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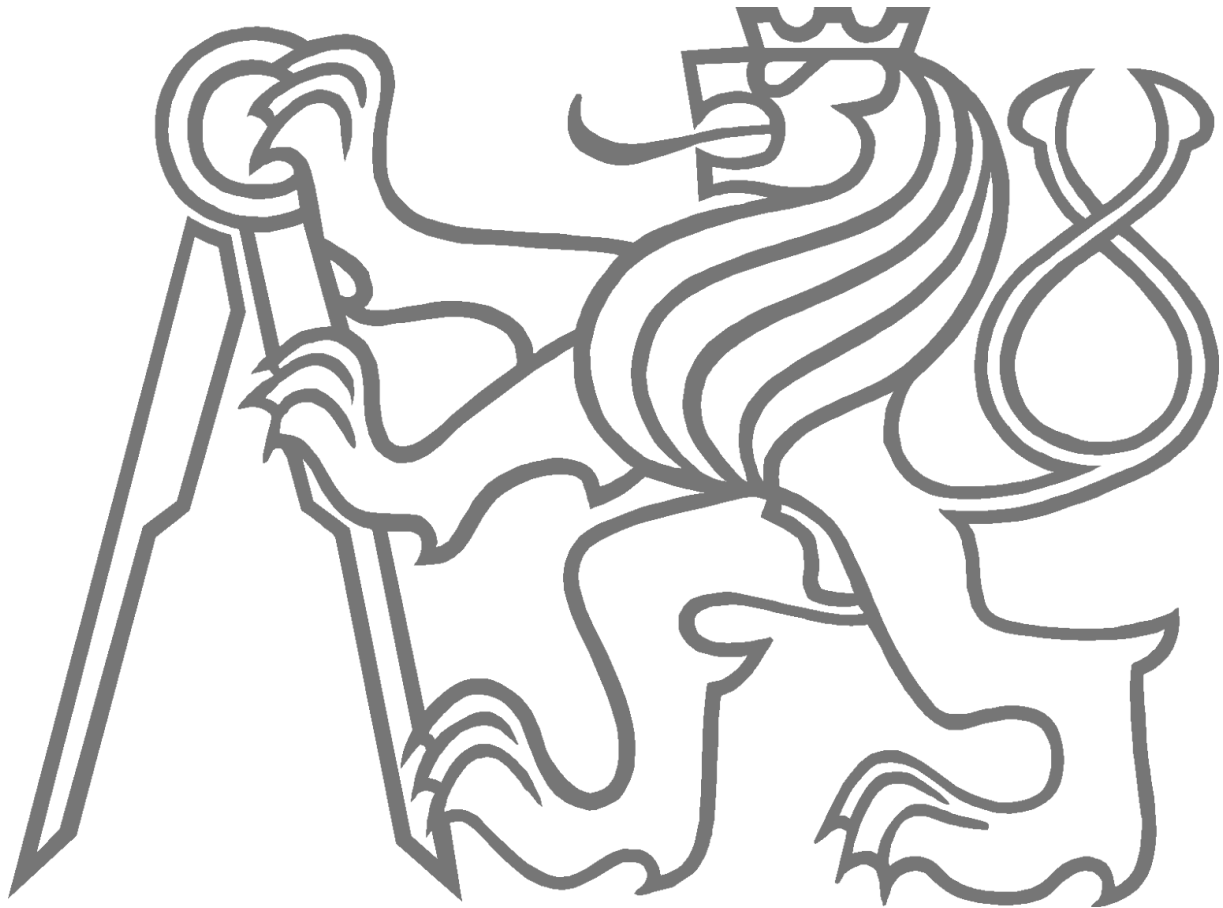# Department of Process Engineering

Rheology of collagen material
Bachelor Thesis

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Keywords: collagen flow, rectangular slit, type 2 collagen flow, the flow behavior of collagen, power-law model collagen, Herschel-Bulkley model collagen

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Abstract

The thesis deals with the investigation of the flow behaviour of collagen material. Literature search focused on collagen material description, the flow behaviour of collagen, experimental techniques to obtain information about flow behaviour of collagen material and theoretical approaches to explain and describe of the flow of material similar to collagen. From the literature search, the best device for measurement collagen material in the form of dough is slit rheometer. There are lack information about rheology of collagen material. Pure viscous power-law model or Herschel-Bulkley model are used for the flow description of collagen material.

The experiment results from literature were used for the rheology of collagen investigation. Measurements were performed on slit rheometer with three different capillaries. Slits have a rectangular shape with constant width and various thickness of 2, 4 and 8 mm.

This thesis uses regression analysis done on the slit rheometer with different heights. The power-law model and Herschel-Bulkley model are applied after regression analysis. Data is obtained from 5 different pressure transducers and evaluated in my own script in MATLAB. The exit pressure relation for slit rheometer is also covered in this work.

Keywords: collagen flow, rectangular slit, type 2 collagen flow, the flow behaviour of collagen, power-law model collagen, Herschel-Bulkley model collagen

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1 – Introduction

This diploma thesis is focused on the investigation of the flow behaviour of the collagen material. Collagen is the most abundant structural protein in mammals. It is a major component of connective tissue, skin, bone, cartilage, and tendons [1]. Understanding the flow behaviour of a material is important for us in order to successfully design a machine or apparatus that able to transfer bulk liquid from a position to another position.

I used previous experimental data from literature and applied two different rheological models to find out the flow behaviour of the collagen. In order to perform those operations, I used MATrix LABoratories(MATLAB) to assemble my own script to handle the process. This script will identify the flow behaviour by regression and optimization methods which will be applied to experimental data. The exit pressure relation is also examined for possible viscoelastic effect identification.

The flow behaviours are mostly covered in the rheology field of science. Rheology investigates the relationship between the deformation or deformation rate to stress in the material. Rheology offers us many techniques (measurement) by which we can investigate the behaviour of the material.

Rheology has the idea that everything has a time scale, and that if we are prepared to wait long enough, then everything will flow [2].

Understanding the flow behaviour is crucial for us in order to understand how the material reacts with the deformation caused by stresses. These phenomena can be analogically expressed as viscosity, shear rate etc If we have enough information about how the material flows, we can successfully estimate required parameters of the material in order to design a mechanical equipment and for demanded operation. These data not only help us to design machines but also can help us to estimate the energy cost of the machine.

2 – Literature Search

The literature search is mainly focused on the investigation of the collagen flow described in publications. The main objectives of the literature search are collagen material description, the flow behaviour of collagen, experimental techniques to obtain information about flow behaviour of collagen material and theoretical approaches to explain and describe of the flow of material similar to collagen.

Our topic is mainly focused on the observe and find a suitable rheological model for the type II collagen material.

Type II collagen is fibrillar collagen and the main component of cartilage and skin. The cartilage collagen constitutes 95 % of the collagens and approximately 60 % of dry weight. Mutations in type II collagen result in several types of chondrodysplasia, leading to premature osteoarthritis [3].

Collagen type II is believed that it works by causing the body to produce substances that fight pain and swelling. Effects are still unknown. It is only problematic to the people with the "Egg or Chicken" allergies where the body may react to it.

Type II collagen has also another use in the food industry where it is converted into gelatine material which is used to cover processed food. I can say that the main use of it in the food industry is packaging and production of food additives.

Our focus is regarding the rheological parameters for the collagen made from bovine skin. It has importance due to this dough-like material transportation. This material tends to have high viscous forces which will resist highly against deformation which makes harder to operate on it. If my evaluated data are precise enough, we can design a cost-effective pump in order to transfer the liquid. Currently, high shear screws are used to transfer the liquid. Those screw pumps work by positive displacement where the bulk liquid transfer by the motion of a screw axially.

Although it looks promising material in the pharmaceutical industry, especially for fighting against inflammatory, I believe that administration of NSAID (Non- steroidal anti-inflammatory drugs) and SBID (Steroid based Inflammatory drugs) can be more effective for those cases. Especially corticosteroids have proven effect of autoimmune suppressing behaviours which can be administrated via nasal or oral in order to prevent asthma attacks. Glucocorticoids can be also administrated for the surgery in order to prevent swelling and reducing the pain of the patient. We can take a look from the notes of the research paper which is quoted below. These drugs are made from natural collagen. / These drugs can be substituted by natural collagen.

"Perioperative use of corticosteroids has been advocated for the reduction of pain, oedema, and trismus following oral surgical procedures. ... Corticosteroid doses ranged from 80 to 625 mg hydrocortisone equivalent anti-inflammatory dosage. No significant adverse reactions were noted [4]."

2.1 – Material

The samples of collagen 1, collagen 2, and collagen 3 differ in the mass fraction of the collagen, and also in the date of production. The samples were prepared by the same mechanical treatment from bovine skin, with no significant temperature shocks, for example, gelatinization. The collagen is purely natural material, with no chemical additives [18].

Products made from collagenous material are manufactured at various filed of the industry in many form and composition. Vascular grafts in biomedicine [13] or sausage castings in the food industry [14] are representative products made from collagenous materials. However, there is a lack information about rheological properties of these materials [15], which are necessary for the design of production lines. The capillary rheometers are usually used for measuring rheological characteristics of polymer melts and dough (like collagenous materials). In addition, they are currently using for quick evaluation of the product quality at production lines. The investigated material exhibits the compressibility and standard capillary rheometer is not capable to record the compressibility effect. The new capillary extrusion rheometer is equipped with multiple pressure transducers installed along the capillary. The axial pressure profile, as well as exit pressure at capillary die exit, is evaluated. Moreover, the flow index and consistency coefficient of power-law rheological model is evaluated for collagenous material.

Differences in rheological behaviour between the samples may have been caused by differences in the qualities of the natural bovine skin input material (different season of the year, different farmers, different breeds of cow, different harbour conditions, and so forth)

There is a lack information especially about this kind of material. Only a few publications can be found. The publications about the collagen material are mostly regarding chemical behaviour and medical appliances. It seems that the main idea of using this material is regarding food packaging and supplementary use for cartilage improvement yet there is no evidence to prove its improvement for cartilage.

2.2 – Rheology

Rheology is science, which can help us to identify the flow behaviour of collagen material. Rheology has the idea that everything has a time scale, and that if we are prepared to wait long enough then everything will flow. From a rheological point of view, Deborah number is the ratio which defines how much time is required for the matter to flow (De = relaxation time/time scale). If the ratio is smaller matter tends to flow easily which is also easy to deform [1].

$$De = \frac{\lambda}{t_{flow}} \tag{2.1}$$

Weissenberg number is a scalar number used in the viscoelastic flow. It is defined by the shear rate ($\dot{\gamma}$) times relaxation time (λ).

$$Wi = \frac{elastic\ forces}{viscous\ forces} = \frac{\lambda\mu\dot{\gamma}^2}{\mu\dot{\gamma}} = \dot{\gamma}\lambda \qquad (2.2)$$

Deborah number is often confused with Weissenberg number. The Weissenberg number indicates the degree of anisotropy or orientation generated by the deformation and is appropriate to describe flows with a constant stretch history, such as simple shear. In contrast, Deborah number should be used to describe flows with a non-constant stretch history, and physically represents the rate at which elastic energy is stored or released [1].

For the higher ratios material can be considered as solid and materials have higher resistance to shear or tensile stress, which cause more time required to flow.

The term called as viscosity plays an essential role in rheology. Viscosity is resistance to gradual deformation by shear stress or tensile stress. Higher the viscosity means that higher the internal friction of the liquid. Pure shear viscosity is used for Newtonian fluid and it has two types – dynamic and kinematic. Apparent viscosity is used for non-Newtonian fluids.

We often need a high viscosity to resist flow at the low forces posed by gravity, but at the same time, one needs a low viscosity for the same product under the high forces experienced in pouring, squirting or brushing. This is where the non-Newtonian viscosity varying with applied force or flow rate properties are very important [2].

Viscosity or Apparent Viscosity will affect the flow of the material. Flow means that the fluid is moving from one point to its adjacent point.

There are two basic kinds of flow with relative movement of adjacent particles of liquid; they are called shear and extensional flows. In shear flows liquid elements flow over or past each other, while in extensional flow, adjacent elements flow towards or away from each other. [2].

There is another property of material which is called as "Elasticity". Elasticity is the ability of a body to resist a distorting influence and to return to its original size and shape when that influence or force is removed. In another word, the ability of an object or material to resume its normal shape after being stretched or compressed; stretchiness. If the material is elastic, the object will return to its initial shape and size when these forces are removed. These quantities are related to solid-like material.

For both types of fluids, the rate of strain depends on the stress applied and the type of material. Example models of viscoplastic liquids include Bingham plastic, Hershel-Buckley and Cassone (Viscoplastic Materials). For Viscoelastic models, the simplest is the Maxwell and Kelvin-Voigt model, which are combined at varying degrees of complexity to form more 'real' responses. These models give a reasonable example of how typical fluids behave at different strains.

The newtonian fluid is a fluid in which the viscous stresses arising from its flow, at every point, are linearly proportional to the local strain rate-the rate of change of its deformation over time. That is equivalent to saying those forces are proportional to the rates of change of the fluid's velocity vector as one moves away from the point in question in various directions.

More precisely, a fluid is Newtonian only if the tensors that describe the viscous stress and the strain rate are related by a constant viscosity tensor that does not depend on the stress state and velocity of the flow. The Newtonian fluids are the easiest models we can describe due to linear relation.

In a short way, viscoplastic material can go back to original shape until yield stress is reached. For the viscoelastic materials, deformation can occur at any time. Therefore, since the fluidity is depending on the type of stresses which deforms the material to let it flow, the type of material is important for rheology.

If we talk about a material viscosity part, the viscosity is defined as the ratio of the shear stress to the shear rate. In shear flows where the flow is generated by sliding one layer atop another,

with the fluid in-between,

The quantities of interest are the shear rate, $\dot{\gamma} = \frac{V}{h}$

(V is the velocity of the top plate in the x-direction, the bottom plate is fixed, h is the distance between the plates), and the shear stress, $\tau = F/A$ (F is the shear force on the top plate, A the fluid contact area). The shear stress is an odd function of the shear rate. In addition, with viscoelastic fluids, there may be a normal force on the plates. When a steady flow is established, the viscosity is defined as [2]

$$\eta = \frac{\tau}{\dot{\gamma}} = \frac{\frac{F}{A}}{\frac{V}{h}} = \frac{Fh}{AV} \ [Pa.s]$$

(2.3)

For a Newtonian fluid, its viscosity is a constant (having units Pa.s) depending only on the temperature and pressure only unlike to others which are depended with deformation rate or time; nor does such a liquid display any elastic properties or extensional anomalies. This is the behaviour called "shear thinning". For the materials which behaves more like solid (e.g. dough), this method will be nonsense due to its continuously increasing.

The viscosity of all simple liquids decreases by temperature increases due to increasing Brownian motion.

There is also different behaviour of some materials where they will increase viscosity with the shear stress. This type of materials called "dilatant" in other terms "shear thickening". This behaviour is only one type of deviation from Newton's Law, and it is controlled by such factors as particle size, shape, and distribution. That is the reason why it exists in suspensions. The properties of these suspensions depend on Haymaker theory and Van der Waals forces and can be stabilized electrostatically or sterically.

With enough shear rate, all liquids will become non-Newtonian.

2.2.1 Theory of flow

In this chapter, we will be more focused on mathematical models for viscoelastic and viscoplastic materials. In case of ideal materials such as plastic, viscous and elastic are failed to explain the behaviour of the material, we must proceed to better models which is the combination of the ideal models in series either parallel such as viscoplastic and viscoelastic [3].

In this chapter, we are more focused on the viscoelastic and viscoplastic materials mathematical behaviours.

Firstly, we will start off with the definition of viscous behaviour and their types.

This question is often best answered by example. Imagine a styrofoam cup with a hole in the bottom. If I then pour honey into the cup I will find that the cup drains very slowly. That is because honey's viscosity is large compared to other liquids' viscosities. If I fill the same cup with water, for example, the cup will drain much more quickly.

Viscosity is a measure of a fluid's resistance to flow. It describes the internal friction of a moving fluid. A fluid with large viscosity resists motion because its molecular makeup gives it a lot of internal friction. A fluid with low viscosity flows easily because its molecular makeup results in very little friction when it is in motion.

Gases also have viscosity, although it is a little harder to notice it in ordinary circumstances [5].

The generalized formula for viscosity can be expressed by Newton's equation for fluids:

$$\frac{F}{A} = \eta\dot{\gamma} \qquad (2.2.1)$$

Where A represents area and F is for the force. $\dot{\gamma}$ stands for the shear rate as we discussed in the previous chapters.

Most common fluids, called Newtonian fluids, have a constant viscosity. There is a greater resistance as you increase the force, but it's a constant proportional increase. In short, a Newtonian fluid keeps acting like a fluid, no matter how much force is put into it.

In contrast, the viscosity of non-Newtonian fluids is not constant, but rather varies greatly depending on the force applied. A classic example of a non-Newtonian fluid is Oobleck (sometimes called "slime," and often made in elementary school science classes), which exhibits solid-like behaviour when a large amount of force is used on it. Another set of non-Newtonian fluids is known as magnetorheological fluids. These respond to magnetic fields by becoming nearly solid but reverting to their fluid state when removed from the magnetic field [6].

We are going to also introduce some Viscoelastic properties. We can firstly start off by examining the name itself: Viscoelastic where visco stands for viscous properties of the material and elastic stands for elastic behaviour of the material. As we can understand from the name of the material, we can observe both behaviours in those types of materials.

As a reminder, I will describe the short definition of the elasticity.

Elasticity is a property of a material that ability to bear stress and go back to its initial state after force/stress is removed. The material will have an elongation of its initial shape during the stress state. This property firstly introduced to us by Sir Robert Hooke in 1678 where it describes the linear relation between elongation and axial stress which is named after its creator "Hooke's Law". This behaviour described by formula as

$$\sigma = E\varepsilon \tag{2.2.2}$$

Where σ stands for stress and ε stands for strain and lastly E is the ratio of unit stress to unit strain also known as Young's modulus. E is a unique material variable where it depends on material compositions.

Our material collagen consists of fibrous structures where it may elongate under the stress which will affect the material deformation.

The dependence of viscoelastic response can be described by identical ordinary differential equations in time.

These models are helping visualize molecular motions employs "spring-dashpot" models. These mechanical analogy use "Hookean" springs.
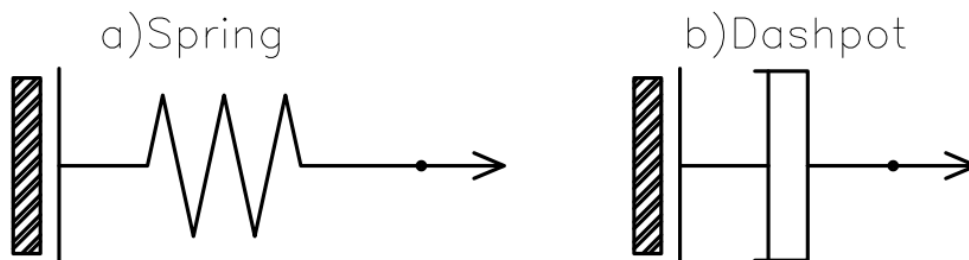

Fig. 2.2-1 Hookean spring and Newtonian dashpot

The simplest representation of spring and dashpot model is the **Maxwell** model where we can describe more for those properties and Model itself.

