurements of the Newtonian solution, for the fully developed capture area and for $Re = 37.29$ ($\dot{Q} = 40.597$ mL/h) is shown in Figure 3.2. Different symbols represent different sets of measurements, which means different channels were used, and the x value is not exactly the same, but is always far from the channel inlet. An analytical solution for the expected value of $U_{C,FD}$ for this flow rate is also shown. This solution approximates the channel cross-section to a rectangle. It was obtained based on the equations presented in the literature [12].

Notoriously, there are very little differences between sets of experimental data, showing good reproducibility. What is more, these show very little random error between them, remaining mostly constant. There is also a clear overlap with the analytical solution.

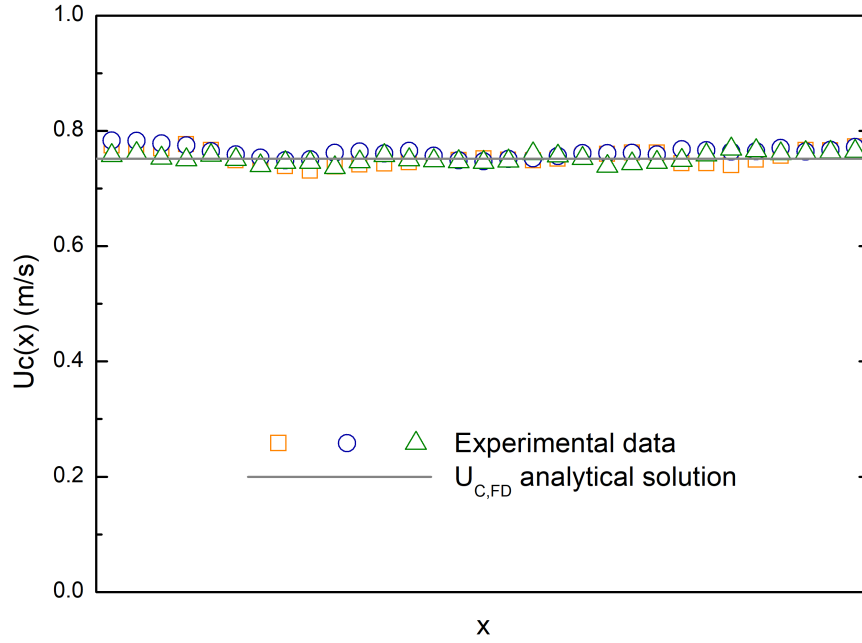


Figure 3.2: $U_C(x)$ evaluated far from the channel inlet ($x/H \gg 0$, measured x range is ≈ 0.76 mm) for XG0, with $Re = 37.29$, correspondent to $\dot{Q} = 40.597$ mL/h.

Sets of data for other values of Re showed similar reproducibility and random error, and remained very close to the analytical solution. This validates the technique used in centre-line velocity measures for the Newtonian solution.

The centre-line velocity was then studied at the inlet. Whilst the random error is small, it was sufficiently large that the plot of $U_C(x)$ would some times intersect the threshold value of $p\% \times U_{C,FD}$ for more restrictive values of p , e.g. 99. This was even more notorious in the inlet measurements regarding the visco-elastic solutions, as will be seen in Subsection 3.2.3.

For this reason, as mentioned in the previous section, a decision was made to approximate the collected experimental data to a fitting function. Whilst some recent work has been done to determine how the velocity evolves in the entrance region [20], the literature is still limited to simple cases, and no theoretical function that describes the analysed cases exists. Nevertheless, looking at the data, it is possible to see some clear trends: the velocity evolves rapidly at points closer to the inlet, and then stabilises at an asymptotic value. This makes sense with what is expected regarding the evolution of the velocity profile (see Section 1.1).

Two different functions which matched this trend were considered. The fitting of the experimental data to these functions was conducted in OriginPro™ v2022.b using the Levenberg-Marquardt algorithm, also known as the damped least-squares method.

Initially, an exponential decay function was defined:

$$U_C^{exp}(x) = U_{C,FD}^{exp} + a_{exp} e^{-\frac{x}{t_{exp}}} \quad (3.1)$$

with parameters $U_{C,FD}^{exp}$, a_{exp} and t_{exp} , where it should be highlighted that $U_{C,FD}^{exp}$ represents the asymptotic value that the function approaches as x increases, and a_{exp} and t_{exp} govern the shape of the curve. This function is simply defined and meets the imposed criteria, with its derivative (representing the rate at which velocity changes along x) being initially high for low values of x and decaying as the velocity reaches its asymptotic value.

Besides that, another function was defined, divided into a linear function to approximate the rapid acceleration observed in points closer to the channel inlet, and a rational function that sets its asymptotic value:

$$U_C^{fit}(x) = \begin{cases} mx + b & \text{if } x \leq s \\ \frac{k}{x-x_0} + U_{C,FD}^{fit} & \text{if } x > s \end{cases} \quad (3.2)$$

This function presented a lot of varying parameters. However, these were simplified by imposing that the function and its derivative must be continuous at $x = s$:

$$ms + b = \frac{k}{s-x_0} + U_{C,FD}^{fit} \quad (3.3)$$

$$m = -\frac{k}{(s-x_0)^2} \quad (3.4)$$

Solving equation 3.3 for b , and equation 3.4 for k and substituting in 3.2 yields:

$$U_C^{fit}(x) = \begin{cases} U_{C,FD}^{fit} + m(x+x_0-2s) & \text{if } x \leq s \\ U_{C,FD}^{fit} - \frac{m(s-x_0)^2}{x-x_0} & \text{if } x > s \end{cases} \quad (3.5)$$

If $x_0 \geq s$ there would also be a vertical asymptote, which besides not making physical sense, hindered the convergence of the fitting process of the software. It was then necessary to impose $x_0 < s$, which was done by defining an auxiliary variable $d = s - x_0$, with $d > 0$, and substituting in Equation 3.5 to finally obtain:

$$U_C^{fit}(x) = \begin{cases} U_{C,FD}^{fit} + m(x-d-s) & \text{if } x \leq s \\ U_{C,FD}^{fit} - \frac{md^2}{x+d-s} & \text{if } x > s \end{cases} \quad (3.6)$$

with four varying parameters, where $U_{C,FD}^{fit}$ is the asymptotic value, m is the extension rate close to the inlet, s marks the separation between the linear and rational branches of the function, and d determines the sharpness of the curve on the rational part of the function.

