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**Abstract:**

The book is structured into five comprehensive chapters. The first chapter lays the foundation by introducing the principles and terminology of rheology, along with its applications and various analogical models. In the second chapter, a detailed exploration of different rheological behaviors is presented. The third chapter delves into the core subject, focusing on the rheological behavior of fresh concrete. The fourth chapter shifts attention to the rheology of granular media and polymers. Finally, the fifth chapter outlines various methods and measurement instruments used in the art of rheology.

# RHEOLOGY OF MATERIALS



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## **Introduction**

This book is intended for **second-year Master's** students in **Civil Engineering**, specializing in "**Civil Engineering Materials**." It is written in accordance with the curriculum set by the Algerian National Pedagogical Committee.

The book is divided into five chapters. The **first chapter** focuses on the base of rheology (principles and terminology), their applications, and various analogical models.

The **second chapter** provides a detailed presentation of different rheological behaviours.

The **third chapter** delves into the core of this book, discussing rheological behaviour of fresh concrete.

The **fourth chapter** is devoted to the rheology of granular media and polymers

The **fifth and last chapter** provides methods and measurement instruments in the art of rheology.

## Chapter I :      **Basics of Rheology**

### **I.1 Introduction and definition**

The term rheology was introduced by the chemist Eugene Bingham. This discipline emerged from the inability of classical theories in fluid and solid mechanics in the 19th century to predict the behaviour of many materials with poorly defined behaviour, which lies between that of a perfect elastic solid and a Newtonian fluid. *Rheology, therefore, is a branch of mechanics that deals with the flow and deformation of materials under the influence of stresses.* This definition was accepted when the American Society of Rheology was founded in 1929. Rheology encompasses several fundamental disciplines such as strength of materials, fluid mechanics, fluid mechanics, plasticity, and more.

Rheology is divided into several types of studies:

- ✓ Experimental Rheology: Experimental determination of behavioural relationships (between stress and deformation or deformation rate).
- ✓ Structural Rheology: Explanation of behaviours based on the material's structure.
- ✓ Theoretical Rheology: Providing mathematical models for a limited number of behaviours, independently of the microscopic structure.

Cette définition a été acceptée lorsque la société américaine de rhéologie a été fondée en 1929.

### **I.2 Application Areas of rheology**

Rheology has found wide application in various fields, and can be applied in different types of studies including:

- The agri-food industry, such as cheese , chocolate, and yogurt production ;
- Coating and paint layers ;
- The pharmaceutical industry ;
- The petroleum industry (oil extraction transportation, and refining) ;
- Flow behaviour of materials and sediments in various applications, and more.

## **I.3 Fundamentals of Fluid Mechanics**

### **I.3.1 Simple Fluids**

A simple fluid can be defined as one that follows Newton's law of behaviour, which states that the shear stress in the fluid is directly proportional to the rate of deformation, known as the shear rate. This linear relationship is encapsulated in the Newtonian equation, where the shear stress is equal to the product of the dynamic viscosity of the fluid and the shear rate. In other words, the resistance to flow exhibited by a simple fluid can be quantified by its dynamic viscosity, which represents the internal friction within the fluid. This fundamental equation provides a framework for understanding the behaviour of simple fluids and serves as a basis for studying their flow characteristics in various applications and industries.

### **I.3.2 Viscosity and Rheology of Simple Fluids**

Viscosity is a fundamental property that characterizes a fluid's resistance to flow. Dynamic viscosity, specifically, is a measure of the internal friction within the fluid, determining how easily it deforms under the influence of an applied shear stress. Another type of viscosity, known as kinematic viscosity, is obtained by dividing the dynamic viscosity by the fluid's density.

Viscosity is commonly expressed in various units, such as Pascal-seconds ( $\text{Pa}\cdot\text{s}$ ) or poise (P), where 1 P equals  $0.1 \text{ Pa}\cdot\text{s}$ . Additionally, centistokes (cSt) is a widely used unit for kinematic viscosity.