I. Viscoelastic Materials Mathematical Models
   This chapter consists of four parts. Maxwell, Kelvin-Voigt-Meyer, Four-Elements and Multimode models.
   i) Maxwell

   Maxwell model consists of one spring and one dashpot connected in series. The dependence of viscoelastic response can be described by identical ordinary differential equations in time.

   These models are helping visualize molecular motions employs "spring-dashpot" models. These mechanical analogy use "Hookean" springs. (Check Fig. 1 for more details)
   In this model, where spring element represents a Hookean Behaviour, and the dashpot element, a Newtonian viscous behaviour. These elements are arranged in series with the understanding that the displacements (strains) are additive, and the forces (stresses) are equal across the elements.
   $$\sigma = k\epsilon \qquad (2.2.3)$$

   where σ and ϵ are analogous to the spring force and displacement, and the spring constant k is analogous to Young's modulus E; k, therefore, has units of N/m2. The entropic uncoiling process is fluid like in nature, and can be modelled by a "Newtonian dashpot", in which the stress produces not a strain but a strain rate:

   $$\sigma = \eta\dot{\epsilon} \qquad (2.2.4)$$

   Where over dot indicates time differences. We can use the ratio of viscosity to stiffness. The unit of τ is time, and it will be seen that this ratio is a useful measure of the response time of the material's viscoelastic response.

   ii) Kelvin–Voigt–Meyer
   The picture below this text shows Kelvin–Voigt–Meyer element where strains are equal and stresses are additive. This type of materials has both elasticity and viscosity. It can be represented by a purely viscous damper and purely elastic spring connected in parallel.
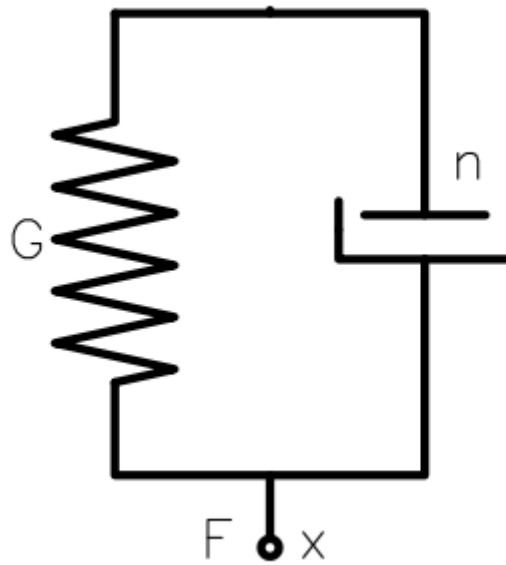
Fig2.2-2 Kelvin Voigt element

$$Gx + \eta\dot{x} = F \qquad (2.2.5)$$

If we identify x with the strain γ_{ij} and F with the stress S_{ij} , we will have the Kelvin–Voigt–Meyer model.

$$G\gamma_{ij} + \eta\gamma_{ij} = \dot{F} = S_{ij} \qquad (2.2.6)$$

It has similar relaxation time with Maxwell model which is $\lambda = \eta / G$
It is considered as a solid. That means it can resist against shear stress which causes flow for an infinite amount of time, therefore no deformation will occur.

iii) Four Elements

This part has similarities with the Kelvin-Voigt element with an extra strain and force. It combines Kelvin and Maxwell in series.
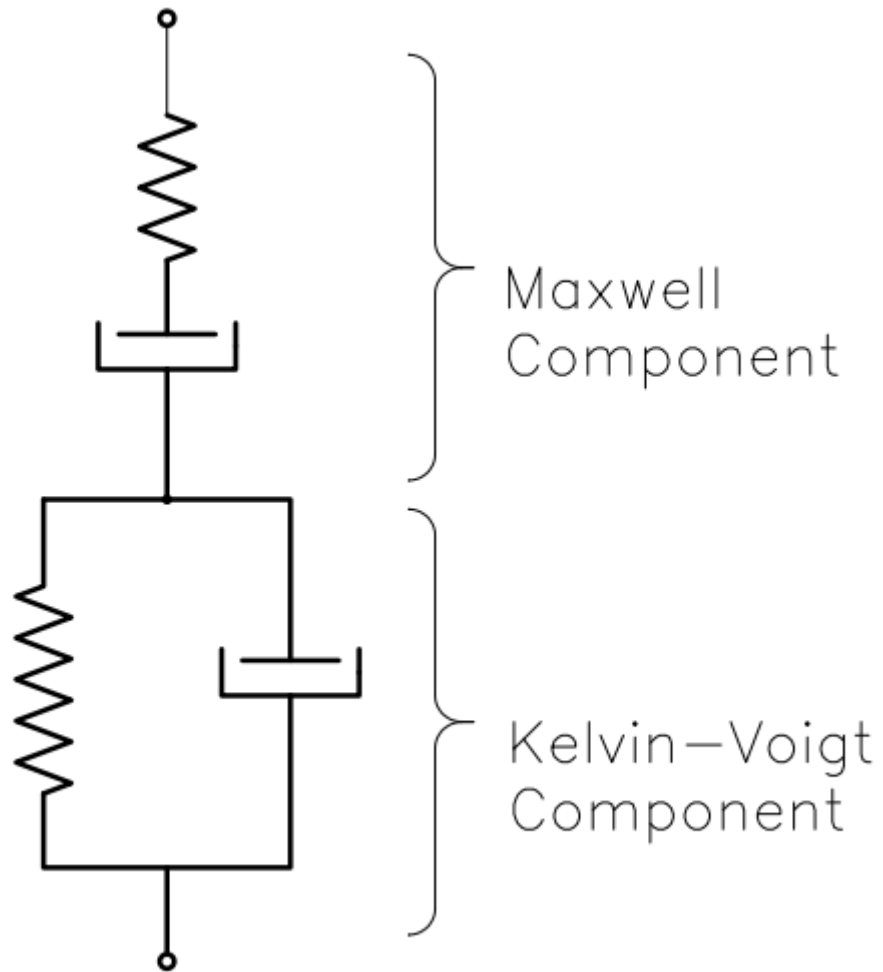
Fig2.2-3-Four Elements element

This part consists of a combination of "Hookean spring-dashpot" models. We will use spring for Hookean solid and dashpot for Newtonian viscous. Therefore for each viscous and Hookean solid we will indicate but in the component of the previous models. As we can see in the graph above it can be parallel connected spring and dashpot (Kelvin Component) and serial connected spring and dashpot (Maxwell Component) vice versa. The golden rule for that it has four elements of the combination.

iv) Multimode models
This is not a common type of model. This means the object we are working is composed more than 2 parts. In this case

$$\gamma_{ij} = \sum_{n=1}^{N} \gamma_{ij} \tag{2.2.7}$$

II. Viscoplastic Materials Mathematical Models
    This chapter is about materials which have viscosity and plasticity together. This
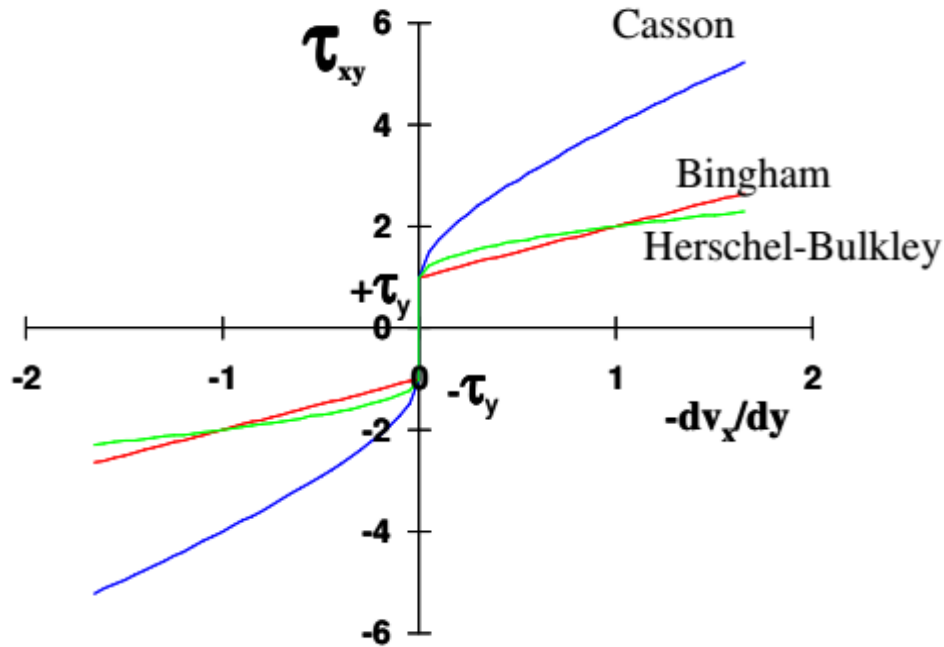   chapter consists of 3 models. Bingham plastic, Hershel-Buckley and Cassone

Fig2.2-4 Shear Stress / Rate graph for variable models[10]

Bingham Plastic Model	$\tau = \tau + \dot{\gamma}\mu$	$|\tau| > \tau y$	(2.2.8)
Herschel-Buckley model	$n\,\tau = \tau_0 + K\dot{\gamma}$	$|\tau| > \tau y$	(2.2.9)
Cassone model	$\tau = \tau_0 + \dot{\gamma}\mu$	$|\tau| > \tau y$	(2.2.10)
All Models	γ	$|\tau| \leq \tau y$	(2.2.11)

Graph indication data is listed above. As we can see the lines in the graph has an
equation for their shear stress (τ). μ is constant plastic viscosity, τ_0 is the yield
stress, K is the consistency index, and n is the power-law index. For understanding
and evaluating better results from the graph, we can use tensor calculus to
compute. So far, we are not interested in viscoplastic materials, therefore we will
not introduce it in tensors.

i) Bingham plastic
    A Bingham plastic is a viscoplastic material that behaves as a rigid body at
    low stresses but flows as a viscous fluid at high stress. It is named after
    Eugene C. Bingham who proposed its mathematical form [4]

    Due to its critical point and behaviour change, we can define that as

$$\frac{\partial u}{\partial y} = \begin{cases} 0, \tau < \tau_0 \\ \dfrac{\tau - \tau_0}{\mu_\infty}, \tau \geq \tau_0 \end{cases} \tag{2.2.12}$$

As we can see in the graph above, after it passes its yield stress, it will start to flow.

j) Herschel-Bulkley
This model was introduced by Winslow Herschel and Ronald Buckley in 1926. It has a non-linear relation between shear stress and shear rate. Shear rate is not directly proportional to shear stress. The relationship is given as

$$\tau = \tau_0 + K\dot{\gamma}^n \tag{2.2.13}$$

This T_0 is a critical point (Yield stress) and where K is the consistency index, and n is the power-law index. This is, in essence, a measure of non-Newtonian-ness. For a Newtonian fluid Power Law Index = 1; for a shear-thinning fluid it is between 0 and 1 and for a shear thickening fluid it is greater than 1. This power-law index comes from viscosity formula for Newtonian and non-Newtonian formulas.

$$\eta = K\dot{\gamma}^n \tag{2.2.14}$$

Where n is the power-law index.

k) Cassone Model
This model is developed by Cassone in 1959 for ink distribution, it is mostly used in food distribution. The materials which follow Cassone model a straight line results when the square root of shear rate, (γ), is plotted against the square root of shear stress, (σ), with slope Kc and intercept K0.

The International Office of Cocoa and Chocolate has adopted the Cassone model as the official method for interpretation of flow data on chocolates. However, it was suggested that the vane yield stress would be a more reliable measure of the yield stress of chocolate and cocoa products. The Casson model in incompressible flow can be written as

$$\tau_{ij} = \begin{cases} 2\left(\mu_B + \dfrac{\tau_0}{\sqrt{2\pi}}\right)e_{ij}, & \pi > \pi_c \\ 2\left(\mu_B + \dfrac{\tau_0}{\sqrt{2\pi_c}}\right)e_{ij}, & \pi < \pi_c \end{cases} \tag{2.2.15}$$

Where τ_0 is the known yield stress

$$\tau_0 = \frac{\mu_b\sqrt{2\pi}}{\beta} \tag{2.2.16}$$

Where μ_b is known as plastic dynamic viscosity of the non-Newtonian fluid, π is the product of the component of deformation rate with itself, where

$$\pi = e_{ij}e_{ij} \tag{2.2.17}$$

The $e_{ij}e_{ij}$ is the i, j-th component of the deformation rate.

π_c is the critical value based on the Non-Newtonian model.

During the Casson fluid flow ($\pi > \pi_c$), it will be

$$\mu = \mu_B + \frac{\tau_0}{\sqrt{2\pi}} \tag{2.2.18}$$

If we implement p_y value from Eq.2.2.16 to Eq.2.2.18, we will have

$$\vartheta = \frac{\mu_B}{\rho} + \left(1 + \frac{1}{\beta}\right) \tag{2.2.18}$$

In the final equation (Eq.2.2.18), Casson fluid is dependent on density, plastic dynamic viscosity and Casson parameters. The parameters are shown in Table.2.2.1.

Parameters	Definition
ϑ	Dynamic viscosity
μ_b	Plastic dynamic viscosity
ρ	Density
β	Casson parameter

Table2.2.1 – Casson model parameters

This fluid is assumed to have an infinite viscosity at zero shear rate and zero viscosity at an infinite rate of shear [8].

As we explained above those models which we are interested in viscoelastic models. As we can see above, we use dashpot-spring models in combination to make new models. The connection type of the models may vary. It can be serially connected, mostly in fluid and it can be parallelly connected, mostly in solids. Depends on the type of connection we may have the same deformation rate or same force on each element (Same force for serially connected and same deformation for parallelly connected elements.).

We can show force in a dashpot model: $c\frac{dx}{dt}$

And force in spring model: cx

C is indicated as constant and x is deformation, where dx/dt is deformation over time.

We are more focused on derivational based spring-dashpot elements than Integral constative equations.

If we apply Hooke's law for linear elasticity

$$\sigma_{ij} = C_{ijkl}\epsilon_{kl} \tag{2.2.19}$$

For each component of strain and stress

$$\sigma_{ij}(t) = \int_{t_0=0}^{t} C_{ijkl}(t-\tau)\frac{d\epsilon_{kl}}{d\tau}d\tau \tag{2.2.17}$$

This constitutive equation can accommodate any degree of anisotropy.

Each modulus can have a different time dependence.

For constitutive equation Young's Modulus (E), shear modulus (G) and Poisson ratio (γ) and frequency used rather than tensorial [5].

This work does not have the ambition to describe the behaviour of the substance by viscoelastic models, it only tries to find the methodology of determining the exit pressure in the capillary outlet and based on its value different from atmospheric pressure, to assess whether the substance shows viscoelastic behaviour or not.

2.2.2 Experimental Methods

All type of materials on nature has its minimum energy(rest) state. All the materials tend to stay in this form. When a force acting on a material to deform it, as we can see in the stretched spring where it will release the extra energy as soon as acting forces are eliminated. It will not occur where the elastic region has been exceeded. From this point of view, we can understand that movement from the rest state represents a storage of energy, which manifests itself as an elastic force trying to reproduce the static status quo.

Most concentrated structured liquids are shown strong viscoelastic effects at *small* deformations, and their measurement is very useful as a physical probe of the microstructure. However, at large deformations such as steady-state flow, the manifestation of viscoelastic effects—even from those systems that show large *linear* effects—can be quite different [1].

All types of liquids can have elastic behaviour within a short amount of the or high frequencies.

As we indicated in previous chapters for mathematical modelling of the liquid materials, we can simplify them with a spring and dashpot models (Where spring represents elastic behaviour and dashpot represents viscous behaviour).

As we mentioned before depends on parallel or serial connection type, they might have the same force or deformation acting on each element. In serially connected elements force will be same for each element in the node and vice versa.

If we connect them serially we will have viscoelastic fluid which is represented by the Maxwell model. If we connect them parallel we will have viscoelastic solid which is represented by the Kelvin-Voigt model.

We have a constitutive equation for viscoelastic materials, for each component of strain and stress.

$$\sigma_{ij} = \int_0^t C_{ijkl}\,(t-\tau)\frac{d\epsilon_{kl}}{d\tau}d\tau \tag{2.2.2.1}$$

This constitutive equation can accommodate any degree of anisotropy. Each element of the modulus tensor has a different time dependence.

If we indicate c as constant and x as a deformation rate and t as time;

For spring element: cx

For dashpot element: $c\frac{dx}{dt}$

In the spring elements since it obeys Hookean behaviour, the strain is proportional to the applied stress. So we can indicate it as

$$\sigma = G\gamma \tag{2.2.2.2}$$

Where G[Pa] is the elasticity modulus. Therefore, as we can understand from this formula spring element has a linear relationship between the stress-deformation rate.

We can apply the same manner to the dashpot element.

$$\sigma t = \eta\dot{\gamma} \tag{2.2.2.3}$$

Where η is the viscosity of the material and $\dot{\gamma}$ is the deformation rate which is dependent on time.

For acquiring the viscoelastic response of the material, the material will undergo some experimental procedure for monitoring resulting stress or strain output. There exist a different type of methods to examine the material. If we suddenly load the material and examine the strain output, it will be a creep test of the material. If we apply oscillatory stress to a material to examine the test method will be oscillatory. It is mostly named after the type of stress or strain applied to examine the material. In this chapter, we will examine some common types of test methods and explain how to obtain quantities from those test.

There exist a different type of tests. In the figure below there exist summaries for those tests.
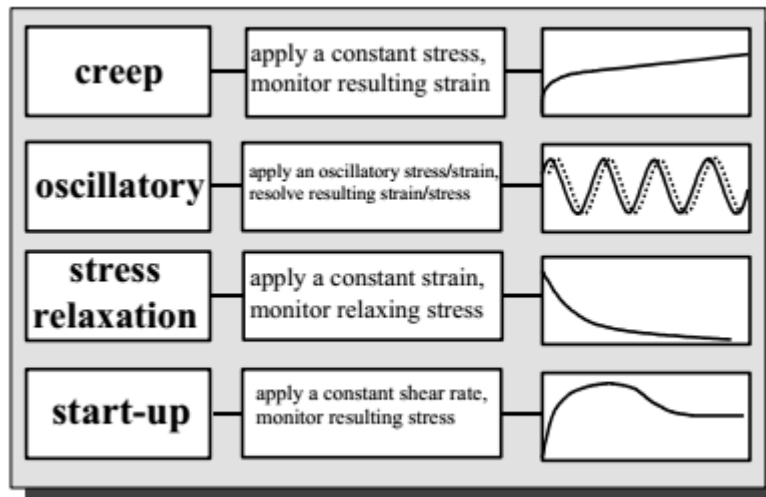
Fig2.2.2.1- Experimental methods [2]

1) Creep

Creep properties are very important in studying certain practical situations where high stresses and long times (and sometimes high temperatures) are involved, such as suspension bridges or pressurised nuclear reactor vessels.