The software used for the fitting does not allow for branched functions, nor does it define a Heaviside step function. As such, one was defined using the absolute value function, as shown in

Equation 3.7 which was the one inserted in the software:

$$U_C^{fit}(x) = U_{C,FD}^{fit} + m \left[\left(\frac{1 - \frac{|x-s|}{x-s}}{2} \right) (x-d-s) + \left(\frac{1 + \frac{|x-s|}{x-s}}{2} \right) \left(\frac{-d^2}{x+d-s} \right) \right] \quad (3.7)$$

The fit of these functions to the experimental data of the Newtonian solution was quite satisfactory, with all fits showing $R^2 > 0.9$, and the vast majority showing $R^2 > 0.95$. The extra varying parameter usually resulted in better results for the $U_C^{fit}(x)$. Nonetheless, some minor systemic problems were discovered, that were deemed necessary to address. To represent these problems, two examples of measurements of $U_C(x)$ at the inlet are shown in Figure 3.3. Figure 3.3a) represents a measurement at $Re = 37.28$, correspondent to $\dot{Q} = 40.597$ mL/h. Figure 3.3b) represents a measurement at $Re = 32.99$, correspondent to $\dot{Q} = 40.597$ mL/h. The axis for U_C

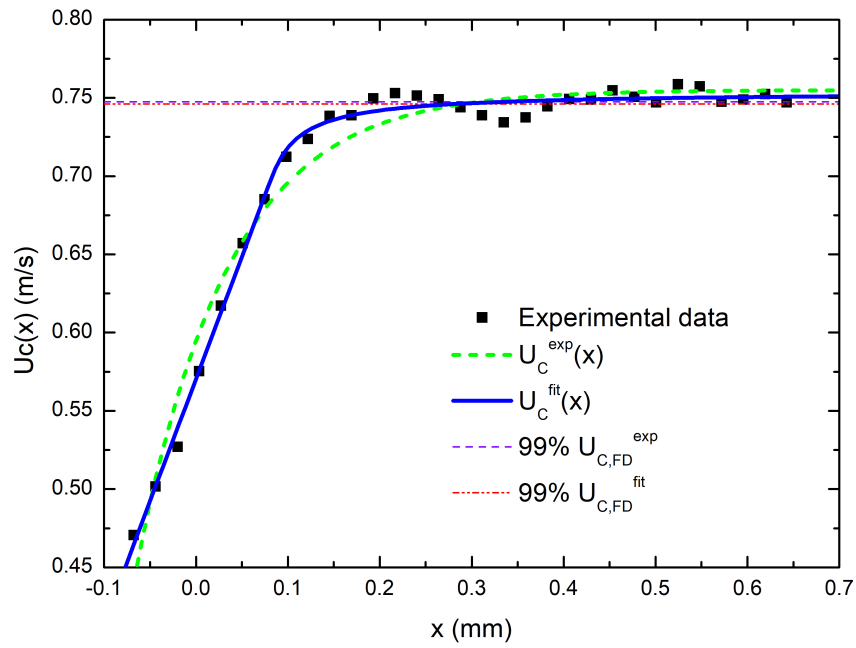
Whilst as mentioned before, both functions used fit the data well, the extra varying parameter in the definition of $U_C^{fit}(x)$ usually allowed the software to find a better fit condition. However, a crucial aspect to consider, is that the exponential decay function $U_C^{exp}(x)$ deviated from experimental data following a pattern, which is shown in Figure 3.3a). $U_C^{exp}(x)$ was often below the curve of experimental data near the "elbow" between the initial fast acceleration and the stable increase as it reaches its asymptotic value. This is a particularly important area, as points of intersection to determine the entrance length may appear here, particularly if lower values of $p\%$ are considered. With that said, it was decided that it was best to adjust the experimental data to the fitting function of $U_C^{fit}(x)$.

However, this function is also not without flaws. If experimental random error places the most downstream values measured at a higher point than expected, this function becomes very sensitive to this error. This may predict asymptotic values $U_{C,FD}$ which are higher than reality, and may lead to significantly different entrance lengths measurements, as seen in Figure 3.3b). This phenomenon becomes even more prevalent in measurements of the visco-elastic solutions, as will be seen in the next sub-section. What is more, whilst for the Newtonian solution experimental data was collected at $x \gg 0$, which showed a stable centre-line velocity, this was not the case for the visco-elastic solutions. As such, the asymptotic value could not be approximated by experimental measurements done to fully-developed flow. Similarly, theoretical predictions given by the analytical solution did not overlap the experimental data for the Newtonian solution with high enough precision that they could be used as threshold values to determine the entrance length. This problem is further accentuated in the case of the visco-elastic solutions.

With all of these considerations in mind, it was decided to approximate the centre-line velocity evolution to the fitting function defined in Equation 3.6, $U_C(x) = U_C^{fit}(x)$. However, the asymptotic value $U_{C,FD}$ would be determined by $U_{C,FD}^{exp}$, as this function proved to be a good approximation outside of the considerations discussed regarding Figure 3.3a).