When considering fluid behaviour, Newtonian fluids and non-Newtonian fluids are distinguished. Newtonian fluids exhibit a constant viscosity, meaning their flow behaviour is not influenced by stress or shear rate variations. On the other hand, non-Newtonian fluids display a viscosity that depends on the applied stress or shear rate. Non-Newtonian fluids can exhibit behaviours such as shear-thinning (decreasing viscosity with increasing shear rate) or shear-thickening (increasing viscosity with increasing shear rate). Understanding the distinction between Newtonian and non-Newtonian fluids is crucial in characterizing and predicting the flow behaviour of various fluids encountered in practical applications.

## **I.4 Experimental Rheology concepts**

Experimental rheology refers to the study of the flow and deformation behaviour of materials through experimental techniques. It involves the measurement and analysis of the mechanical properties of materials under various conditions. By utilizing appropriate measurement techniques and analyzing the resulting data, researchers and engineers can gain valuable insights into material behaviour, aiding in the development of new products optimization of manufacturing processes and solving complex engineering challenges. Several key concepts are integral to understanding experimental rheology:

### **I.4.1 Shear Stress and Shear Rate**

Shear stress represents the force per unit area applied to a material in response to deformation. It is a measure of the resistance encountered by the material during flow. Shear rate, on the other hand, quantifies the rate at which adjacent layers of the material move relative to each other. The relationship between shear stress and shear rate is crucial in characterizing the flow behaviour of materials.

### **I.4.2 Rheological Measurements**

Experimental rheology involves the use of instruments called rheometers to measure the rheological properties of materials. Rheometers apply controlled deformations or stresses to a sample and measure the resulting response. They provide valuable data on parameters such as viscosity, viscoelasticity, yield stress, and flow behaviour.

### **I.4.3 Viscosity**

Viscosity is a fundamental property that characterizes a material's resistance to flow. It quantifies the internal friction within a material when subjected to shear forces. The measurement of viscosity provides insights into the material's flow behaviour, ranging from Newtonian fluids with constant viscosity to non-Newtonian fluids with viscosity dependent on shear rate or stress.

Viscosity plays a crucial role in understanding the behaviour of fluids. It characterizes the fluid's resistance to flow, indicating how easily or difficultly it can be poured stirred, or transported. Viscosity arises due to the molecular interactions within the fluid, where the molecules exert attractive and repulsive forces on each other, leading to a resistance against flow. Essentially, it quantifies the fluid's "thickness" or "stickiness," with highly viscous fluids exhibiting a thicker consistency and greater

resistance to flow compared to low-viscosity fluids. The measurement and analysis of viscosity are essential in various scientific and engineering fields, including chemistry, physics, materials science, and fluid dynamics, contributing to the design and optimization of processes involving fluids, such as in the development of lubricants, paints, food products, and industrial processes.

### ***Dynamic Viscosity***

Dynamic viscosity is a fundamental property that quantifies a fluid's internal resistance to flow. It represents the material's ability to resist deformation under the influence of an applied shear stress. The dynamic viscosity of a fluid is directly related to the friction between adjacent fluid layers as they move relative to each other. Understanding the concept of dynamic viscosity and its units of measurement is crucial in studying fluid behaviour and predicting flow characteristics. By measuring and analyzing the viscosity of fluids, scientists and engineers can gain insights into their flow properties enabling the design and optimization of various processes and applications in fields such as engineering, manufacturing, and fluid dynamics.

### ***Kinematic Viscosity***

Kinematic viscosity is a fundamental property that describes the fluid's resistance to flow in relation to its density. It is defined as the ratio of dynamic viscosity to fluid density. While dynamic viscosity quantifies the internal friction within the fluid, kinematic viscosity takes into account the fluid's density, providing a measure of how easily the fluid flows under the influence of applied forces.