In the creep test, uniform stress is advantageous if the investigator is interested in examining the non-linear response. In bending and torsion type creep test stress is not spatially uniformed. If tensile or compressive creep tests are to be done on relatively large specimens of a stiff material, deadweight loading becomes difficult due to the requirements of the extra weights.

We can perform creep test on the material which behaves more elastic than viscous. It is the cruel part of the material. For example, we can apply creep test to dough material but we cannot apply it to Newtonian Fluids which behaves linearly viscous like water.

In the Barnes H.A. - Handbook of elementary rheology book, it is said that "This kind of stress-controlled test was later used for softer materials such as ice cream, butter and soap, and the name creep stuck so that when the new generation of controlled-stress instruments for structured liquids came along, they had testing regimes called creep analysis."

In polymers, one must precondition specimens by applying load cycles consisting of creep and recovery segments, to achieve reproducible results in creep. Specimens of biological origin exhibit similar effects.

For a better understanding of the usage of the creep test, we can examine a material that could be suitable for Burger Model.

$$\frac{\gamma(t)}{\sigma} = \frac{1}{G_1} + \frac{1}{G_2}(1 - e^{t/\lambda}) + \frac{t}{\eta_1}$$
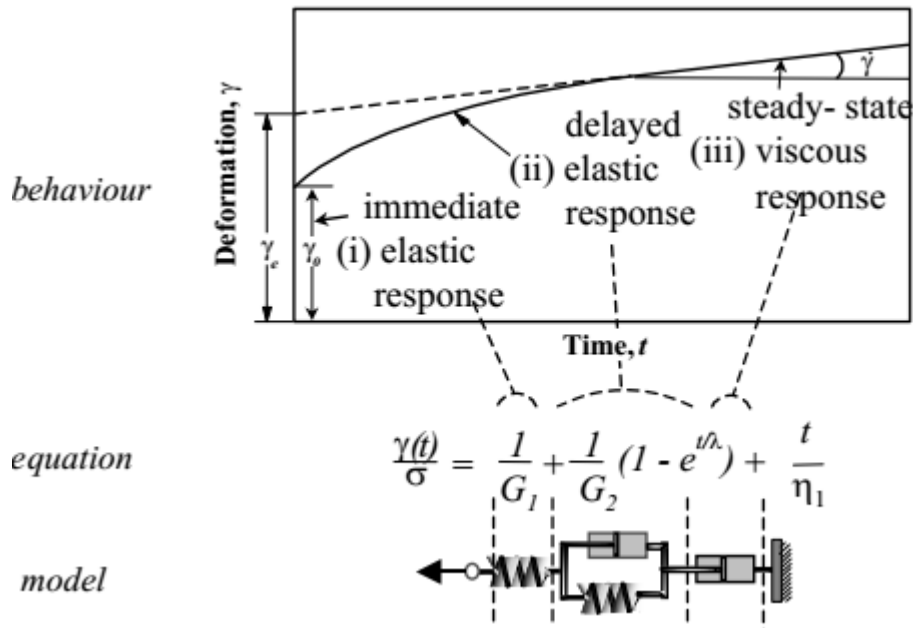
behaviour

equation

model

Fig2.2.2.2 - Creep test [2]

As we can see above we just wrote the mathematical equation by substituting Deformation to the left-hand side of the equation. We can see that the node starts with a spring element. Due to this element, we can expect to see an elastic response under the load as we can see in the materials. In the delayed elastic response where the deformation rate becomes slower and slower, ending up as a very
slow but steady-state deformation at the longest times which means the steady flow is achieved. As we can see in this part the deformation is time-dependent now. This part has a slope. When the slope of the graph becomes $1(\frac{d\gamma}{dt})$ we can say that a steady-state is achieved for the flow. As we can see in the model when the steady-state is achieved material becomes fully viscous which means it can flow as fully liquid. We can see the best way of explanation in the graph below.
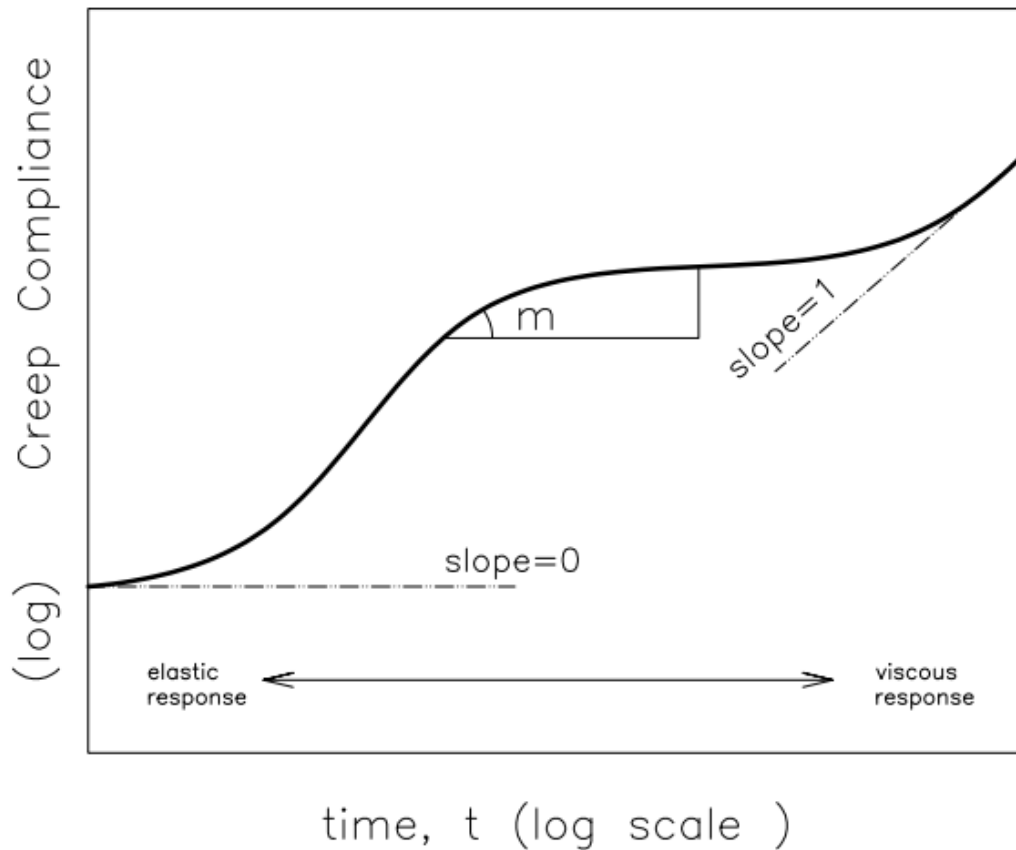
Fig2.2.2.3 - Creep time [2]

As we can see in the graph we will reach fully viscous response when the slope reaches to 1 which means that material deformation response is linear.

In the creep test, solid part(spring) has an immediate response to the strain and liquid part(dashpot) has an ever-increasing strain, which displayed on a graph of strain-time, would be a straight line starting at the origin, with the slope giving the shear rate, $\dot{\gamma}$.

Therefore, we can say when we want to write a mathematical equation for models;

Spring: $\gamma = \frac{\sigma}{G}$ which is derived from $\sigma = G\gamma$

Dashpot: $\gamma = \frac{\sigma t}{G}$ which is derived from $\sigma t = G\gamma$

We always write equations with respond of deformation. For better understanding, we can explain it inside the serial connected dashpot-spring elements (Maxwell Model).

$$\gamma = \sigma\left(\frac{1}{g} + \frac{t}{\eta}\right) \tag{2.2.2.4}$$

If we applied the same test to the Kelvin-Voigt model where elements are parallel connected, as we mentioned before deformations on each element will be same. Due to that fact

$$\gamma = \frac{\sigma}{G}[1 - e^{-\frac{t}{\tau}}] \tag{2.2.2.5}$$

So, as we can see, $\frac{\sigma}{G}$ represents elastic behaviour while If we evaluate this formula, we will get for exponential part

$$\frac{\sigma}{G} e^{-\frac{t}{\eta G}} = \frac{\sigma}{G} e^{\frac{Gt}{\eta}}$$

Where

$$t = \eta\gamma$$

Therefore

$$\frac{\sigma}{G} e^{\frac{\eta\gamma}{\eta}} = \frac{\sigma}{G} e^{\gamma}$$

We can understand that $\tau = \frac{\eta}{G}$ which is also called as <u>retardation time</u>.

This variable occurs on nonlinear elastic materials like viscoelastic materials. It is the delayed response to the stress of the material and it can be described as a delay of elasticity.

As we can see above we can connect elements serially and parallelly and write the corresponding equation to that by summing the corresponding formulas as we can see in the burger model example.

2) Oscillation

As we can perform creep test on the materials which are evaluated over time (Between a given time interval), we can perform a similar operation on the material with an oscillation over a range of frequency. The crucial part is short time corresponds to high frequencies, longer times corresponds to low frequency. The elastic region is mostly covered in a shorter time interval (Higher frequency) as well as the viscous region covers lower frequency.

For performing an oscillatory test, we input stress either strain in the form of sine-waves we will get the sinusoidal response of strain-stress output solid-like behaviour in-phase (inset) with the input and liquid-like behaviour with the $\frac{\pi}{2} = 90°$ Phase offset.

For better understanding, we can give an example of AC power. In 3-phase voltage, there will be offset of $120° = 0.66\pi$ between the currents.

Fig. 2.2.2.4 – 2 phase waves with offset

As we can see, if the blue line is the normal current orange line(reddish) has an offset of 90°. This graph indicates two sinusoidal waves between 0-100-time interval.

The output which we will obtain varies with angular velocity ω which is $2\pi f$ where f is the frequency. SI units of the outputs are in Pascal [Pa].

Solid-like behaviour characterized by Storage modulus and indicated by G'. Liquid-like behaviour characterized by Loss modulus and indicated by G''.

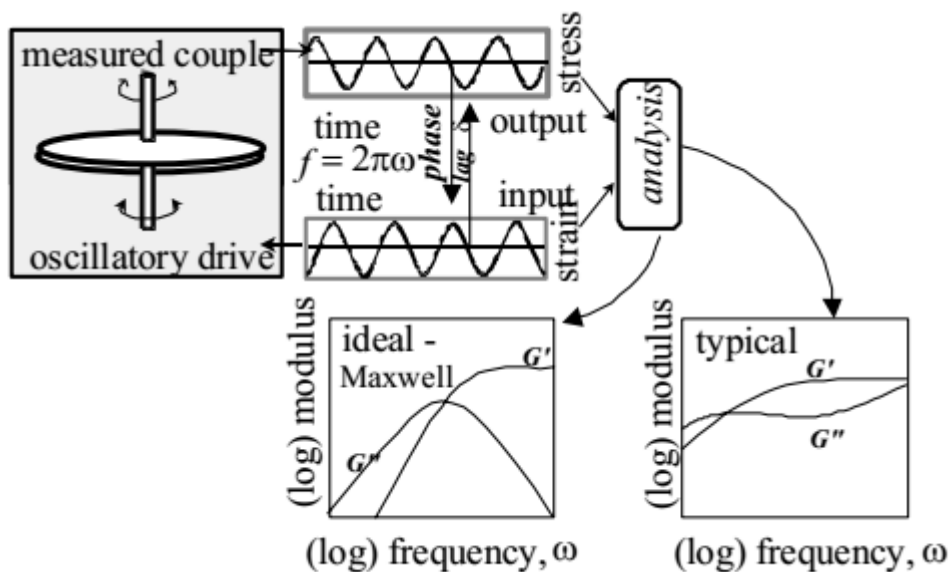Fig2.2.2.5 - Oscillation test [2]

Depends on the type of different geometry of the rheometer tools calculation may vary. We can say that rheometer is an advanced version of viscometers. We will explain briefly how to calculate variables under assumptions of

- Incompressible, Newtonian fluid, steady-state case

- One-dimensional case

- Velocity component in the x-direction depends only on the y-axis

- No pressure drop in the system (the driving force is the moving upper plate)

- There are no changes in the direction of z-coordinate

- No volume forces

Under those assumptions, if we enrol the plane into two parallel plates to get rid out of cylindrical coordinates (Cylindrical coordinates would be more beneficial), we will have flow equal to
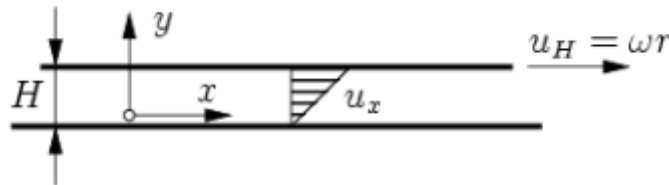
Fig. 2.2.2.6 – Velocity profile in a pipe [16]

If we create the velocity profile for each case, it will be

$$u_x = \frac{u_h}{H} y \qquad (2.2.2.6)$$

Where ω is the angular velocity which is dependent on frequency or round per minute? And the dynamic stress tensor would be

$$\tau_{xy} = \frac{\mu u_h}{H} \qquad (2.2.2.7)$$

Where the required or applied force is

$$dF = \tau dS \qquad (2.2.2.8)$$

dS is the surface area of the tool. It varies.

In the last applied torque is

$$M_t = \int_0^R dM_t = \int_0^R dF * r = \int_0^R \tau dS * r = r \int_0^R \tau dS \qquad (2.2.2.9)$$

And where to power output of the machine is

$$P_{Machine} = M_t * \omega \qquad (2.2.2.10)$$

From those simplified equations, if we know how much torque, force or power applied to the machine, we can calculate μ of the material. This formulas only satisfy under the assumptions which are indicated above. Every material has different dimensions and, there exist couple imperfections, due to these facts most of the new rheometers has their programmes to auto-calculated as well as their modules.

In some rheometers, it is explained as in the graph below

- the dynamic viscosity - $\eta' = G''/\omega$,

- the loss tangent - $\tan \delta = G''/G'$,

- the complex modulus - $G* = \sqrt{G'^2 + G''^2}$,

- the complex viscosity - $|\eta *| = \left[(\eta') + \left(\dfrac{G'}{\omega^2} \right) \right]^{\frac{1}{2}}$.

Fig2.2.2.7 - Rheological parameters [2]

Some rheometer can cause a problem due to its mechanical inertia. It can effect the high-frequency test.

Normally for ideal spring and dashpot model, their relative other model (dashpot for spring and vice versa) will be equal to zero. For in the more explanatory way:

Ideal Spring is where Storage Modulus(G') is G(Modulus) and Loss modulus(G") is equal to zero.

In Ideal Dashpot $G'' = \eta\omega$ and $G' = 0$.

When those two models are serially connected as in the Maxwell Model, mathematically their Loss and Storage modulus will be equal to

$$G' = \frac{G(\omega\tau)^2}{1 + (\omega\tau)^2} \qquad (2.2.2.11)$$

$$G'' = \frac{\eta\omega}{1 + (\omega\tau)^2} \qquad (2.2.2.12)$$

Where $\tau = \eta/G$ which stands for relaxation time.

One important part is during the very low frequencies G" (Loss modulus) is much larger than G' (Storage Modulus), therefore dashpot model behaviour(viscous) dominates. We can use Deborah number in here to understand and guess which kind of behaviour will be dominant. Deborah number is the relaxation time over test time (τ/t). In our case, it will be $D_e = \omega\tau$.

If the Deborah number is small it indicates that material is predominant on liquid-like behaviour. Otherwise, it will be more dominant on solid-like behaviour. We can make it simplified as if it requires more time to be deformed it is more close to solid.
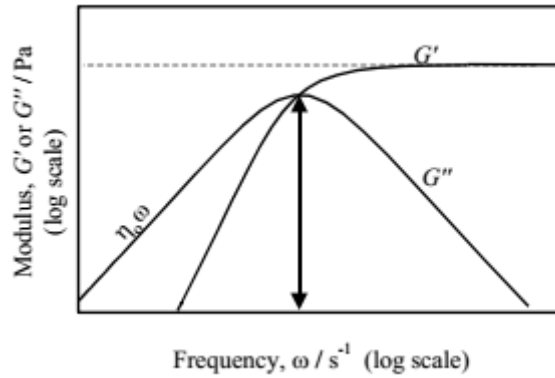
Fig2.2.2.8 - Modulus/Frequency graph [2]

As we can see in the figure above, during the oscillatory test of a Maxwell model type of material, there will be a critical cross over the frequency that $G'' = G'$ and $\omega = 1/\tau$

We can define dynamic viscosity as $\eta' = G''/\omega$. For the Maxwell model, we can write $\eta' = \eta - \tau G''$. By using this formula we can check if the data really responds to the Maxwell model or not.

In case of double or more Maxwell model introduced in, we will shortly sum up their loss and storage modules.

If an i number of elements introduced into the Maxwell model, we must follow similar manners which we did in Creep Test Burger Model. Their mathematical sum will be for each modulus is [2]

$$G'(\omega) = \sum_i G_i \frac{\omega\tau^2}{1 + \omega^2\tau^2} \tag{2.2.2.13}$$

$$G''(\omega) = \sum_i G_i \frac{\omega\tau}{1 + \omega^2\tau^2} \tag{2.2.2.14}$$

3) Relaxation

Relaxation testing can be conducted at any point in the history of a constant strain rate or creep test. Ideally, the total length of the specimen is held constant. As a result, the stored elastic strain energy in the specimen is dissipated through plastic deformation in the specimen, so that the measured value of stress supported by the specimen decays with time.[6]

$$\frac{de}{dt} = -k_s \frac{d\sigma_a}{dt} \tag{2.2.2.15}$$

The first part de/dt is the permanent strain rate, $\frac{d\sigma_a}{dt}$ is the rate of decay of applied stress. The last part k_s is the specimen compliance.

If all of the stress is relaxed, the maximum permanent strain which can be accumulated in the specimen during relaxation equals the elastic strain at the start of the relaxation. In principle, the strain rate can be estimated from the relaxation rate at a number of discrete points on the stress scale, the load being dropped manually between these short relaxation periods.