Figure 3.4 shows the normalised centre-line velocity along the normalised length of the channel for one of the three sets of measurements conducted at the channel inlet for XG0. Figure 3.4a) shows all the collected points and fitting functions as a whole, whilst Figure 3.4b) highlights the

a)



b)

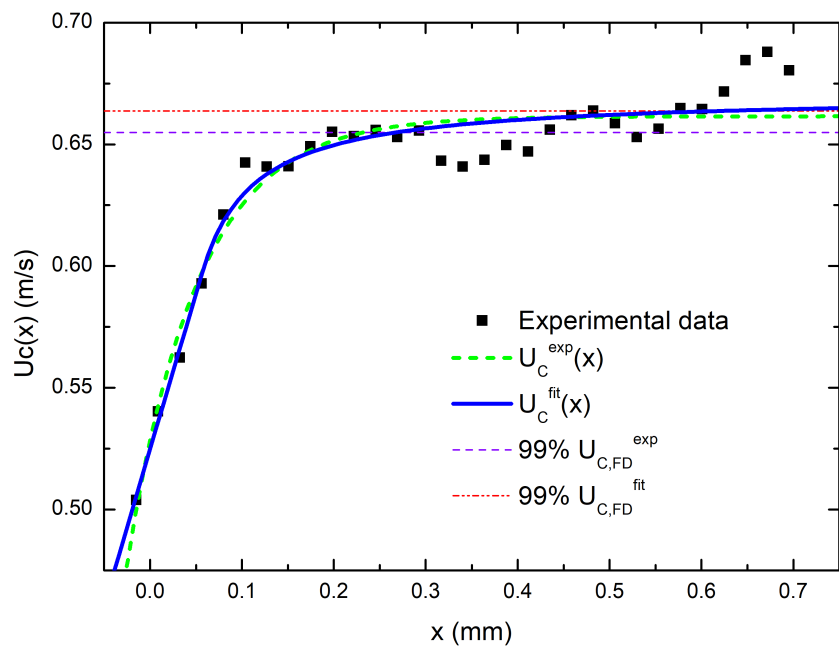
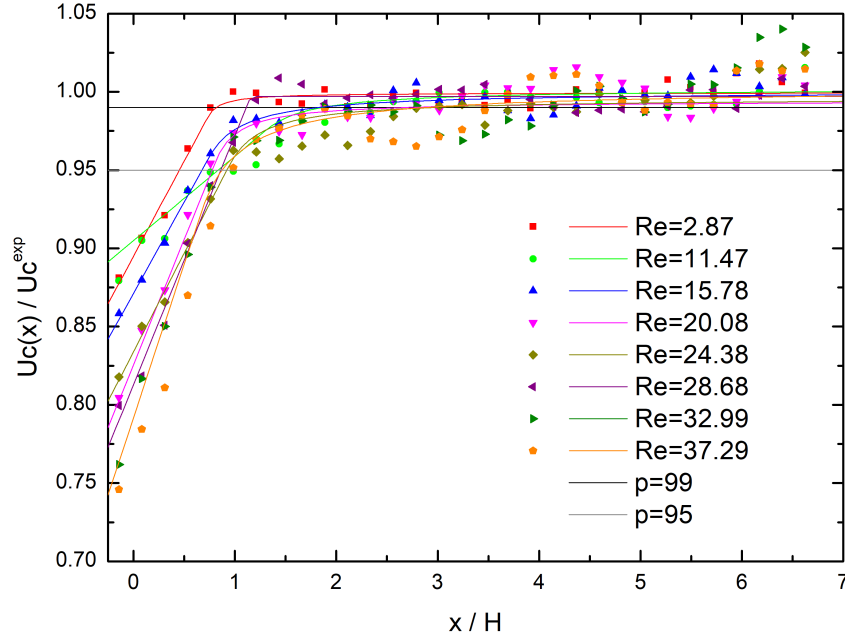


Figure 3.3: Examples of $U_C(x)$ and fitting functions at the entrance of the channel for XG0, with a) $Re = 37.29$, correspondent to $\dot{Q} = 40.597$ mL/h and b) $Re = 32.99$, correspondent to $\dot{Q} = 35.913$ mL/h.

a)



b)

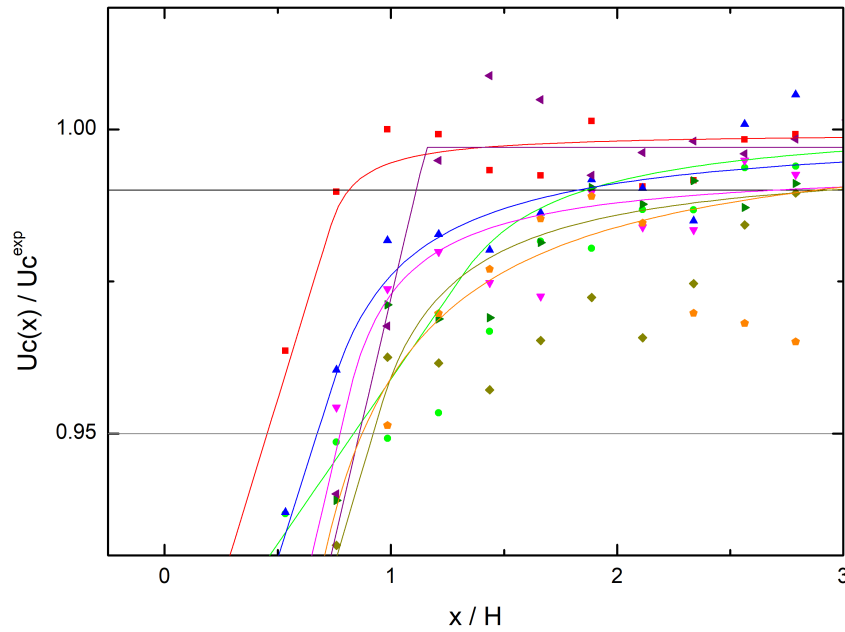


Figure 3.4: Normalised $U_C(x)$ and fitting functions at the entrance of the channel for XG0 and different Re values. a) shows all the collected points and fitting functions. b) highlights the intersections of the fitting functions with threshold lines. Legend shown in a) applies to b).

intersections of the fitting functions with lines showing different values of p , which are used for the determination of L_{ep} .