Table I-1 Typical values of dynamic and kinematic viscosities obtained at 20°C and 1 bar

Fluid	Dynamic Viscosity (Pa·s)	Kinematic Viscosity (m <sup>2</sup> /s)
Water	0.001	$1.003 \times 10^{-6}$
Air	$1.8 \times 10^{-5}$	$1.8 \times 10^{-5}$
Olive Oil	0.082	$8.18 \times 10^{-5}$
Honey	2.0 – 10.0	$2 \times 10^{-6}$ - $10 \times 10^{-6}$
Motor Oil (SAE 30)	0.27 - 0.31	$2.70 \times 10^{-7}$ - $3.10 \times 10^{-7}$
Glycerin	0.9	$9.0 \times 10^{-7}$

### ***Influence of Thermodynamic Properties on Viscosity***

Temperature has a significant influence on viscosity, and understanding the concept of temperature dependence is crucial in characterizing fluid behaviour. As temperature increases, the viscosity of most fluids tends to decrease, resulting in easier flow. This behaviour is attributed to the increased molecular motion and reduced intermolecular forces at higher temperatures. Viscosity-temperature relationships can be described by various mathematical models. One commonly used model is the Arrhenius equation, which relates viscosity to temperature using an exponential relationship. The Arrhenius equation incorporates an activation energy term to capture the temperature dependence of viscosity.

In addition to temperature, pressure can also have a significant impact on viscosity especially for compressible fluids. Under high pressure conditions, the intermolecular forces and molecular packing can change, leading to alterations in the flow behaviour. For compressible fluids, pressure-viscosity models are used to describe the pressure dependence of viscosity. These models account for factors such as molecular interactions and density changes under pressure.

Understanding the temperature and pressure effects on viscosity is essential in various fields, including material science, chemical engineering, and geophysics. It allows for accurate predictions of fluid behaviour under different conditions, enabling the design and optimization of processes and systems involving temperature and pressure variations.

Table I-2 Influence of temperature on water viscosity

Temperature (°C)	Viscosity (Pa·s)
0	1.792
10	1.308
20	1.002
30	0.797
40	0.653
50	0.547

As shown in the table I-2, the viscosity of water decreases significantly as the temperature increases. This behaviour is commonly observed in many liquid substances. There is no strict relationship between viscosity and temperature, but a mathematical model developed by researcher Guzman-Andrade can be used:

$$\eta = a. e^{\frac{b}{T}}$$

Where the constants "a" and "b" depend on the nature of the liquid and "T" represents the absolute temperature.

#### **I.4.4 Viscoelasticity**

Viscoelastic materials exhibit both viscous (fluid-like) and elastic (solid-like) behaviour. They can store and release energy under deformation. Viscoelasticity is characterized by parameters such as storage modulus (elastic response) and loss modulus (viscous response). Understanding the viscoelastic behaviour of materials is crucial in various fields, including polymer science, food processing, and biomedical engineering.

#### **I.4.5 Time and Temperature Dependence**

Many materials display time-dependent and temperature-dependent rheological behaviour. Time-dependent phenomena include stress relaxation (decrease in stress over time under constant deformation) and creep (gradual increase in strain under constant stress). Temperature affects the molecular motion within materials, influencing their viscosity, viscoelasticity, and flow behaviour.

#### **I.4.6 Analog Models (Building Blocks of Rheology)**

Qualitative results obtained from simple tests on a variety of materials allow us to classify them according to their basic behaviours. These behaviours can be represented by three elementary mechanical systems:

Elastic behaviour: Materials exhibiting elastic behaviour deform under stress but return to their original shape once the stress is removed.

Plastic behaviour: Materials exhibiting plastic behaviour undergo permanent deformation under stress, and they don't completely recover their original shape once the stress is removed.

Viscous behaviour: Materials exhibiting viscous behaviour deform continuously under stress, and their deformation is dependent on the rate of applied stress. They don't recover their original shape once the stress is removed.

Figure I-1 illustrates the four basic building blocks representing the discussed behaviours:

The spring: symbolizes the behaviour of a perfect linear elastic material, where there exists a one-to-one relationship between the applied stress and the resulting deformation. This represents materials that return to their initial states after unloading (Figure I-1a).

The damper: symbolizes the behaviour that represents a linear relationship (Figure I-1b) or a nonlinear relationship (Figure I-1c) between the applied stress and the rate of deformation. If this relationship is linear, it is referred to as pure viscosity. The viscosity of the material acts as an absorption of a portion of the imposed energy. Materials with a linear relationship (b) are governed by Newton's law.