Suddenly deforming a viscoelastic liquid to a predetermined strain results in stress that, once attained, begins to fall off rapidly with time. This decrease eventually slows down and sometimes appears to approach a steady-state value at long times.
If the liquid under test corresponded to a Maxwell model, we can use a similar approach which we used in

$$\frac{\sigma(t)}{\gamma} = Ge^{-\gamma} = Ge^{-\frac{t}{\tau}} \tag{2.2.2.16}$$

As we derived before this formula comes from Parallel connected elements in a creep test. When we concern about its deformation formula was

$$\gamma = \frac{\sigma}{G}e^{\gamma} \tag{2.2.2.17}$$

$$G = \frac{\sigma}{\gamma}e^{\gamma} = \frac{G}{e^{\gamma}} = \frac{\sigma}{\gamma} \tag{2.2.2.18}$$

And as the last one

$$Ge^{-\gamma} = \frac{\sigma(t)}{\gamma} \tag{2.2.2.19}$$

For the viscoelastic liquids, the stress relaxation is described as Maxwell elements in parallel. Therefore we can just sum them together. The general formula will be

$$\frac{\sigma(t)}{\gamma} = \sum_{\hat{\imath}} G_{\hat{\imath}}e^{-\frac{t}{\gamma}} = G_1 e^{-\gamma} + G_2 e^{-\gamma} + G_3 e^{\gamma} + \cdots \tag{2.2.2.20}$$

The parameters are obtained by non-linear curve fitting programmes.

4) Slit-rheometer

The slit die has long been used to determine the viscosity of fluids. Experimentally, pressure drops are measured along the fully developed region of the die as a function of flow rate. Wall shear stress is calculated from the pressure gradient and shear rate from the volumetric flow rate.

Finally, the viscosity is determined by the ratio of shear stress to shear rate.

If we indicate wall stress as S_{zz} which is commonly described as "pressure", and exit pressure as P_{exit} then we have to make sure that the profile of S_{zz} is linear with respect to Z, to ensure that the flow is fully developed in the region where the values of S_{zz} are measured.
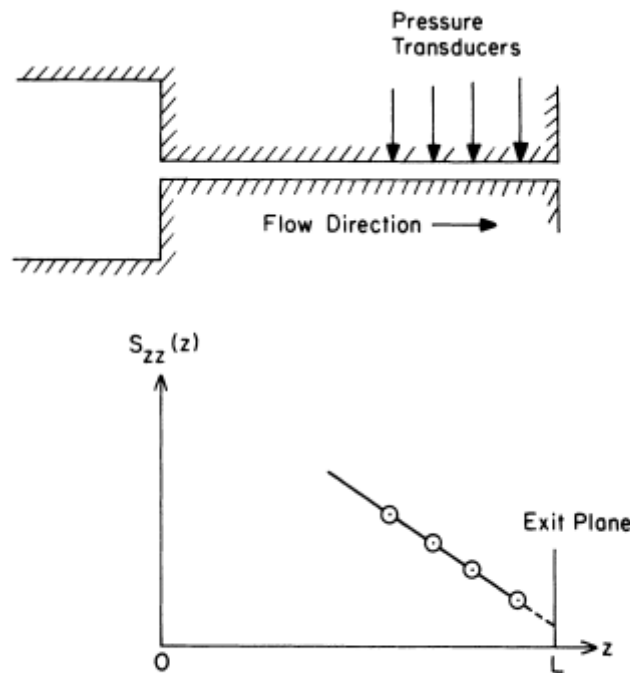
Schematic depicting wall normal stress measurements along the axis of a slit die.

Fig2.2.2.9 - Slit rheometer – Exit pressure [12]

If we look at the graph above we can understand that when the full flow is achieved, the gradient of wall pressure will be equal to the pressure gradient.

$$\nabla s_{zz} = -\nabla \rho \qquad (2.2.2.21)$$

The length of the die is important to achieve to developed flow, but it does not vary only with the type of the fluid (λ characteristic time) but also with shear rate ($\gamma^{\cdot}$).

If the die dimension is not fixed which means $\nabla p \neq const.$, therefore fluid temperature may vary along the path which will end up changing the rheology.

There exist a critical shear rate $\dot{\gamma} = \sigma_{12}$ where viscous fluid shear heating becomes significant in slit which can be estimated by solving equations of motions and heat transfer which includes viscous heat dissipation.

Secondly, one must make sure that an extrapolation of S_{zz} to the exit plane of the die can be justified for all intents and purposes. The validity of extrapolation would depend on the type of fluid [7].

We can image some similar shape in the figure Fig9 and mounted with pressure transduces along the path far away from entrance.
If the width of the rheometer tube bigger than its height than(w>h), exhibit extrude swells. This will increase P_{exit} which is triggered by shear stress or shear rate. You can see it in more details on the graph below.

Profiles of wall normal stress along the axis of a slit die for low-density polyethylene at 200 °C: ⊙ 214·8 s⁻¹; ▲ 161·2 s⁻¹.
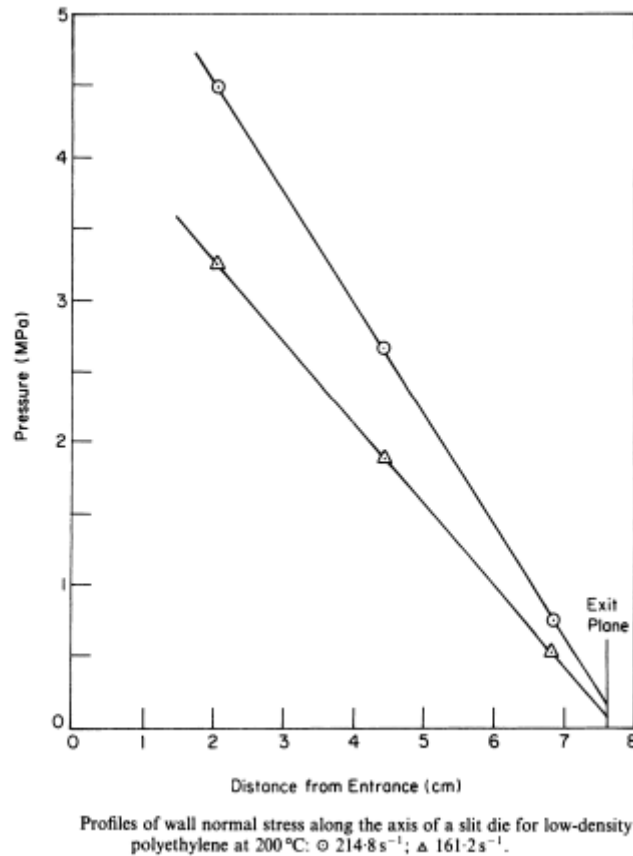
Fig2.2.2.10 - Distance of capillary exit pressure [12]

We can determine Stress at the wall (σ_w) by

$$\sigma_w = -\nabla p \frac{h}{2} = -\left(\frac{\partial p}{\partial z}\right)\frac{h}{2} \qquad (2.2.2.22)$$

We can also define shear at the wall ($\dot{\gamma}_w$)

$$\dot{\gamma} = \left(\frac{2n+1}{3n}\right)\frac{6\dot{V}}{WH^2} \qquad (2.2.2.23)$$

Where $\dot{V}$ stands for volumetric flow rate and n is the slope of the $\log(\sigma_w)$ against $\log(\frac{6Q}{WH})$.

We can always define viscosity as $\eta = \sigma/\dot{\gamma}$

According to the Han and Davis Formula derived for extra polarization process.

$$N_1 = P_{exit} = (1 + \frac{1 + d\ lnP_{exit}}{d\ ln\sigma}) \qquad (2.2.2.24)$$

Summary of chapter

The chapter summarizes the theoretical background about the measurement of flow properties of non-Newtonian fluids. Also, it provides disposable information about collagen material at literature. With respect to obtained information and previously done experimental work, the results of experiments with slit rheometer and collagen material will be analysed by my proposed script. The methodology of analysis of flow behaviour of the collagen material will involve the information from literature search mentioned above. It seems that according to the consistency of investigated material the creep, relaxation or oscillation tests are not possible to perform.

3 Experiments

The experimental bulk liquid is type I bovine collagen with high diluted where only %9.5 is the material itself. The samples stored in the refrigerator with 8°C.

The capillary is divided into two parts and they are connected to the cylinder by the flange. In the lower part of the capillary, the pressure transducers are mounted, and it has a plane surface and creates only the lower wall of the rectangular shape of the capillary. In the upper part of the capillary, the rabbet is milled which creates the rectangular cross-section of the capillary. The upper plate is easily removable which allows quick change of capillary shape. The length of the capillary is L = 200 mm. There were 3 different capillaries used with varying thickness (2, 4, 8 mm). The chosen geometry for all capillaries is rectangular. All capillaries are fixed with steel frame. More details can be seen at Fig.14

During the experiment used pressure transducers are XPM4 (TE Connectivity) where 5 of them installed inside the capillary and 1 of them installed into the storage tank. Flow rate and movement of the piston is recorded by potentiometric sensor WPS-500 (Micro-Epsilon). Every sensor in the measurement are connected into the universal data acquisition system QuantumX MX840B (HBM Manufacturer). The used software for data acquisition system is LABVIEW. The Pressure transducers records data in Bar value.
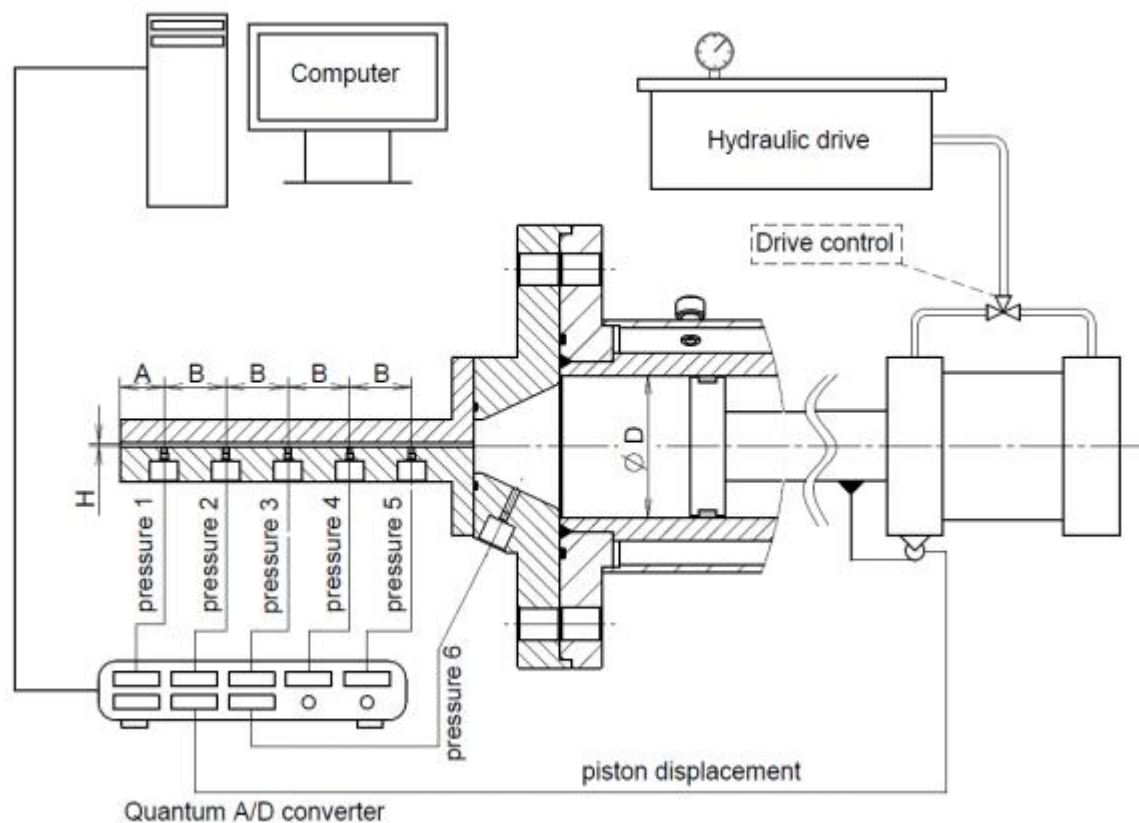

Fig.3.1 – Schema of capillary extrusion rheometer [17]
A = 25 B = 35 D = 80 L = 200 All units are in [mm]

As we can see in the figure above, capillary itself divided into two parts where the left-hand side contains pressure transducers from 1-5. The rabbet is milled in order to create a rectangular slit. It is easily removable such system can be seen on flange connection where

after dissembling the bolts, the slit part can be replaced with another geometry of slit which gives access to multiple measurement mobility.

The sample bulk liquid is inserted from the right-hand side of the capillary which corresponds to the rear part of the cylinder. The piston is retracted after each measurement in order to create space for filling and experiment on the next sample. The measurement is stopped when the piston reaches the dead end. The extruded material is not re-used after the measurement.

Steps of data evaluation are:

1) Data recording

   Data is saved into the text file with timestep with 0.01 Second. There are 9 columns exist in the text file. Columns are actual time, transducer pressures in bars, piston displacement and the current time in seconds.

2) Data filtering and treatment

   Since data has noise, it has to be filtered. Also, unit transformation has to be performed.

3) Calculation of reogram quantities from data

   Demanded quantities are evaluated from raw data. According to mentioned equations.

4) Build the reogram

   Calculated data is used for reogram. Reogram is generated and plotted to check the trend.

5) Perform regression of reogram by a mathematical model

   Application of the rheological models applied in this chapter by using regression analysis by the method of least squares deviations.

6) Identifying parameters of the model

   Using own script to identify the parameters of the model which is the best to generated reogram.

3.1 Results from experiment

We obtained mostly pressures at each transducer. The given pressures are in ATM value. We, later on, converted them in Pa value. We also obtained Time and Displacement at each instance. The software output was in "(empty)" file format where it does not bear any file format extension. The file itself, however, contains only numbers given at each row. Each row represents the instances. There are no descriptions where initialized in the columns therefore we had to follow prescribed instruction to understand which column belongs to which value. Example of raw data can be found as

Time	Pressure [Bar]						Displacement	Time
[s]	1	2	3	4	5	6	[mm]	[s]
3813.477	-0.00163	0.0005	0.00148	-0.00142	0.00102	-0.00184	0.33016	**3813.477**
3813.487	-0.00361	0.00056	0.00116	-0.00123	0.00129	-0.00176	0.33024	**3813.487**
3813.497	-0.00185	0.0004	0.00147	-0.00117	0.00148	-0.00171	0.3303	**3813.497**
3813.507	-0.0038	0.00026	0.00192	-0.00125	0.00157	-0.00167	0.33026	**3813.507**
3813.517	-0.00177	0.00019	0.00175	-0.00131	0.00151	-0.00177	0.33019	**3813.517**

Table.3.1.1 – Raw data

This table is a re-arranged form of what is given in the file. It is only important for us where our data treatment code written in MATLAB does not require their name but only their column. As an example, in order to obtain time data, we can use "Matrix-name (:1)" which will obtain the whole first column. The given data contains a specific name in order to describe which capillary and which experiment it belongs. The main order is "kap_*thickness*_pokus#Number" where thickness represents either three different capillary thickness such as 2,4,8 and #Number represents experiment number. As an example, one of the file names which we treated is "kap_2_pokus1". Firstly, we adjust data by converting them into Pascal values for the pressure. The conversion is 1 Bar = 100000 Pa.

Time[s]	Pressure[Pa]						Displacement[mm]	Time[s]
	1	2	3	4	5	6		
3813.477	-163	50	148	-142	102	-184	0.33016	**3813.477**
3813.487	-361	56	116	-123	129	-176	0.33024	**3813.487**
3813.497	-185	40	147	-117	148	-171	0.3303	**3813.497**
3813.507	-380	26	192	-125	157	-167	0.33026	**3813.507**
3813.517	-177	19	175	-131	151	-177	0.33019	**3813.517**

Table.3.1.2 – Adjusted raw data with pascal pressure values

Before starting to the treatment of the data, we display the pressure against time graph in order to find a suitable range for the treatment.
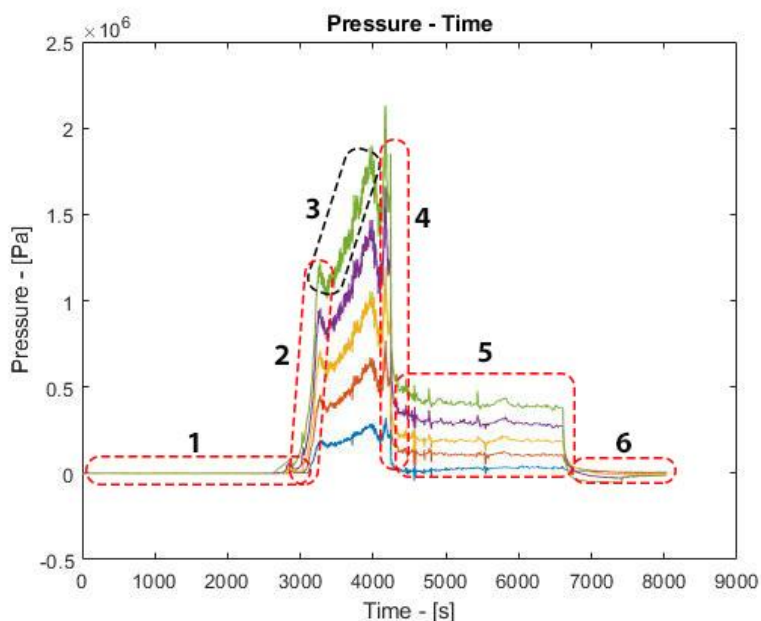

Fig.3.1.1 – Pressure against time

Recorded Pressure Data over the course period of time is shown

Indicated Steps from the Graph is

1- Recording starts

2- Filling capillary with collagen

3- Measuring part, Collagen flows through capillary piston moves to the dead-end of the cylinder

4- End of the experiment, piston reached the dead-end

5- Piston remains on its position at the dead-end, but collagen leakages from capillary

6- Movement of piston backwards to start position, recording of empty data before stopping record

The moment when the end-switch stops the piston was recorded by a displacement transducer and is also clearly visible from the recorded pressures. A steep fall, and later on a gradual fall.

These data are selected with a specific range of increment and after it, it is treated with Savitzky-Golay in order to lower the impurities. Further data evaluation is made in Chapter 4 – Analysis of Data.

4. Analysis of data

In the experiment, we use Slit rheometer to get **pressure**, **displacement and time** data from the experimental data. We used type II collagen material extract from bovine skin. Extracted data example is shown in Table3-1. For the sake of simplicity and saving the space, shown data table from below limited up to 5 rows. Normally data may contain 8 000 to 12 000 rows. We use Capillary 2 data trial 1 ("kap_2_pokus1") data for the demonstration used in the examples (Check Table.1).