Once again, a good fit to the experimental data is obtained from the fitting function. It is also once again clear that lower Re numbers correspond to bigger flow pre-development before the channel inlet.

It is worth highlighting the measurements for which the fitting functions behave differently from the batch, for $Re = 11.47$ and $Re = 28.68$. For $Re = 11.47$, a seemingly much lower acceleration is seen near the channel inlet. It can be speculated that this is a result of experimental error which overestimates the velocity close to the inlet and reduces the slope of the linear portion of the fitting function. What is also interesting is how this results in an intersection with $p = 99$ which seems coherent with the data from the other points, but to a very "out of place" intersection for $p = 95$. The opposite can be said for $Re = 28.68$, where it can be speculated that there is an overestimation of the velocity in the transition between the branches of the fitting function, which leads to a much more abrupt change. The intersection with $p = 95$ seems coherent, but intersection with $p = 99$ takes place at a much lower value of x/H than that of similar Re numbers. This shows how even little experimental error in a few points can lead to outlier points in the analysis of the entrance length. Entrance length results are shown and discussed in more detail in Section 3.3.

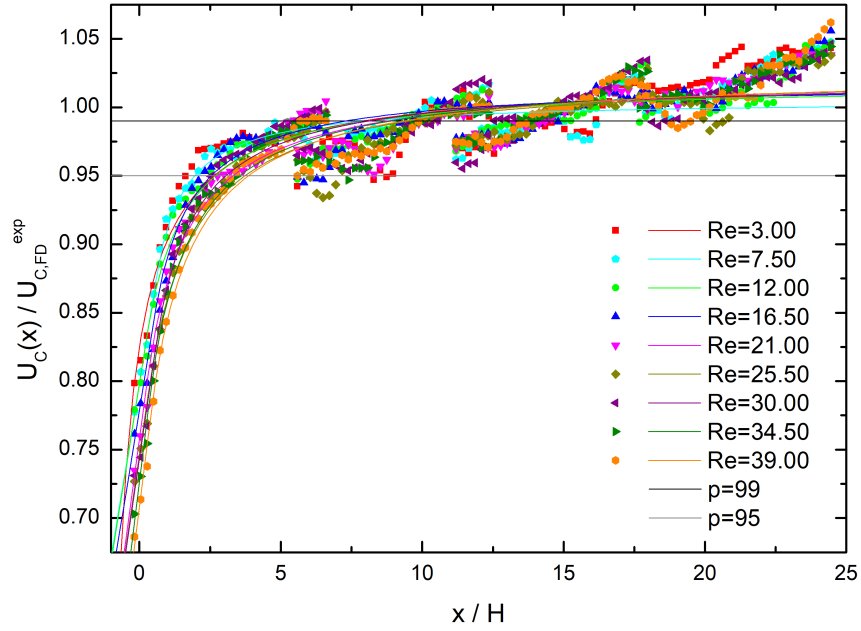
3.2.3 Visco-elastic solutions

The data analysis discussion held in the previous subsection for the Newtonian solution also applies to the visco-elastic solutions. The same fitting function was used, calculated using the same software and method. Similarly, the same method was used for estimating the asymptotic value corresponding to the centre-line velocity of the fully developed flow $U_{C,FD} = U_{C,FD}^{exp}$.

Figure 3.5 shows the normalised centre-line velocity along the normalised length of the channel for one of the three sets of measurements conducted at the channel inlet for XG100. Figure 3.5a) shows all the collected points and fitting functions as a whole, whilst Figure 3.5b) highlights the intersections of the fitting functions with lines showing different values of p , which are used for the determination of L_{ep} . Figure 3.6 shows the same but for XG1000.

Similar results were obtained for XG250 and XG500. A few things stand out from Figures 3.5 and 3.6. Experimental data consistently in both graphs shows incoherence and lack of continuity. The outlines of different capture areas measured (see Subsection 2.4.3) are very clearly seen, with the velocity not matching for the same values of x , and being consistently higher at the end of a capture area than at the beginning of the next one. In the case of XG100, an argument can be made that the velocity is still developing in the highest value of x measured. That is what seems to be indicated also by the fitting function, which, as mentioned in the previous subsection, is sensitive to experimental error creating an increase in velocity for higher values of x measured. However, a still developing velocity is clearly not the case for XG1000, where the velocity itself seems to have stabilised (as should happen as the flow becomes fully developed), the evolution of $U_C(x)$ within each capture area suggest that there is still some acceleration. As these results are incoherent with

a)



b)