The slider: models the occurrence of permanent deformations when the load is sufficient (Fig. I-1d). If the threshold for the appearance of permanent deformation does not change with the loading, the behaviour is referred to as perfect plasticity. Furthermore, if the deformation before flow is neglected the model is rigid-perfectly plastic.

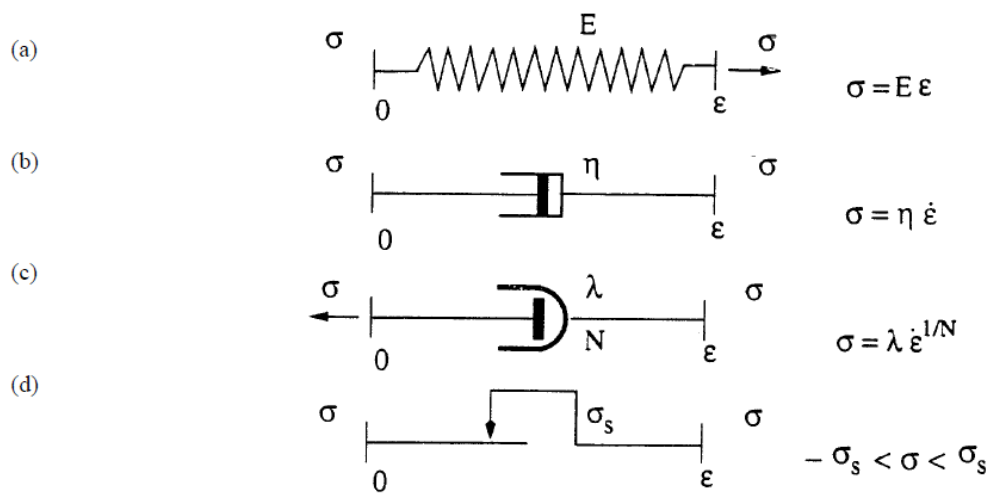


Figure I-1: Illustrates the four basic building blocks representing the discussed Behaviours

## I.5 Notions of Laminar Shear Flow

### I.5.1 Définition

A material subjected to a set of forces is prone to deformation, and the movements of different points within the material depend on the distribution and intensity of the applied forces, laminar shear flow is generated for certain distributions of these forces. During such flow, the material is considered to have a structure of adjacent layers or lamellae. The deformation of the material occurs through the relative sliding of these different layers, without any transfer of material from one layer to another. Laminar shear flow is generated using rheometers. It is from such flow that the rheological parameters of fluids can be determined.

### I.5.2 Shear Movement (assumptions of the model)

Shear Flow (Model Assumptions) Shear stress (denoted as  $\tau$ ) is the fundamental dynamic quantity in rheology. During laminar shear flow, two successive layers in contact with each other move relative to one another. Frictional forces occur at the interface of these two layers, exerting tangential forces on the surface of the layer. These forces are known as shear forces (Figure I-1).

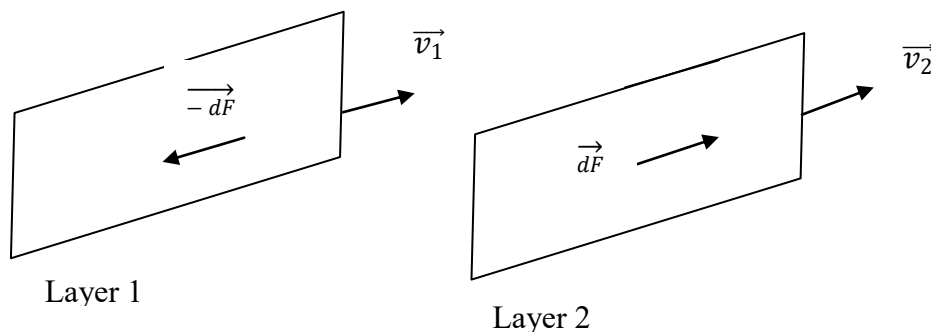


Figure I-2: Forces acting on two neighbouring layer elements.