We have different pressure data which is extracted from 6 different pressure transducers located on different positions of the Slit rheometer. We used only 1-5 of the pressure transducers because 6th transducer is placed in the container and its values were not used for evaluation in this work.
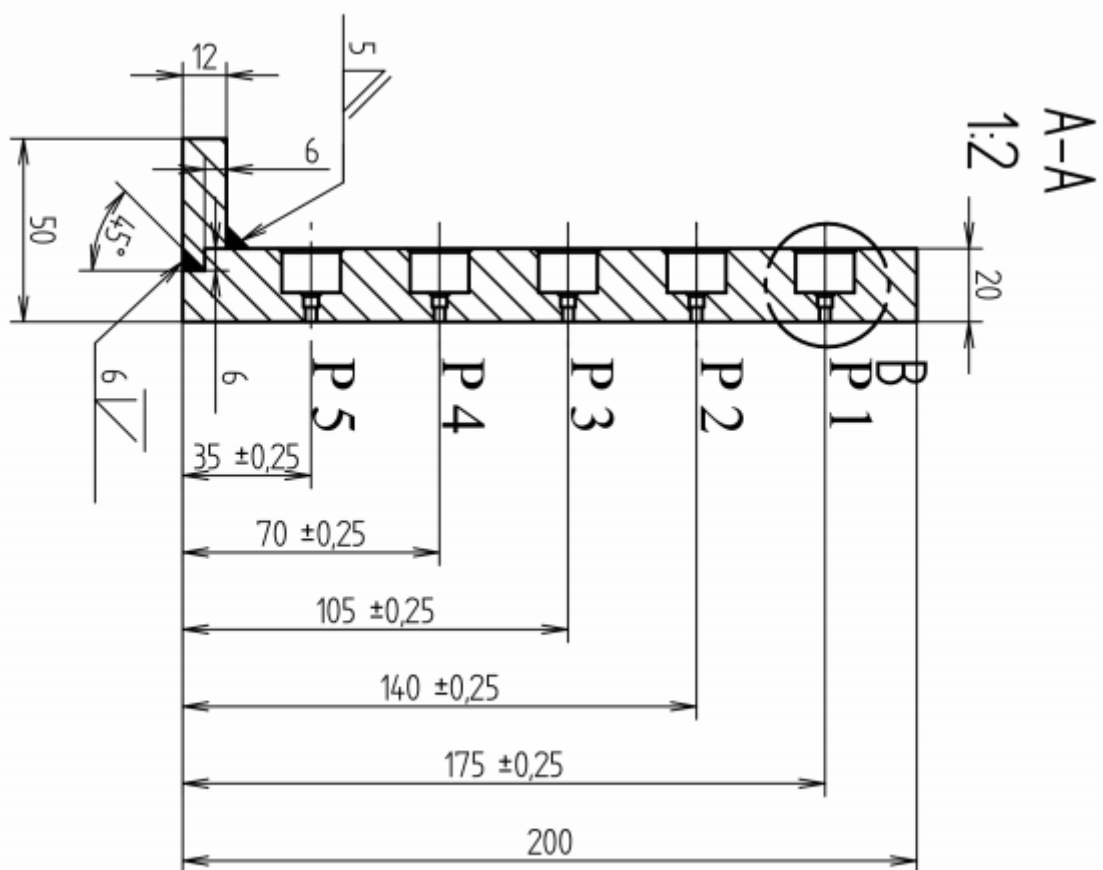
Fig.4.1 - Capillary pressure transducers

As we can see from the geometry of the Slit rheometer, it has 6 different pressure transducers located at different distances. Except for the P_1 and P_2 transducers, all the transducers have 35 mm difference to each other. We will later use these pressure values (first and the last transducer in capillary only) data in order to evaluate wall stress and exit pressure relation (P_{exit}) which was introduced in Slit rheometer.

We firstly used those pressure data and time data to plot against each other in order to find a suitable margin for the experiment. For a better explanation, we will use also data for the Capillary 2 Trial 1 with the full scope (Check Fig.15).) All presented figure with experimental and evaluated data are related to one original experiment Capillary 2 Trial 1, to show the methodology of data treatment. This methodology was applied to all other experiments.

The data presented in the figure represents the whole time of measurement. But only the part of the recorded data corresponds to flow of the collagen through the slit. In this case, the evaluated interval of the data is between 3366 to 4164 s.

As we can see above, there is only some suitable time scope which we can evaluate. This is due to the reason of capillary needs to filled with material fully first in order to get the logical rheological data. We can see these phenomena in the graph below
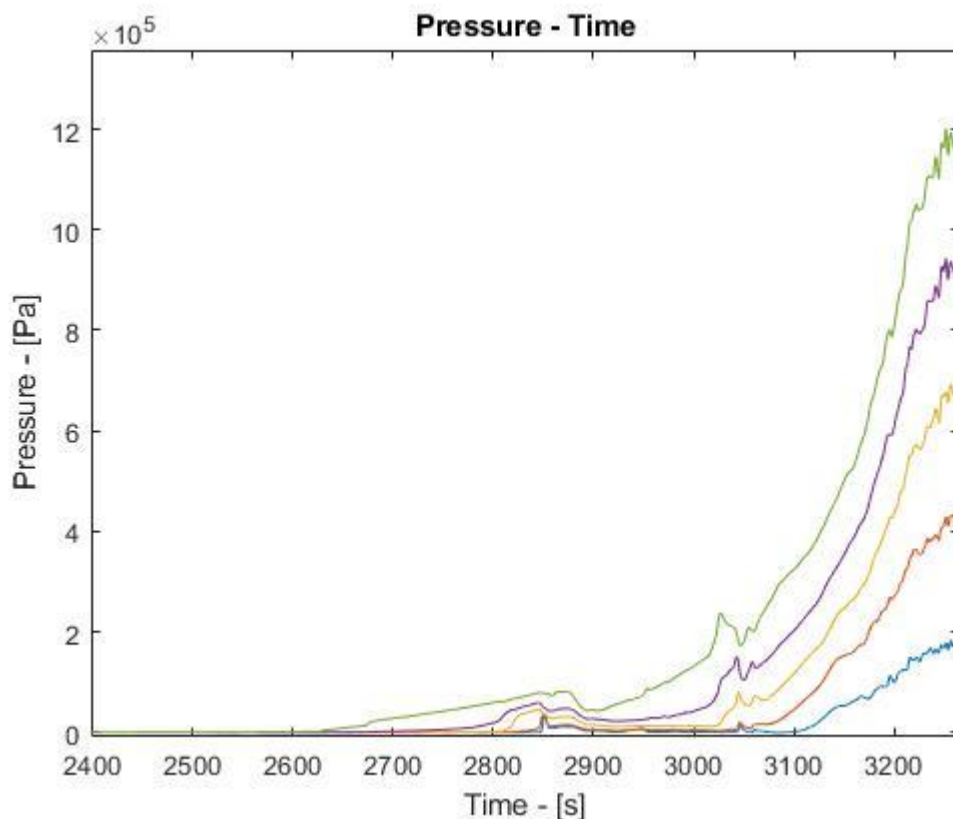
Fig.4.2 – Left side of the pressure time graph
It indices the part where capillary just started to be filled

As we can see up to 2060 approximately, there is no material present in the capillary therefore no pressure is recorded. We can see that when the material starts to approach the pressure transducers during the transient state (the filling operation is not fully completed), there will be a jump in the graph.

Since we are interested in less as possible we should consider evaluating data in the fully transient where the flow is continuous in the capillary. Example of the graph shown below.

Fig.4.3 – Pressure time after capillary is filled

As we can see above, we skip the big jump in the graph where pressure differences make more sense than before where it gradually increases with the piston displacement. We did this approach by filtering the data by taking only the steady-state region in the graph. Recorded data differ from each other because of manual control of piston movement. The benefit of the manual control movement of piston with gradually increasing velocity of p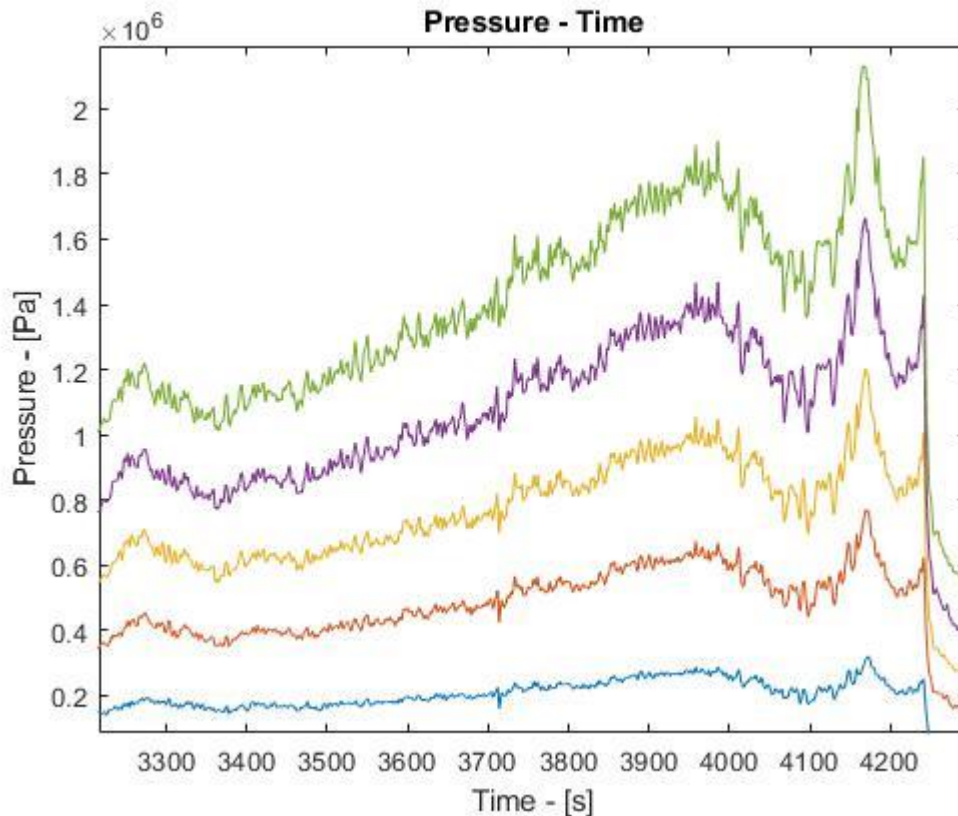iston is recording the whole reogram in one experiment (data is recorded for a range of flow rate respectively range of shear rate).

In order to find ΔP we need to find pressure differences extracted from the pressure transducers. We used Pressure differences in the developing region where it is bounded by Pressure 1 and Pressure 5. Since 6th Pressure transducer is not in the capillary, it is not suitable to use it. We used Pressure 1 and Pressure 5 values in order to get ΔP.

What we used further is instead of using timescale with small numbers such as 3.81348734 with an increment of 0.02 we implement a method that takes each time instances as a step, therefore instead of total second lapsation, we used steps and it helps us to extract and evaluate data easily. This method is only used when selecting a specific range of data where it is easier than finding specific time value to search. Program sometimes can round the values after five decimals from the comma which results in a wrong selection of the range. By this method, we are able to select the exact value at the given exact time by initializing each corresponding pressure by steps. This method is only used in this part, after selecting the range for the suitable data we can go back to time scale (Which will have also the same selected range as other values). An example is shown at Fig.4.4.

With this method, we get the pressure differences for each time step of the experiment.
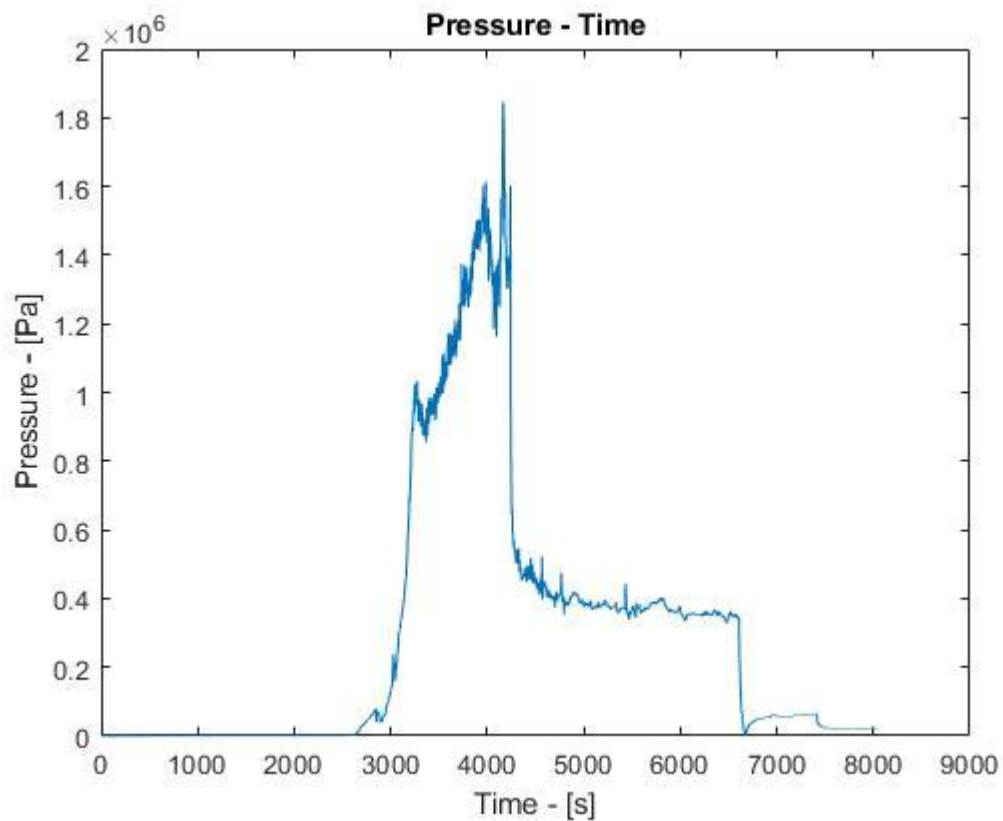
Fig.4.4 – Pressure difference against time

As we can see above, from this pressure difference we limited its range which covers only measuring state (Capillary is fully filled with the material) we decided before. This range limitation will leave us with this graph. (from 3366 steps to 4164 steps are used in this example). We separated data for treatment from all recorded data.
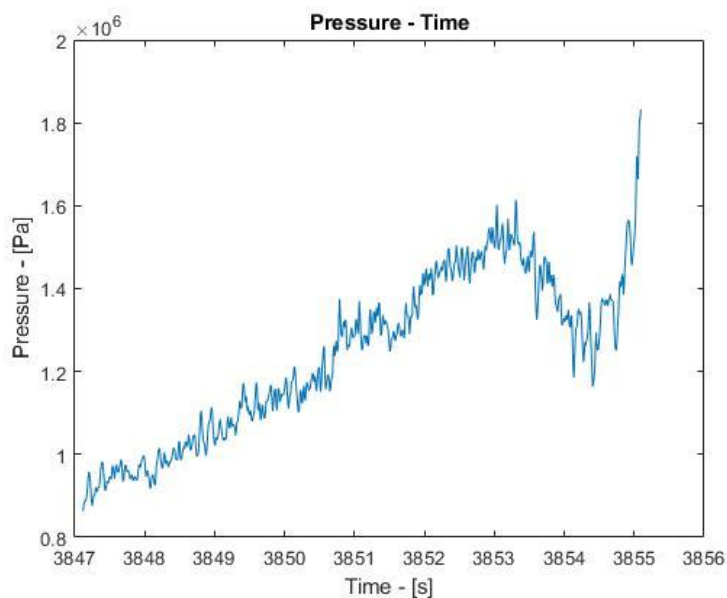
Fig.4.5 – Selected range of pressure difference plot