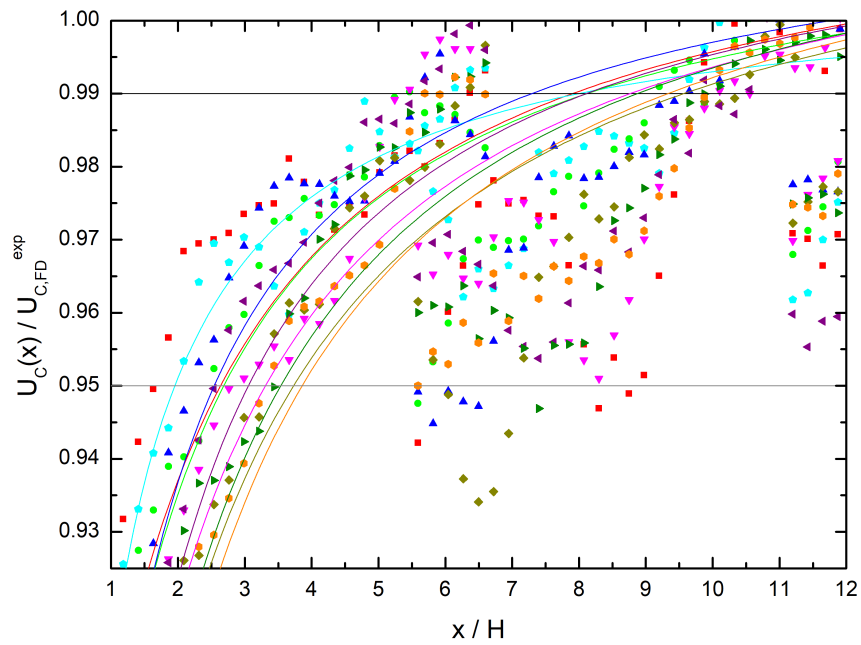
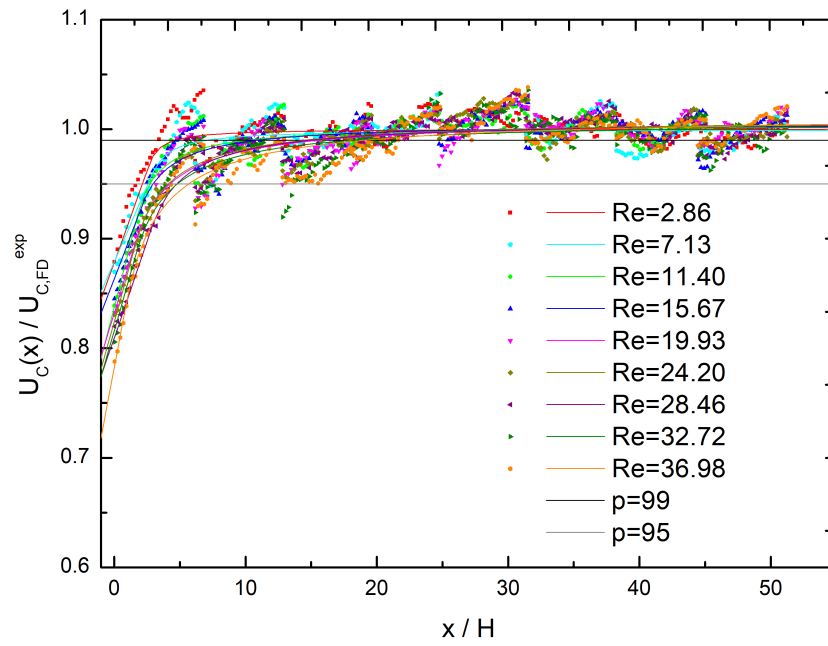


Figure 3.5: Normalised $U_C(x)$ and fitting functions at the entrance of the channel for XG100 and different Re values. a) shows all the collected points and fitting functions. b) highlights the intersections of the fitting functions with threshold lines. Legend shown in a) applies to b).

a)



b)

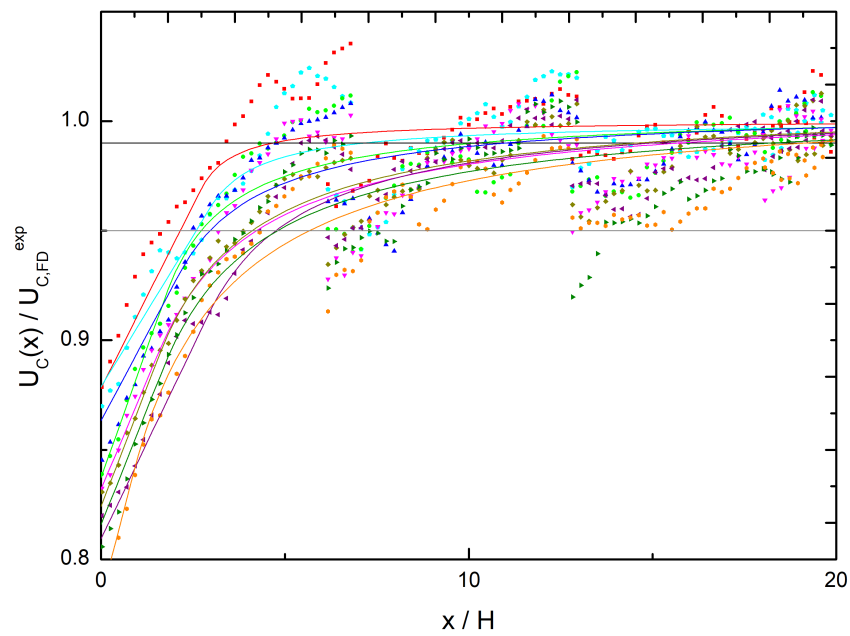


Figure 3.6: Normalised $U_C(x)$ and fitting functions at the entrance of the channel for XG1000 and different Re values. a) shows all the collected points and fitting functions. b) highlights the intersections of the fitting functions with threshold lines. Legend shown in a) applies to b).

themselves, don't make physical sense (velocity is increasing, but overall remaining constant), this suggests a systemic error in the measurements.