Assuming that layer (1) is moving with a velocity  $v_1$  greater than the velocity  $v_2$  of layer (2), layer (1) exerts a shear force  $dF$  on layer (2) parallel to the movement, tending to accelerate layer (2). In turn, layer (2) exerts a shear force ( $-dF$ ) on layer (1), tending to slow it down. By relating these shear forces to the surface area, we define the shear stress  $\tau$ :

$$\tau = \frac{dF}{dS}$$

$\tau$  Represents a force per unit of surface area and is expressed in Newton's per square meter ( $\text{N/m}^2$ ) or more conveniently in Pascal's (Pa).  $dS$  is the elemental area of the considered entity.

It should be noted that in the calculation of the resultant forces, pressure forces acting perpendicular to the surface are not taken into account due to their small value compared to shear forces.

### I.5.3 Deformation and Shear Rate

These two quantities are fundamental kinematic parameters in rheology. The definition of shear deformation is presented in the simplest particular case of shear flow with plane symmetry. The material is sheared between two parallel planes one of which is mobile while the other remains stationary (Figure I-3).

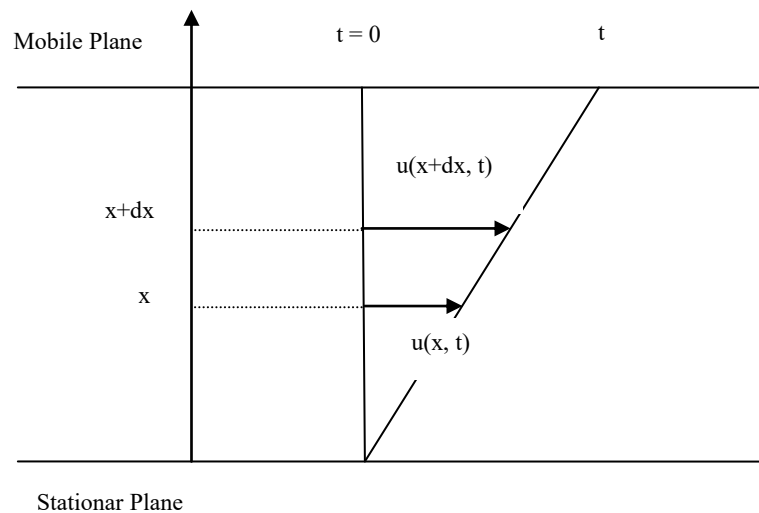


Figure I-3: Laminar shear flow between two parallel planes.

Arbitrarily, it is assumed that the material particles of two adjacent layers are located at a given instant  $t = 0$  in a straight section. At a later time  $t$ , the particles will have traveled the distance  $u(x, t)$  for the particle located at the abscissa  $x$ , and  $u(x+dx, t)$  for the particle located at the abscissa  $x+dx$ . Shear deformation is defined by the following equation:

$$\gamma = \frac{du(x,t)}{dx} = \frac{(u(x+dx,t)-u(x,t))}{dx}$$

Where:

- $\gamma$  is the shear deformation;
- $u(x, t)$  represents the displacement of the particle located at abscissa  $x$  at time  $t$ ;
- $u(x + dx, t)$  represents the displacement of the particle located at abscissa  $x + dx$  at time  $t$ ;
- $dx$  is the distance between the two particles.

This equation quantifies the relative displacement between adjacent particles over a unit distance and defines the shear deformation.

$\dot{\gamma}$  Represents the time derivative of shear deformation. It has the dimension of inverse time and is expressed in  $s^{-1}$  (reciprocal seconds) or commonly referred to as "per second".

The time derivative of shear deformation measures the rate of change of shear deformation with respect to time. It provides information about the speed at which the material is undergoing shear deformation.

#### **I.5.4 Equation of State**

The determination of these two quantities (shear stress and strain or shear rate) allows us to define the rheological equation of state of the material, which is the relationship between them:  $\gamma = f(\tau)$  or  $\dot{\gamma} = f(\tau)$ . Rheograms are graphical representations of the rheological equation of state of the material. The most common representation involves expressing the variation of shear stress with shear rate.

Determining these parameters allows, among other things, to obtain the viscosity of the studied fluid (in the case of Newtonian fluids), a quantity often sufficient to accurately characterize the rheological behaviour of materials. Viscosity is expressed in Pa·s (Pascal-seconds) and is denoted by  $\eta$ . Table I-1 provides viscosity values for common substances at room temperature.