As we can see on the Fig.4.5, desired selection range is applied and data can be processed further from this step. We can go back to time scale again after deciding which steps should be considered too worth to evaluate. Instantaneously we applied the same steps for the displacement value.
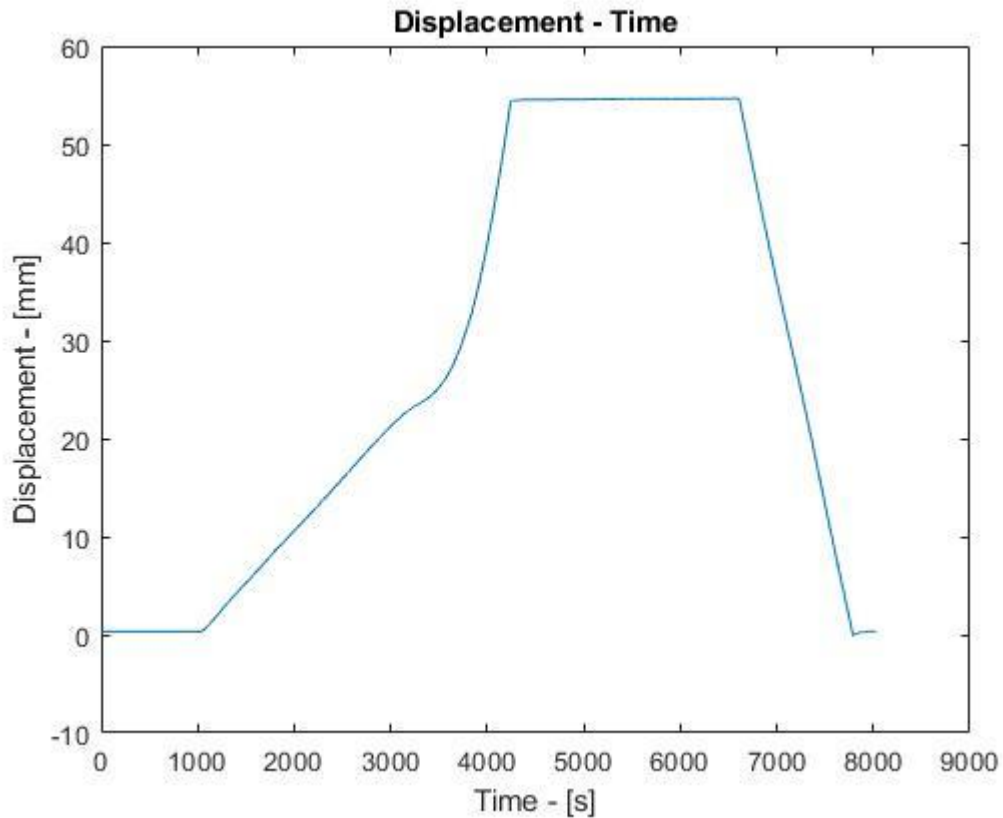
Fig.4.6 – Displacement time plot before evaluation
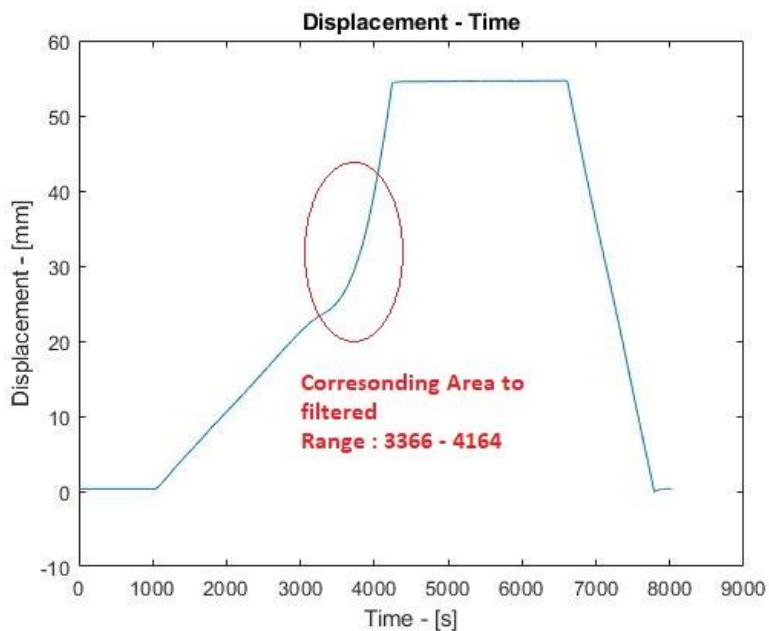
Fig.4.7 – Indication of selected range for displacement time graph
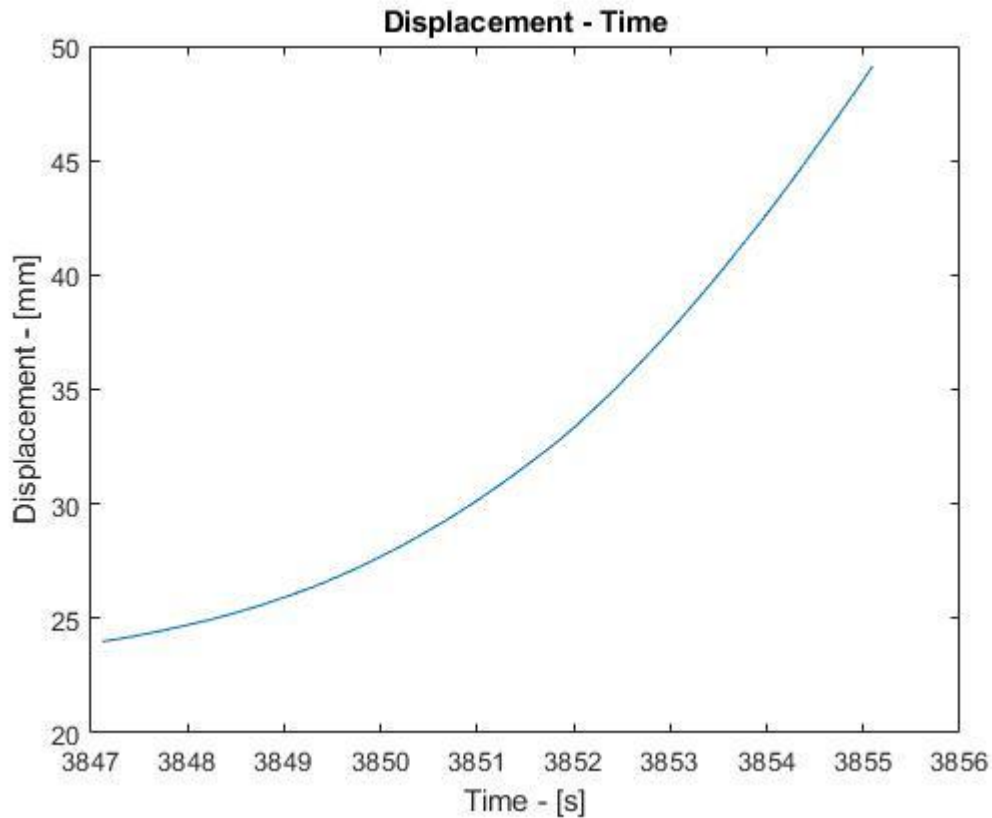
Fig.4.8 – Result of range filtering for displacement time plot

In fig. 4.6 the record of whole displacement trend is presented, with described regions related to experiment parts. Then at Fig.4.7 selected range corresponds to area which is shown in red circle. Fig.4.8 is close-up view of the selected region.

For better explanation of this process, time values stored in an Array where each instances correspond to indices. We used this indices to find limit range then we can just apply this range limitation by using same indices.

Fig.4.9 has wavily characteristic. We used Savitsky – Golay(SG) filtration method in order to filter the data.

We implemented SG filter of 2nd order with the frame length of closest odd number to data limit range. By simpler meaning it is the data limit range in case of odd numbers and data limit range -1 for the even numbers in order to cover each part of the curve. For this experiment chosen frame length is 555. The obtained Pressure-Time and Velocity-Time are given at Fig.4.9 and Fig.4.10.
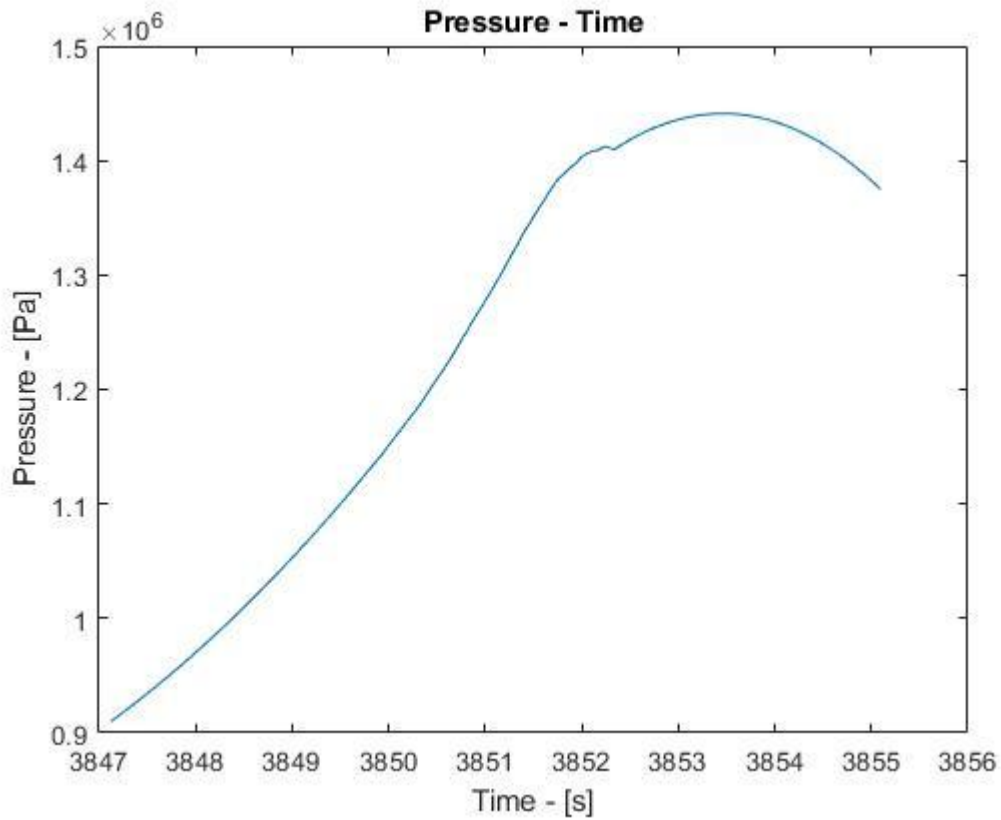
Fig.4.9 – Pressure Time after SG filtration
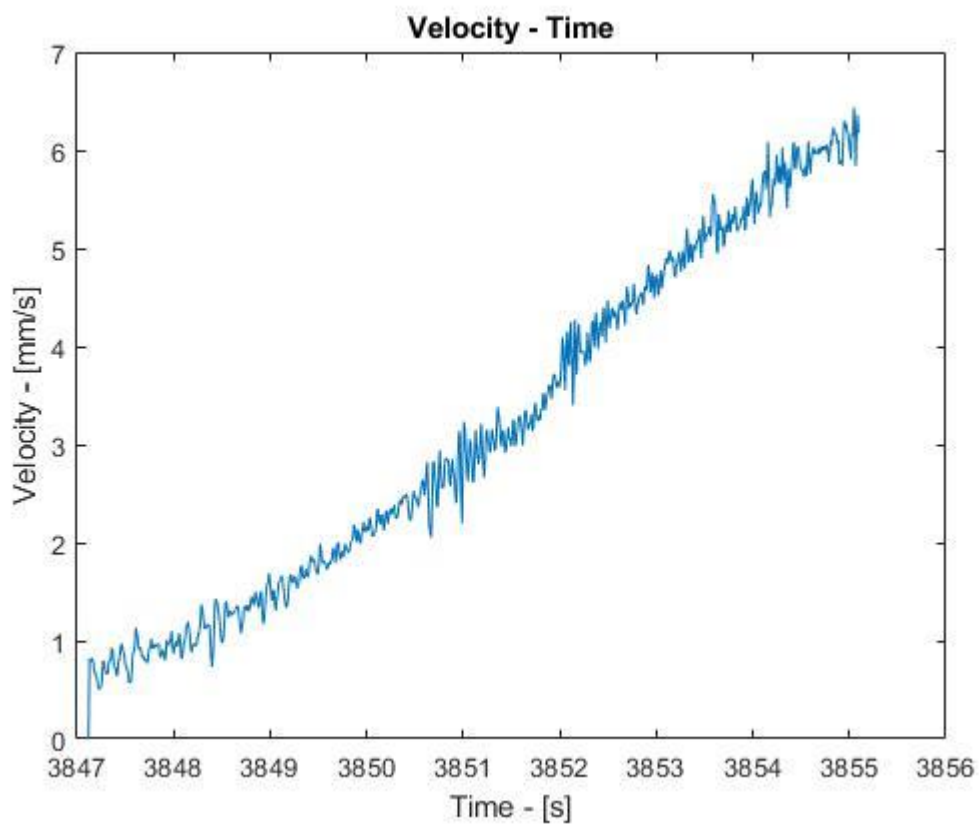
Fig.4.10 – Velocity Time before SG filtration

We also need to get velocity in order to find Volume flow rate which will be used in shear rate.

Since we know what is the position of the piston on given time, therefore, instances, we used basic equation for the velocity which is $v = \frac{dx}{dt}$ which translate into different in position according to the time in the numerical method which we implemented. It is the derivation of position to the time in other meaning. Therefore we can derivate the displacement in order to find the velocity.
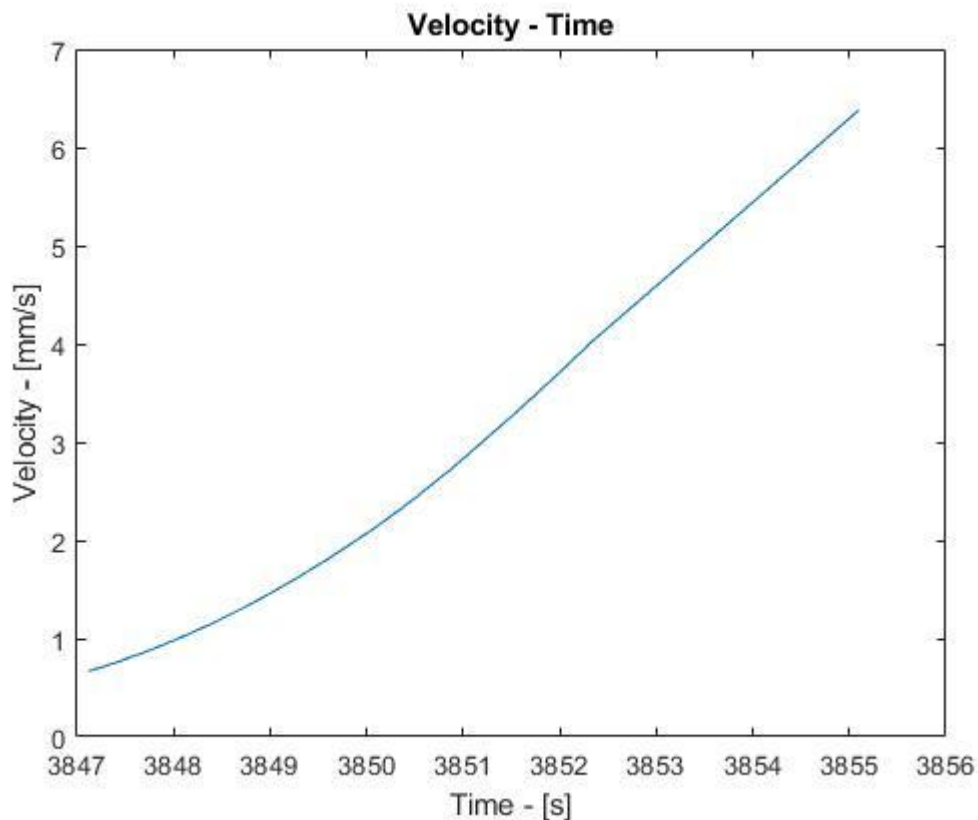
Fig.4.11 – Velocity Of The Piston

Since we have every data in order to calculate the necessary element to plot Shear Stress(Tau) against Shear Rate(Gamma), we can just evaluate the result by those.

Volumetric Flow rate = cross-section of the channel x velocity where geometrical parameters are ($D_{piston} = 80mm, S_{piston} = 0.2\ m^2$)

Since Piston has 80 mm with cylindrical diameter, the surface calculation will be the same with circle surface are

$$S_{piston} = \pi * D_{piston}^2 \tag{4.1}$$

$$\dot{V} = S_{piston} * \overrightarrow{v_{piston}} \tag{4.2}$$

Where geometrical parameters are from pressure transducers and capillaries. The length between pressure transducer one and five is constant which is equals to 150 [mm]. Therefore shear stress is

$$\tau = \frac{F}{A} = \frac{\Delta P * H}{2 * Length} \tag{4.3}$$

And shear rate is

$$\dot{\gamma} = \frac{dv_x}{dy} = \frac{V}{h} = \frac{6 * \dot{V}}{BH^2} \tag{4.4}$$

This formula is Newtonian version of slit rheometer where n = 1. Please check Eq.2.2.2.23.

After it we found parameters, we used Power Law model and Hershel Bulkley Model to apply Curve Fit to those values.

```
[f,v]= fit(gamma,tau,'a*x^b+c'); % Hershel Bulkley Fluid
[g,n]= fit(gamma,tau,'a*x^b'); % Power Law Model
```

We used MATLAB fit tool in order to find suitable fit tools.

As can be seen from the code above, we used "Curve Fit" tool from MATLAB library in order to find a suitable two different equation of the curve in the form of

Power Law previously used in Eq.2.2.14. The Herschel Bulkley model was given in Eq.2.2.13.

We evaluated data for capillaries with 2, 4 and 8 mm thickness. We had a total of 40 different measurements for all capillaries. Due to some errors, only some of them are suitable for the evaluation. We also see that capillary 8 data was not suitable due to the high errors. We can see those problems also in the evaluated data. You can see the data in Appendix B and Appendix C.

It is proven that at the end of capillary was not filled with collagen material. Due to this reason, there is an under pressure compared with the Atmospheric pressure. This experiment fails due to this reason. See Fig.4.9.

Fig4-15 Under pressure Capillary 8

Capillary with 8 mm is not suitable for this kind of material with this methodological approach.

We also had problems when evaluating for Hershel Bulkley model. In all data, we encountered with negative τ_0 values. As an example, please check **Appendix C**

4.1 Result of Power Law

According to our result which we evaluated, we can say that the Power-law model is one of the best suitable model for this experiment. As we described before power-law consist of an approximation curve which is based on 2 constants. By mathematical definition, we can say that there are flow consistency and flow behavior index for the power law. For remembering it again our formula was

$$\tau = K\gamma^n$$

K – Consistency Index
n – Flow behaviour Index

As seen above, we used MATLAB code in order to find approximation by power law. This approximation can make us understand the viscosity of the fluid. We can use these data in case of transformation of the bulk liquid. The data for capillary 2,4 and 8 has been treated in

the same way. Parameters of rheological models for these capillaries are summarized in tables presented in **Appendix B.**

H	K	Confid. Int.	n	Confid. Int.	$\dot{\gamma}_{min}$	$\dot{\gamma}_{max}$
[mm]	[Pa.s^n]	[Pa.s^n]	[-]	[-]	[s^{-1}]	[s^{-1}]
8	625	± 381	0.3	± 0.23	1916	14577
4	3328	± 133	0.18	± 0.013	234	629
2	1148	± 561	0.3	± 0.08	314	1845

Confid. Int. - confidential interval

As we can clearly see above, a capillary with 8 mm thickness (Capillary 8) has a wide confidential interval of identified parameters, so the experimental data are wrong.

It is since, at the end of the capillary, the cross-section of the slit was not fully filled with collagen material. Due to this reason, there is an under pressure compared with the Atmospheric pressure. This experiment fails due to this reason.
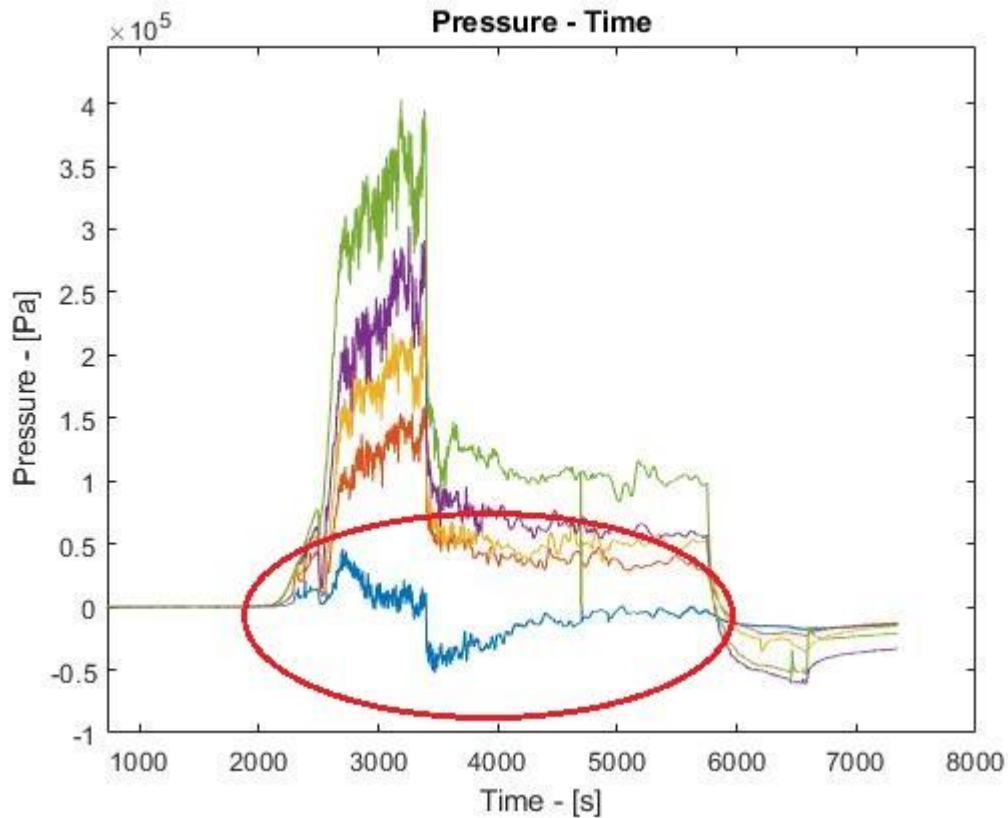
Capillary with 8 mm is not suitable for this kind of material with this methodological approach. Therefore we will not evaluate further for Capillary 8.