The experimental data was adjusted to the fitting functions described in the previous subsection and shown in Figures 3.5 and 3.6. The fitting of the function to the experimental data showed lower values of R^2 when compared to XG0, even if still reasonable (higher than 0.7). This was expected due to the experimental noise observed. Although the experimental data showed signs of a systemic error occurring in the measurements, it was still dimmed possible that the fitting function dampened the effects of this error, and so the entrance lengths were calculated based on the fitting functions. However, since relative error in the data is considerable, measuring it for high, restrictive values of p was dimmed to not make physical sense. Entrance length for the visco-elastic solutions was therefore only evaluated for $p = 95$.

3.3 Entrance length

As decided in Subsection 3.2.2, the entrance length L_{e_p} was determined as the x value for which the fitting function would reach $p\%$ of the defined asymptotic value:

$$U_C^{fit}(L_{e_p}) = p\% \times U_{C,FD}^{exp} \quad (3.8)$$

Substituting $U_C^{fit}(L_{e_p})$ by its definition in Equation 3.6 and solving for L_{e_p} gives:

$$L_{e_p} = \begin{cases} \frac{p\% \times U_{C,FD}^{exp} - U_{C,FD}^{fit}}{m} + d + s & \text{if } L_{e_p} \leq s \\ \frac{md^2}{p\% \times U_{C,FD}^{exp} - U_{C,FD}^{fit}} + d + s & \text{if } L_{e_p} > s \end{cases} \quad (3.9)$$

Once the values of the fitting functions were determined, the entrance length was obtained from Equation 3.9.

3.3.1 Newtonian solution

Although the visco-elastic solutions are only evaluated at $p = 95$, the Newtonian solution's measurements showed little experimental error. What is more, values in the literature exist for the dimensionless entrance length as a function of Re for $p = 99$. $L_{e_{99}}$ was then calculated for all the measurements of the three sets of measurements done for XG0. The obtained results, a linear regression from them obtained and the plots of the equations present in the literature [1, 27] are shown in Figure 3.7. The equations presented in the literature [1, 27] normalise the entrance length using the hydraulic diameter of the channel cross-section. These were adjusted considering the aspect ratio of the channels used in [1, 27], in order to be compared with the results shown here for Le/H .

The three sets of data show an okay linear correlation between Re and Le/H individually, and the linear correlation obtained from the three sets of data seems representative enough of the trend displayed by the data. The previous section showed how little experimental error may lead to

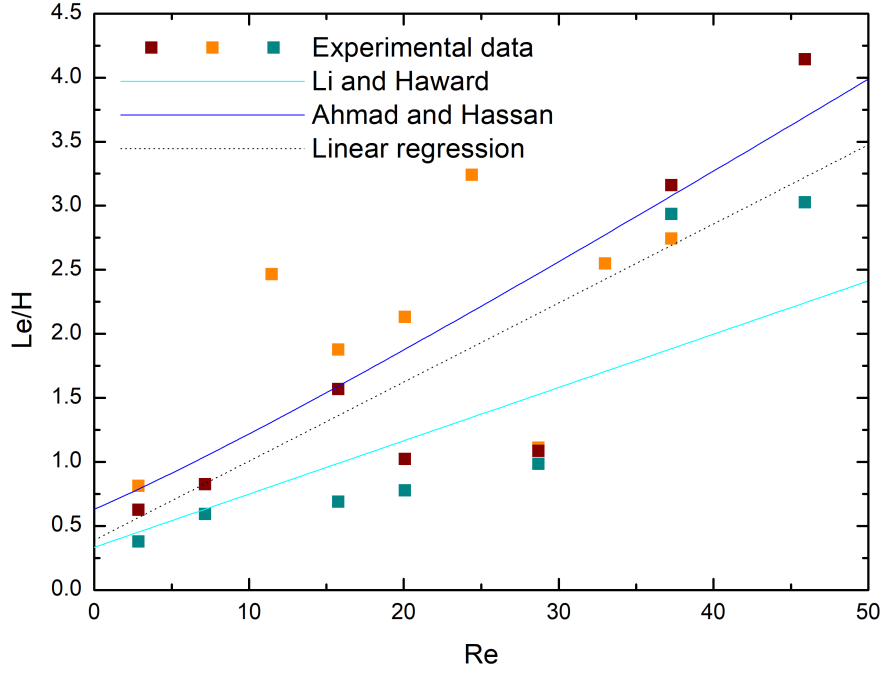


Figure 3.7: L_{e99}/H for XG0 and comparison with equations from the literature [1, 27].

outlier points (see Figure 3.4). It is worth pointing out that Figure 3.4 shows data from the set of measurements which shows higher values of entrance length seen in Figure 3.7. The obtained equation from the linear regression was:

$$\frac{L_e}{H} = 0.057Re + 0.337 \quad (3.10)$$

For a better comparison with circular channels, and with the equations present in the literature [1, 27], Equation 3.10 was also generalised for normalisation using the channels' hydraulic diameter instead of height. Considering a rectangular channel, this led to the equation:

$$\frac{L_e}{D_h} = 0.040Re + 0.233 \quad (3.11)$$

As can be seen in Figure 3.7 this yields an expression which is in between the results obtained by Ahmad and Hassan [1] and Li and Haward [27], slightly closer to the first. This aligns well with what was expected, as Ahmad and Hassan [1] use a channel with a reservoir upstream of the inlet, resulting in little pre-development, and Li and Haward [27] describe the entrance length past a contraction, which has significant pre-development due to flow in a channel upstream of the inlet. The geometry studied in this thesis (see Section 2.3) is expected to have slightly more pre-development than Ahmad and Hassan [1] and less than Li and Haward [27], which is reflected in the obtained results.