In addition, results from capillary 4 are not very promising. There is no such agreement between them and already published data for the same material. It is possible, that also these data or some of the recorded files are wrong.

4.2 Result of Hershel-Bulkey Law

According to our comparisons, most of the data for HB fails miserably due to the negative pressure evaluation. For instance, look at the HB fit model we apply

$$\tau = K * \gamma^n + \tau_0$$

Where τ_0 is yield stress of the material. Yield stress is the position where material just passes the elastic region where it moves to the plastic region. In those regions, deformation is permanent which is crucial for a liquid material to flow. As we know that liquid material cannot bear any shear stress and as soon as we pass the elastic region, shear will start to act on the material to deform which causes the flow itself.

In these experimental data, our formula used to fit in miserably fails due to the negative results in Yield Stress. This value is not possible for a material. If this result was real, it will result that any material at the initial state is already deformed and it is leading to mechanical failure. Due to these reasons, we will just show the data and not any logical evaluation can be made out of this. It is clear that this method fails for this data and it is not suitable for their description.

Confid. Int. – Confidential Index obtained by the standard deviation.

H	K	Confid. Int.	n	Confid. Int.	τ_y	Confid. Int.	$\dot\gamma_{min}$	$\dot\gamma_{max}$
[mm]	[Pa.s^n]	[Pa.s^n]	[-]	[-]	[Pa]	[Pa]	[s^{-1}]	[s^{-1}]
8	-	-	-	-	-	-	-	-
4	6980	±8608	1.07	±1.13	-2379	±12285	234	629
2	9151	±20048	0.6	±0.5	-8557	±26372	314	1845

Capillary 8

For capillary 8 due to the experimental error occurred which is explained in (45) and absurd fitting values it is not shown for the sake of the reader and saving space. Capillary 8 data is only proved that it is completely useless and waste to work on capillary with 8 mm thickness for this specific material with this set-up.

As we can see all above the values, even without the Yield stress(τ_0), the values are extremely high for the both K(Consistency Index) and n(Flow Behavior Index). It is not only the values themselves but Standard deviations are sometimes as high as average values. We can conclude that the HB model is completely unnecessary for this set-up and material.

Furthermore, data for the given capillary settings are presented in **Appendix C**.

4.3 Exit Pressure

Exit pressure evaluation is used for slit rheometer via statistical approximation methods. The method is to find pressure given at each transducer against the position.

It is expected that, if the material behaves according to Newton law of viscosity, exit pressure will be equal approximately to atmospheric pressure. If the material is viscoelastic, the exit pressure should be greater than atmospheric pressure, according to literature.
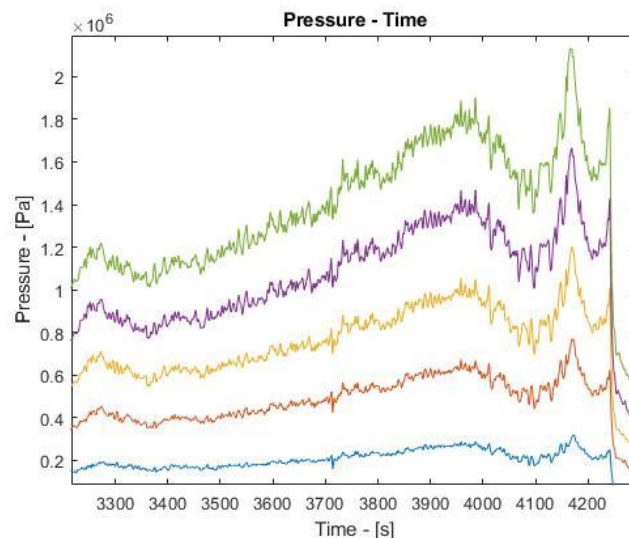
Fig.4.3.1 – Pressure Trend

The picture above indicates the pressure values obtained from the transducers where it gets a certain region. The region itself is the place where pressure is present. We used automated MATLAB script in order to divide the region into 5 equally distanced places. On the region borders, we calculated the average value from a specific point and 100 neighborhood points. Those initials are selected by 50 steps from right and 50 steps from left by the region border. The region, in fact, represents a different shear rate of measurement. Shear rate is increasing as well as the pressure values for data process treatment from left to right on the x (time) axis.
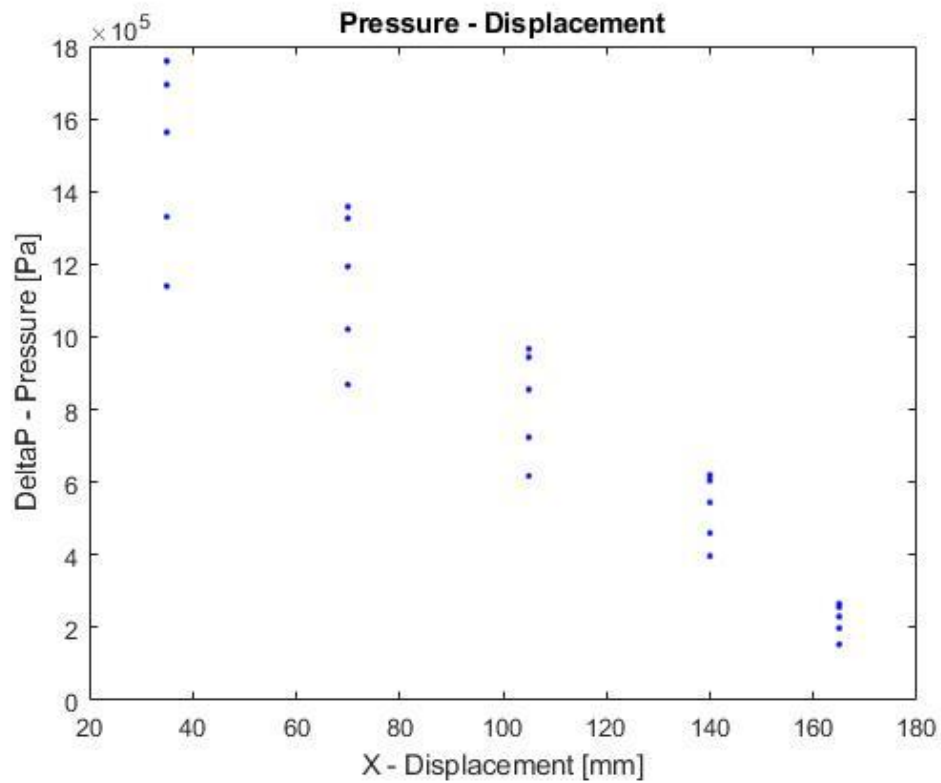
Fig.4.3.2 – 5 Pressure Values at 5 regions

We divide the range by 5 and find this pressure displacement graph. We use statistical analysis to find a suitable polynomial equation for those points. Each group of points in figure 4.3.2 represents pressure values for the specific transducer (x-axis). The increasing value of the pressure (y-axisis) in each group corresponds to the increasing value of the shear rate.

The equation is

$$y = ax + b \qquad (4.3.1)$$
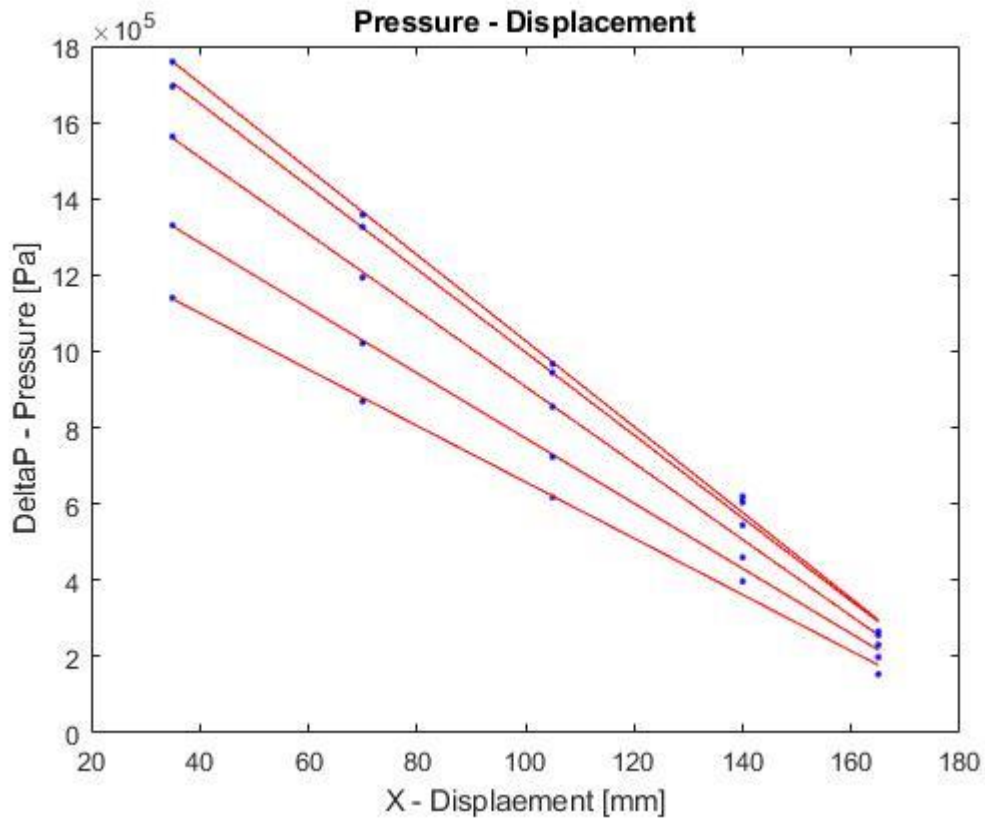
Fig.4.3.3 – Approximated Lines for Pressure regions

Then we obtain a statistical approximation of pressure trend along the capillary for each shear rate (five values) We evaluate pressure at 200 mm – which represents the end of capillary – exit pressure.
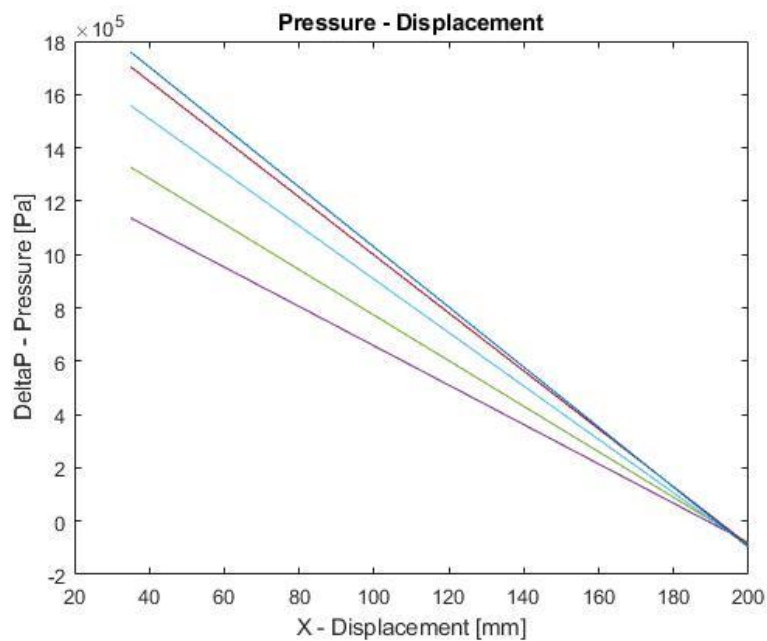
Fig.4.3.4 – Approximated Value at Exit Pressure

Briefly, we have for each experiment 5 polynomial functions which are in the form of $ax + b$. 200 value is used in x value in order to find approximation at exit pressure value. Since as clearly seen above each <u>experiment</u> has 5 of those equations, we will have a value of 5 different a and 5 different b therefore 5 different P_{exit} for a <u>single experiment.</u> Our result table length is depended on how many experiments are done for the given capillary. As it is explained before in Chapter 4.1, we are not going to apply this method on Capillary 8.

Capillary 2

H = 2 mm	$p_{exit,1}$	$p_{exit,2}$	$p_{exit,3}$	$p_{exit,4}$	$p_{exit,5}$
File name	[kPa]	[kPa]	[kPa]	[kPa]	[kPa]
FITvals_kap_2_pokus1.dat	-81.4	-81.6	-86.3	-89.3	-102.0
FITvals_kap_2_pokus10.dat	-124.3	-143.1	-165.2	-130.1	-163.4
FITvals_kap_2_pokus12.dat	-119.7	-133.6	-131.9	-152.8	-147.7
FITvals_kap_2_pokus13.dat	-117.6	-150.8	-166.0	-139.6	-153.4
FITvals_kap_2_pokus14.dat	-152.9	-145.4	-111.4	-120.1	-125.9
FITvals_kap_2_pokus4.dat	-106.1	-151.6	-156.8	-162.0	-161.4
FITvals_kap_2_pokus5.dat	-132.2	-161.4	-124.9	-124.5	-141.0
FITvals_kap_2_pokus6.dat	-147.9	-144.4	-148.5	-127.2	-175.0
FITvals_kap_2_pokus7.dat	-128.8	-168.7	-168.0	-163.5	-190.2
FITvals_kap_2_pokus8.dat	-126.0	-135.5	-161.9	-166.9	-174.4
FITvals_kap_2_pokus9.dat	-117.4	-151.0	-149.3	-150.3	-161.3
Mean value [kPa]	-140.2				
Confidence interval [kPa]	$\pm$24.6				

As clearly seen above, this experiment failed for capillary two. We get only negative value at 200 which is not logical for material in atmospheric pressure to have negative pressure value.

Capillary 4

H = 4 mm	$p_{exit,1}$	$p_{exit,2}$	$p_{exit,3}$	$p_{exit,4}$	$p_{exit,5}$
File name	[kPa]	[kPa]	[kPa]	[kPa]	[kPa]
FITvals_kap_4_pokus0.dat	-51.0	-77.6	-79.1	-77.8	-75.2
FITvals_kap_4_pokus3.dat	-74.1	-80.1	-75.4	-72.4	-70.6

FITvals_kap_4_pokus5.dat	-53.3	-53.3	-72.8	-64.3	-52.7
FITvals_kap_4_pokus7.dat	-59.5	-75.7	-89.9	-62.7	-53.0
FITvals_kap_4_pokus8.dat	-70.2	-53.4	-82.2	-75.9	-75.5
Mean value [kPa]	-69.1				
Confidence interval [kPa]	±11.2				

As clearly seen above, this experiment also failed for capillary four. We get only negative value at 200 which is not logical for material in atmospheric pressure to have negative pressure value.

For further more data values (a and b values of linear regression), please check <u>Appendix D.</u>

Summary Of The Chapter

It seems that the proposed methodology is simplified to determine exit pressure fails. There is no physical reason to obtain negative exit pressure. The possible explanation is the absence of data correction especially <u>Rabinovitch</u> correction and <u>Bagley</u> correction. However used experimental data are not suitable for using this correction due to the absence of different lengths in capillaries. That is the reason those corrections mentioned are not performed in this work. It is recommended for further work to involve these corrections to the calculations. However, it needs more specific experimental data according to the demands of these corrections (e.g. Minimum 3 different lengths of the capillaries). In our case data for 8 mm width capillary was not suitable for the experiment where data were deteriorated.

5.Conclusion

The thesis deals with the investigation of the flow behaviour of collagen material. Literature search focused on collagen material description, the flow behaviour of collagen, experimental techniques to obtain information about flow behaviour of collagen material and theoretical approaches to explain and describe of the flow of material similar to collagen. From the literature search, the best device for measurement collagen material in the form of dough is slit rheometer. There are lack information about rheology of collagen material. Pure viscous power-law model or Herschel-Bulkley model are used for the flow description of collagen material.

The experiment results from literature were used for the rheology of collagen investigation. Measurements were performed on slit rheometer with three different capillaries. Slits have a rectangular shape with constant width and various thickness of 2, 4 and 8 mm.

The methodology of experimental data treatment was suggested and assembled into the own script. Data were separated, filtered and analysed by regression. Two mathematical models describing flow behaviour were used for fitting the reconstructed program. The power-law model with two parameters and Herschel-Bulkley model with three parameters were used. From the results of the data treatment, the parameters of both models were identified. A suitable model for collagen description is the power-law model, but Herschel-Bulkley fails. The parameters of the power-law model achieve an acceptable agreement with data from the literature.

Also, a script for exit pressure assessment was assembled. It interpolates exit pressure at the dye of the capillary from axial pressure trends within the capillary for different shear rates. But it seem, that proposed method fails, because exit pressure for capillary 2 and 4 mm reach negative values (under pressure). Therefore it is recommended to repeat experiment with three different capillaries for future. But 8 mm capillary is not recommend because it is not suitable for this kind of material due to not fully filled cross-section of the capillary at its die. When data from three capillaries will be collected, it is also possible to apply Rabinowitch and Bagley correction for exit pressure prediction.

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List of Symbols

Parameter	Description	Unit
De	Deborah Number	[-]
Wi	Wissenberg Number	[-]
v	Velocity	[m.s^{-1}]
A	Area	[m^2]
F	Force	[N]
$\dot{\gamma}$	Shear Rate	[s^{-1}]
E	Young's modulus	[Kg.m^{-1}.s^{-2}]
ε	Strain	[-]
σ	Stress	[N.m^{-2}]
k	Spring Constant	[N.m^{-1}]
G	Spring model shear modulus	[Pa]
λ	Relaxation time	[s^{-1}]
τ	Shear stress	[N.m^2]
τ_0	Yield Stress – Shear at Wall	[N.m^2]
K	Consistency Index	[-]
μ	Viscosity	[Pa.s]
ϑ	Dynamic Viscosity	[m^2.s^{-1}]
ρ	Density	[kg.m^{-3}]
γ	Poisson's ratio	[-]
u_x	Velocity profile at x-axis (See Eq.2.2.2.6)	[ms^{-1}]
M_t	Torque	[F.m]
$P_{Machine}$	Power output of the machine	[Watt]
P	Pressure	[Pa]
H	Height	[m]
W	Width	[m]
t	Time	[seconds]
D	Diameter	[m]
S	Surface	[m^2]
$\dot{V}$	Volumetric flow rate	[m^3.s^{-1}]
n	Flow behaviour index	[-]
p_{exit}	Exit pressure of capillary	[kPa]

Appendix A – Matlab script

This appendix covers the main and auxiliary script used in data treatment.