For comparison with the results of the Visco-elastic solutions, L_{e95} was also calculated and is plotted in Figure 3.8.

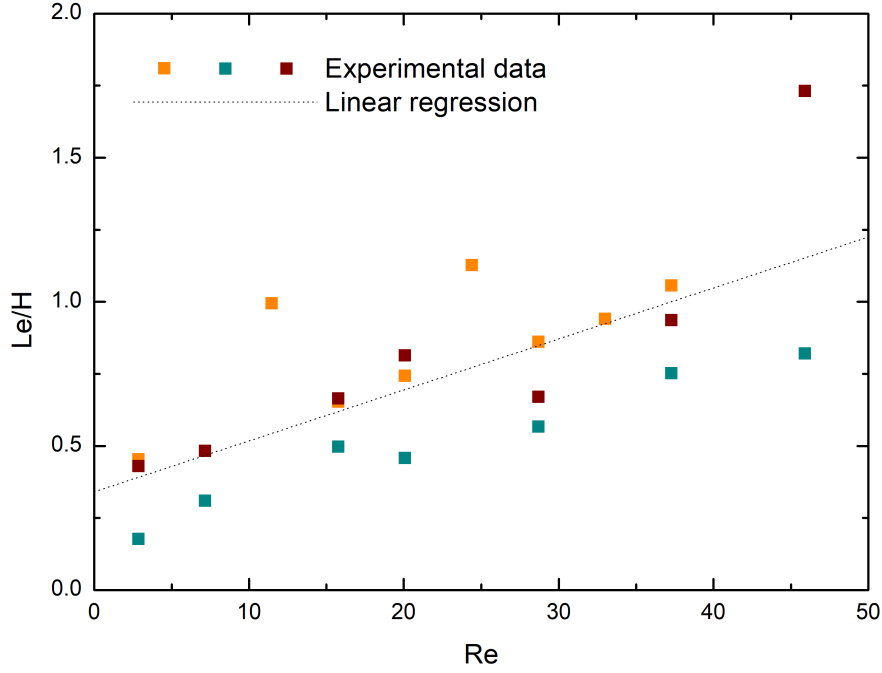


Figure 3.8: L_{e95}/H variation with Re for XG0.

The linear function shows a better fit here, and there is less variation between the results, although some outliers still persist.

3.3.2 Visco-elastic solutions

L_{e95}/H varying with Re is shown in Figure 3.9 for all the studied solutions. It is worth reminding that the Wi number increases with XG concentration.

Incoherence can be found in these results. Most evidently when it comes to the role of elasticity effects in the entrance channel. Considering that differences between the viscosity of XG0 and XG100 are negligible in the used values of $\dot{\gamma}$ (see Figure 2.4), the big difference in the entrance lengths of these two solutions would have to be justified by elastic effects. If the elastic effects are so influential, however, the entrance lengths of XG1000 should be higher than that of XG500, and much higher at the larger Re numbers, where differences in viscosity between these two solutions also become much smaller, and differences in Wi numbers become much greater. Nothing was found in the obtained results that points towards a concrete reason as to why XG1000 measurements are lower, or alternatively why XG500 solutions are higher than expected.

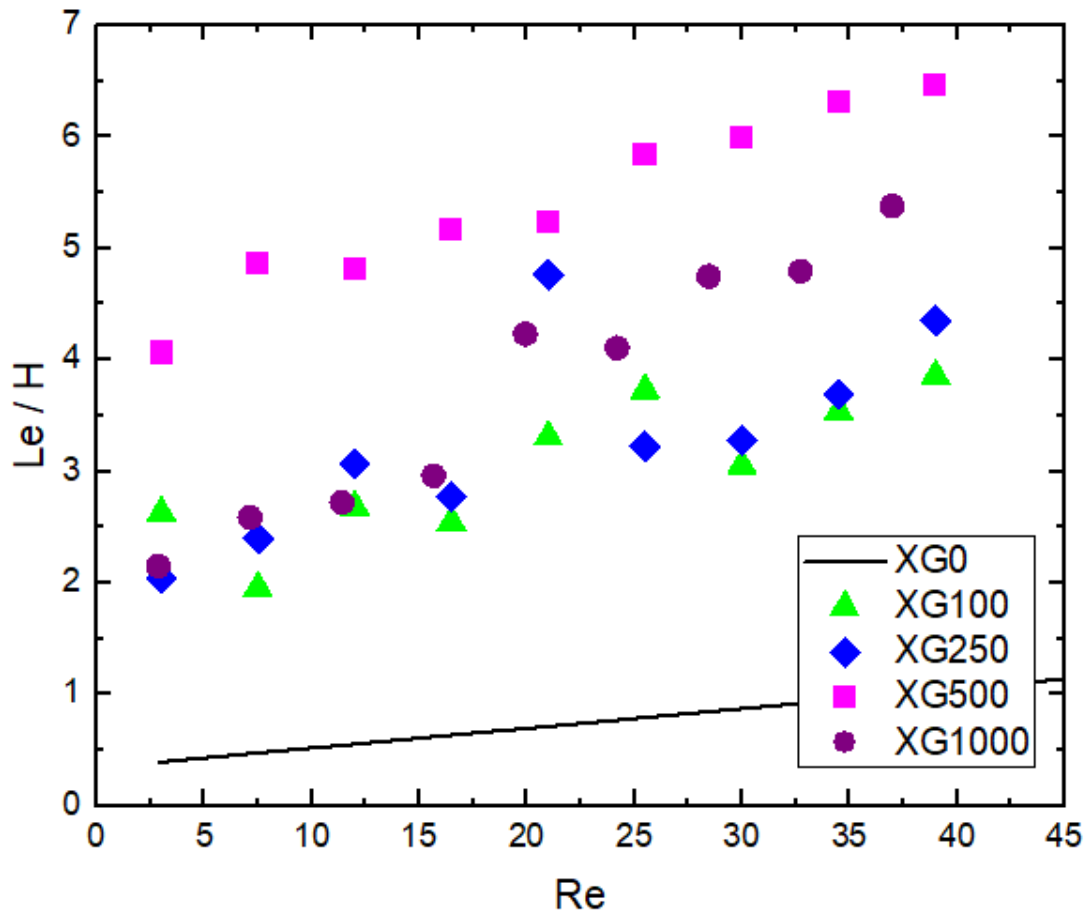


Figure 3.9: L_{e95}/H varying with Re for all the studied solutions.

The most likely explanation, and the only logical explanation found for this incoherence was the systemic error already registered in the centre-line velocity: flaws in the experimental data collected had too big of an impact, and effectively inhibited the aim of the thesis.

No clear cause of the systemic error was found. Seeing as it is present in all visco-elastic solutions measurements, all using different channels, it would seem to be correlated with methodology or equipment used in all measurements (SU mold, syringe, syringe pump or optical set-up). Seeing as the procedure and equipment used for the Newtonian fluid was the same, but these measurements were conducted first, it is hypothesised that experimental use may have led to malfunctions of some of the equipment. However, no specific prove for this was found, and it remains a hypothesis.

Chapter 4

Conclusions and future work

4.1 Conclusions

This thesis aimed to study the development length of viscoelastic fluid flows in microchannels, through the use of μ -PIV.

To this effect, preparation and rheological characterisation of solutions with low levels of elasticity were conducted. The working fluids were polymeric solutions that showed shear-thinning behaviour and relatively small values of the longest relaxation time that would allow to characterise the entrance length at Wi and Re values missing in the literature [27].

Besides that, microchannels for the fluid flow were fabricated and characterised. While some asymmetries were found in the channel geometry, this did not seem to greatly influence results from μ -PIV analysis of the entrance length, which showed symmetrical profiles.

The μ -PIV analysis of Newtonian solution flow through the microchannel showed very little experimental noise, good repeatability and profiles and centre-line velocities which aligned with theoretical predictions. Centre-line velocities evolution downstream of the inlet was measured and approximated to a fitting function, which allowed for the determination of the entrance length at which the velocity reaches 95% and 99% of its asymptotic value. Entrance length results obtained for the 99% threshold aligned with results present in the literature, and a relation between the dimensionless entrance length and the Re number was obtained:

$$\frac{L_e}{H} = 0.057Re + 0.337 \quad (4.1)$$

For a better comparison with circular channels, this equation was also generalised for normalisation using the channels' hydraulic diameter instead of height, which led to the equation:

$$\frac{L_e}{D_h} = 0.040Re + 0.233 \quad (4.2)$$

Results for the visco-elastic solutions' flows were much less consistent, and showed high experimental error. Seeing as the procedure and equipment used for the Newtonian fluid was the same, no clear explanation for the difference in results was found. These are consistently poor for

different fluids and different channels (all fabricated from the same SU-mold, which was also used for the Newtonian solution). Since the Newtonian solutions measurements were conducted first, it is hypothesised that experimental use may have led to malfunctions of some of the equipment used (channel mould, syringe, syringe pump, or optical set-up).

The Newtonian solution's measurements did not use the method for extension of the measured x range using reference points, which means it was only used in measurements with bad data. However, since the method only served to determine the x position of the profiles measured further downstream, the correlation between bad measurements and use of this method is expected to be coincidental and not causal. Because of this, however, it is not possible to evaluate the success of the suggested method.

Whilst it was still possible to approximate the obtained experimental data to a fitting function, and determine, based on that, an entrance length for which centre-line velocity reaches 95% of its asymptotic value, these results were greatly inconsistent. The only clear interpretation was that the flaws in the experimental data collected had too big of an impact, and effectively inhibits achieving the thesis aim of obtaining a dimensionless relation between the entrance length, Re and Wi .

4.2 Future work

Whilst the thesis failed to achieve its goal, it would seem this was due to experimental systemic error. The procedure described, particularly regarding the use of fluids with low elasticity, and the use of reference points for extending the measured length of the channel downstream of the inlet, still seem to be appropriate.

With that said, the most notable future work suggestion is the repetition of the measurements carried out for the visco-elastic solutions.

Based on the experience of this work, a few suggestions are made to improve certain aspects, and facilitate the work to be carried out.

If it is possible to design a channel mould for entrance length measurements, a few suggestions are presented:

- Including a reservoir before the channel inlet could further diminish effects of "pre-development" in the channel entrance, as described by [1]. The inclusion of a reservoir in the channel mould would be compatible with the described fabrication process.
- Diminishing geometrical inaccuracies, such as the trapezoidal shape registered in the microchannel used in this work, would allow for better comparisons with analytical solutions for Newtonian fluids and GNFs.
- Including reference points by design in the mould, near but outside of the channel, and preferably at evenly spaced intervals would allow the channel to act as a ruler and considerably facilitate measurements that need to occur further downstream, and without the need to search for debris. Once again, such a design would be compatible with the fabrication process used.

The implementation of low Wi values resulted in easier to measure velocity profiles and apparently allowed for entrance length analysis based on centre-line velocity. Nonetheless, analysis for higher elasticity, and particularly analysis of flows such as the ones described by Li and Haward [27] with velocity peaks near the channel walls could be interesting. Measurements based on reference points, particularly if using reference points designed into the microchannel mould, could allow for determination of entrance lengths of very high order, such as the ones expected for those flows.

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