Main Code – Main.m
Used for Herschel-Bulkley and power law model appliances

```matlab
clc
clear
%This will make Displacement Stay Always in the 8th row
mainFolder = cd("C:\Users\ahmet\Desktop\DATA");
cd(mainFolder)
Filelist = dir("kap2/*pokus*.dat"); % Filter Range 7581-7587 % Current Used
Filter is = 1957 2067
cd("kap2")
% Get the Values For the Names of the File %
% VeriAdlari = ls("*pokus*.dat"); VeriAdlari = string(VeriAdlari);
% ExperimentDataNames = table(VeriAdlari,'VariableNames',{'n'});
% Get the Values For the Names of the File %
mkdir Evo;
currentFolder = cd;
height = 0.002; % height in [meters]
b = 0.02; % width in [meters]
len = 0.15; %Length in [meters]
% Pre-allocating the array %
sizeMaxLen = length(Filelist);
Filedirs = zeros(1,sizeMaxLen);
% Pre-allocating the array %
for i=1:length(Filelist) %length(Filelist)
    Tables = readtable(Filelist(i).name);
    Time = Tables.t; % Time
    PressureF = Tables.p1*100000; % Pressure 1
    PressureS = Tables.p2*100000; % Pressure 2
    Displacement = Tables.h/1000; % Displacement
    TimeArr = 1:length(Time); % To Find Index Faster
    plot(TimeArr,PressureF,'b-',TimeArr,PressureS,'r-')
    filtStart = input('Filter Start Point :');
    filtEnd = input('Filter End Point :');
    % Applying Range Filtration %
    Time = Time(filtStart:filtEnd);
    PressureF = PressureF(filtStart:filtEnd);
    PressureS = PressureS(filtStart:filtEnd);
    Displacement = Displacement(filtStart:filtEnd);
    % Applying -END- Filtration -END- %
    pfinal = abs(PressureS-PressureF);
    % Go Back to main folder Apply functions %
    cd(mainFolder);
    velocity = Derivator(Time,Displacement);
    golayFitFrameLength = round_odd(abs(filtEnd-filtStart));
    pfinal = sgolayfilt(pfinal,2,golayFitFrameLength);
    Displacement = sgolayfilt(Displacement,2,golayFitFrameLength);
    velocity = sgolayfilt(velocity,2,golayFitFrameLength);
    % Go Back to main folder Apply functions %
    cd(currentFolder) % Move To File Application Folder
    plot(Time,pfinal);
    plot(Time,Displacement);
    % Saving Output Data %
    %OutputDataAll = [time,disp,pfinal,p1,p2]; % OutputData as All
    %unnecessary forms
    directory = 'Evo/'
```

```matlab
    filei= char(string(i));
    extension = '.dat'
    %FinalData Initialization
    SurfacePiston = (power(0.08,2)*pi)/4;
    Volflow = SurfacePiston*velocity; % Getting volume flow rate
    tau = (pfinal*height)/(2*len) ; % Shear Stress
    gamma = (6*Volflow)/(b*power(height,2));
    gamma = transpose(gamma);
    [f,v]= fit(gamma,tau,'a*x^b+c'); % Hershel Bulkley Fluid
    [g,n]= fit(gamma,tau,'a*x^b'); % Power Law Mmodel
    plot(f,gamma,tau);
    [Gval Gpos] = max(gamma);
    [GvalMin GposMin] = min(gamma);
    figExt = '.fig';
    %figName = [directory filei figExt];
    figName = filei;
    savefig(figName);
    % HB MODEL %
    HBExt = '_HBModel';
    HBId = [directory filei HBExt extension];
    HershelBulkleyData =
[f.a,f.b,f.c,v.sse,v.rsquare,v.adjrsquare,v.rmse,Gval,Gpos,GvalMin,GposMin,
filtEnd,filtStart];
    OutputHBModel =
array2table(HershelBulkleyData,'VariableNames',{'A','B','C','SumSquareErr',
'RoundSquare','AdjecentSquare','RMSErr','MaxGamma','MaxGammaPosition','MinG
amma','MinGammaPosition','FilterStart','FilterEnd'});
    writetable(OutputHBModel,HBId);
    % PowerLaw MODEL %
    PLExt = '_PLModel';
    PLId = [directory filei PLExt extension];
    PowerLawData =
[g.a,g.b,n.sse,n.rsquare,n.adjrsquare,n.rmse,Gval,Gpos,GvalMin,GposMin,filt
End,filtStart];
    OutputPLModel =
array2table(PowerLawData,'VariableNames',{'A','B','SumSquareErr','RoundSqua
re','AdjecentSquare','RMSErr','MaxGamma','MaxGammaPosition','MinGamma','Min
GammaPosition','FilterStart','FilterEnd'});
    writetable(OutputPLModel,PLId);
    % Raw Data Save Start %
    velconv = transpose(velocity);
    Rawfilename = [directory filei '_raw' extension];
    OutputData = [Time,velconv,pfinal,gamma,tau];
    OutputTable =
array2table(OutputData,'VariableNames',{'Time','Velocity','Pressure','Gamma
','Tau'});
    writetable(OutputTable,Rawfilename); % Verilerin ciktisi
    % Raw Data Save End %
end
cd(mainFolder)
% writetable(ExperimentDataNames,'Fnames.dat'); Unused due to function is
% implemented on VeriToplayici
fprintf("\nProcess is completed !\n");
%After showing to skocilas erase p1 and p2 and just make final P form and
%use data from it !!!!
```

Derivator function – Derivator.m
Implemented in main code in order to get derivation. This function uses numerical methods in order to obtain derivative

```matlab
function Result = Derivator(x,y)
%Derivator Derivates Y and X
%   Derivates the given two array numerically, T values are x and H values
%   are y
sizeH = size(y);
if(sizeH(1) > sizeH(2))
    sizeH = sizeH(1);
else
    sizeH = sizeH(2);
end
for i=1:sizeH
    if(i==1)
        D(i) = y(i)/x(i);
    else
        D(i)=(y(i)-y(i-1))/(x(i)-x(i-1));
    end
Result = D;
end
```

Prediction of the exit pressure – Elastiksimplified.m
This script is used for obtaining axial pressure profile along the capillary with aim to get exit pressure.

```matlab
clc
clear
mainFolder = cd;
cd(mainFolder)
cd('RAWDATA')
GaugetoPressure = 100000;
CapName = "kap_2";% Change Only Capillary name from here
VeriAdlari = ls(CapName+"*");
VeriAdlari = string(VeriAdlari);
for isim=1:length(VeriAdlari)
% %      fprintf("%d - %s \n",i,VeriAdlari(i));
% % fprintf("Please select a Capillary Data : ")
% % datChoosen = input("\n");
fileID = VeriAdlari(isim);
ProcessFold = cd;
% Table Reading and Assingning the values %
Table = readtable(char(VeriAdlari(isim)));
Time = Table.Var1;
P1 = Table.Var2;
P2 = Table.Var3;
P3 = Table.Var4;
P4 = Table.Var5;
P5 = Table.Var6;
x = Table.Var8;
tArr = 1:length(Time);                              % Range 2754+3549
% Table Reading and Assingning the values %
plot(tArr,P1,tArr,P2,tArr,P3,tArr,P4,tArr,P5);
filtS = input('Filter Start Point :');
filtE = input('Filter End Point :');
% Table Filtering and Re-Assingning the values %
% Table Filtering and Re-Assingning the values %
PTOTAL = [P1*GaugetoPressure P2*GaugetoPressure P3*GaugetoPressure
P4*GaugetoPressure P5*GaugetoPressure];
rangeDiv = floor(abs(filtE-filtS)/5);
G = zeros(1,5); % Pre Allocation
for j=1:5
```

```matlab
        G(j) = filtS + (j*rangeDiv);
        %Disp(j) = x(G(j));
    end
Distance = [35 70 105 140 165]; % P6 to P1 Find X at 175
Disp = zeros(5,5);
DispT = Disp;
Gamma = zeros(5,5);
TransGamma = zeros(5,5);
for j=1:5 % Row Number
    for k=1:5 % Colomn Number
        Disp(j,k) = Distance(j);
        DispT(j,k) = Distance(k);
        TransGamma(j,k) = PTOTAL(G(j),k); % Will write it in the colomn
        %Gamma(j,k) = PTOTAL(G(7-j),k); % In Case
        % PTOTAL has values in the Rows and indices in colomn
        % Normally it starts from P1 by PTOTAL(G(j),k); Command
        % But since Skocilas demanded from high pressure to low
        % We have to use PTOTAL(G(7-j),k);
    end
end
% Swapping The Function to Low To High %
for i=1:5
    for j=1:5
        Gamma(i,j) = TransGamma(i,6-j);
    end
end
tGamma = transpose(Gamma);
Transdist = transpose(Distance);
% Swapping The Function to Low To High %
mkdir 'Pexit'
cd('Pexit')
% FIT AND PLOT VALUES FOR FIT
[F5,V5] = fit(Transdist,tGamma(:,5),'poly1');
[F4,V4] = fit(Transdist,tGamma(:,4),'poly1');
[F3,V3] = fit(Transdist,tGamma(:,3),'poly1');
[F2,V2] = fit(Transdist,tGamma(:,2),'poly1');
[F1,V1] = fit(Transdist,tGamma(:,1),'poly1');
% STARTED PLOTTING
plot(F5,Transdist,tGamma,'b.') % Draws The Trendline
title('Pressure - Displacement')
xlabel('X - Displacement');
ylabel('DeltaP - Pressure');
hold on
plot(F4,Transdist,tGamma,'b.')
hold on
plot(F3,Transdist,tGamma,'b.')
hold on
plot(F2,Transdist,tGamma,'b.')
hold on
plot(F1,Transdist,tGamma,'b.')
savefig("Pexit"+fileID);
hold off
% File Extension Definer
mkdir(CapName)
cd(CapName)
fileExt = ".dat";
% File Extension Definer
% Values Saver for F
FitID = "FITvals_"+fileID+fileExt;
```

```matlab
FitValMatrix =
[F1.p1,F1.p2,F1.p2+F1.p1*200;F2.p1,F2.p2,F2.p2+F2.p1*200;F3.p1,F3.p2,F3.p2+
F3.p1*200;F4.p1,F4.p2,F4.p2+F4.p1*200;F5.p1,F5.p2,F5.p2+F5.p1*200];
FitOutput = array2table(FitValMatrix,'VariableNames',{'p1','p2','sonuc'});
writetable(FitOutput,FitID);
% Values Saver for V
AdjID = "ADJvals_"+fileID+fileExt;
AdjValMatrix =
[V1.rsquare,V1.sse;V2.rsquare,V2.sse;V3.rsquare,V3.sse;V4.rsquare,V4.sse;V5
.rsquare,V5.sse];
AdjOutput = array2table(AdjValMatrix,'VariableNames',{'rsquare','sse'});
writetable(AdjOutput,AdjID);
cd(ProcessFold)
end
```

Appendix B – Parameters of Power-law model of collagen material

Capillary H = 8 mm

File	K [Pa.s^n]	n [-]	$\dot{\gamma}_{max}$ [s^{-1}]	$\dot{\gamma}_{min}$ [s^{-1}]	R^2
10_kap_8_pokus7.dat	352	0.28	17879	3070	0.95
11_kap_8_pokus8.dat	189	0.35	14000	3212	1.00
12_kap_8_pokus9.dat	504	0.26	16425	3214	0.99
1_kap_8_pokus1.dat	734	0.22	14448	1482	0.97
2_kap_8_pokus10.dat	775	0.21	17632	886	0.99
3_kap_8_pokus11.dat	670	0.23	13116	942	0.98
6_kap_8_pokus3.dat	636	0.24	16337	749	1.00
7_kap_8_pokus4.dat	981	0.19	17036	1343	0.99
8_kap_8_pokus5.dat	2	0.98	1976	1887	0.68
9_kap_8_pokus6.dat	1414	0.13	16917	2376	0.82
Average	**626**	**0.31**	**MIN**	**MAX**	
St Dev	**402**	**0.24**	**13116**	**3214**	

Capillary H = 4 mm

File	K [Pa.s^n]	n [-]	$\dot{\gamma}_{max}$ [s^{-1}]	$\dot{\gamma}_{min}$ [s^{-1}]	R^2
1_kap_4_pokus0.dat	1442	0.22	9157	2349	0.99
2_kap_4_pokus3.dat	2136	0.18	11006	821	1.00
3_kap_4_pokus5.dat	1626	0.20	12254	3546	1.00
4_kap_4_pokus7.dat	2273	0.15	12039	1518	0.95
5_kap_4_pokus8.dat	759	0.27	13485	3517	0.84
Average	**1647**	**0.20**	**MIN**	**MAX**	
St Dev	**604**	**0.04**	**9157**	**3546**	

Capillary H = 2 mm

File	K [Pa.s^n]	n [-]	$\dot{\gamma}_{max}$ [s^{-1}]	$\dot{\gamma}_{min}$ [s^{-1}]	R^2
10_kap_2_pokus8.dat	832	0.35	9839	1387	0.96
11_kap_2_pokus9.dat	2093	0.24	11847	1879	0.99
1_kap_2_pokus1.dat	726	0.37	12372	1508	0.99
2_kap_2_pokus10.dat	2446	0.22	9237	940	0.99
3_kap_2_pokus12.dat	1429	0.30	7824	1358	0.99
4_kap_2_pokus13.dat	982	0.32	14144	1330	0.98
5_kap_2_pokus14.dat	541	0.39	9217	317	0.92
6_kap_2_pokus4.dat	2248	0.24	12994	576	0.98
7_kap_2_pokus5.dat	2923	0.20	12815	803	0.98
8_kap_2_pokus6.dat	857	0.34	15109	976	0.98
9_kap_2_pokus7.dat	1094	0.31	8048	604	0.98
Average value	**1470**	**0.30**	**MIN**	**MAX**	
STD	**815**	**0.06**	**7824**	**1879**	

Appendix C – Parameters of Herschel-Bulkley model of collagen material

Capillary H = 8 mm
Capillary H = 8 mm data is only proved that it is completely useless and waste to work on capillary with 8 mm thickness for this specific material with this set-up.

Capillary H = 4 mm

File	K [Pa.s^n]	n [-]	τ_y [Pa]	$\dot{\gamma}_{max}$ [s^{-1}]	$\dot{\gamma}_{min}$ [s^{-1}]	R^2
1_kap_4_pokus0.dat	18062	0.07	-24618	9157	2349	0.93
2_kap_4_pokus3.dat	4967	0.12	-4234	11006	821	1.00
3_kap_4_pokus5.dat	342	0.33	3662	12254	3546	1.00
4_kap_4_pokus7.dat	83715	0.02	-90208	12039	1518	0.76
5_kap_4_pokus8.dat	0	2.66	7299	13485	3517	0.99
Average	**21417**	**0.64**	**-21620**	**MIN**	**MAX**	
St Dev	**35587**	**1.13**	**40284**	**9157**	**3546**	

Capillary H = 2 mm

File	K [Pa.s^n]	n [-]	τ_y [Pa]	$\dot{\gamma}_{max}$ [s^{-1}]	$\dot{\gamma}_{min}$ [s^{-1}]	R^2
10_kap_2_pokus8.dat	80583	0.05	-106522	9839	1387	0.94
11_kap_2_pokus9.dat	9	0.75	11159	11847	1879	1.00
1_kap_2_pokus1.dat	255	0.46	3186	12372	1508	0.99
2_kap_2_pokus10.dat	33	0.63	9235	9237	940	1.00
3_kap_2_pokus12.dat	17	0.73	9398	7824	1358	1.00
4_kap_2_pokus13.dat	9660	0.15	-17776	14144	1330	0.98
5_kap_2_pokus14.dat	0	1.31	8837	9217	317	1.00
6_kap_2_pokus4.dat	32	0.64	9697	12994	576	1.00
7_kap_2_pokus5.dat	35	0.60	9997	12815	803	1.00
8_kap_2_pokus6.dat	12	0.75	7978	15109	976	1.00
9_kap_2_pokus7.dat	13	0.75	7540	8048	604	1.00
Average	**8241**	**0.62**	**-4297**	**MIN**	**MAX**	
St Dev	**24166**	**0.33**	**34872**	**7824**	**1879**	

Appendix D – Parameters of Exit Pressures

Capillary H = 8 mm
Capillary H = 8 mm data is only proved that it is completely useless and waste to work on capillary with 8 mm thickness for this specific material with this set-up.

Capillary H = 4 mm

H = 4 mm	$p_{exit,1}$	$p_{exit,2}$	$p_{exit,3}$	$p_{exit,4}$	$p_{exit,5}$
File name	[bar]	[bar]	[bar]	[bar]	[bar]
FITvals_kap_4_pokus0.dat	-51.0	-77.6	-79.1	-77.8	-75.2
FITvals_kap_4_pokus3.dat	-74.1	-80.1	-75.4	-72.4	-70.6
FITvals_kap_4_pokus5.dat	-53.3	-53.3	-72.8	-64.3	-52.7
FITvals_kap_4_pokus7.dat	-59.5	-75.7	-89.9	-62.7	-53.0
FITvals_kap_4_pokus8.dat	-70.2	-53.4	-82.2	-75.9	-75.5
Mean value [bar]	-69.1				
Confidence interval [bar]	±11.2				

Capillary H = 2 mm

H = 2 mm	$p_{exit,1}$	$p_{exit,2}$	$p_{exit,3}$	$p_{exit,4}$	$p_{exit,5}$
File name	[kPa]	[kPa]	[kPa]	[kPa]	[kPa]
FITvals_kap_2_pokus1.dat	-81.4	-81.6	-86.3	-89.3	-102.0
FITvals_kap_2_pokus10.dat	-124.3	-143.1	-165.2	-130.1	-163.4
FITvals_kap_2_pokus12.dat	-119.7	-133.6	-131.9	-152.8	-147.7
FITvals_kap_2_pokus13.dat	-117.6	-150.8	-166.0	-139.6	-153.4
FITvals_kap_2_pokus14.dat	-152.9	-145.4	-111.4	-120.1	-125.9
FITvals_kap_2_pokus4.dat	-106.1	-151.6	-156.8	-162.0	-161.4
FITvals_kap_2_pokus5.dat	-132.2	-161.4	-124.9	-124.5	-141.0
FITvals_kap_2_pokus6.dat	-147.9	-144.4	-148.5	-127.2	-175.0
FITvals_kap_2_pokus7.dat	-128.8	-168.7	-168.0	-163.5	-190.2
FITvals_kap_2_pokus8.dat	-126.0	-135.5	-161.9	-166.9	-174.4
FITvals_kap_2_pokus9.dat	-117.4	-151.0	-149.3	-150.3	-161.3
Mean value [bar]	-140.2				
Confidence interval [bar]	±24.6				