## Chapter II : Different Rheological Behaviours

### II.1 Newtonian Fluids and Non-Newtonian Fluids (Nonlinear)

#### II.2 Newtonian Fluids

In Newtonian viscous fluids, the shear stress tensor  $\tau$  increases gradient tensor  $du/dy$ , denoted as  $\dot{\gamma}$ . This is Newton's law where the viscosity  $\mu$  is constant. This law is valid only for certain substances characterized by low molecular structures, such as water, for example. The flow curve relating shear stress to velocity gradient is a straight line with a slope of  $\mu$ . For a Newtonian fluid, viscosity is independent of the shear rate and the duration for which the fluid is subjected to stress. It only depends on temperature and pressure.

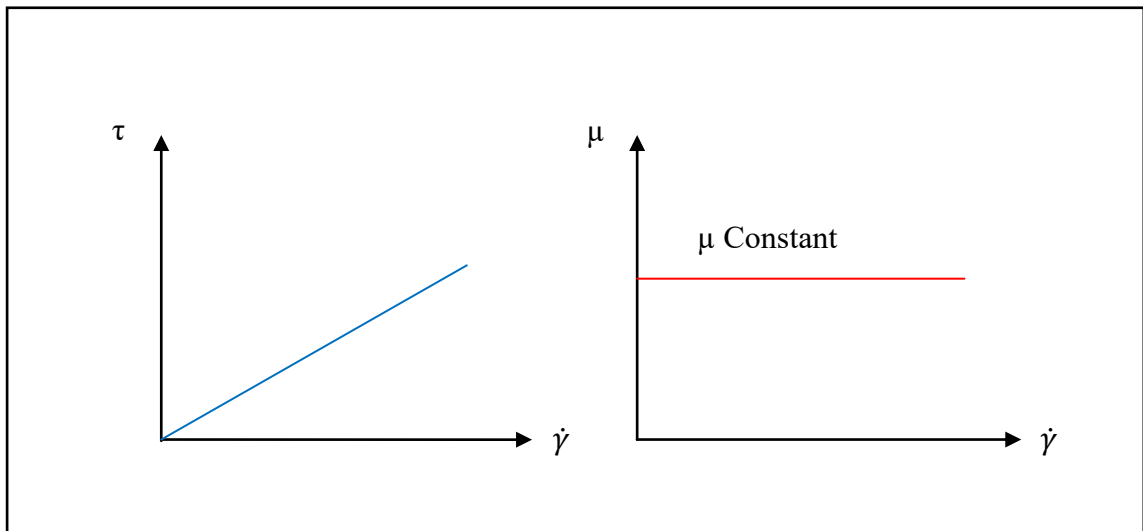


Figure II-1: Rheograms and Dynamic Viscosity Curve of a Newtonian Fluid

The Figure II-1 represents the rheograms and the dynamic viscosity curve of a Newtonian fluid. Rheograms are graphs that show the relationship between shear stress ( $\tau$ ) and shear rate ( $\dot{\gamma}$ ) of the fluid. In the case of a Newtonian fluid, the rheograms are straight lines with a constant slope.

The dynamic viscosity curve represents the viscosity of the fluid as a function of shear rate. As mentioned earlier, for a Newtonian fluid, the viscosity remains constant regardless of the shear rate. Therefore, the dynamic viscosity curve for a Newtonian fluid would be a horizontal line.

These graphical representations help characterize the rheological behaviour of a fluid and determine its viscosity at different shear rates. In the case of a Newtonian fluid, the viscosity, indicating a linear response to applied shear stress.

## II.2.1 Non-Newtonian Fluids

There are several models to simulate the viscosity of non-Newtonian fluids because there is no universal model. These fluids do not all behave in the same way, and some may also exhibit shape memory or other special characteristics that make their behaviour difficult to simulate. It is the domain of rheology to determine which model is suitable for a particular fluid. For such fluids, it is not possible to define a constant viscosity. Instead, a more general concept of apparent viscosity is defined, which depends on various parameters, particularly the velocity gradient, the duration of applied stress, and the elasticity modulus of the fluids.

Non-Newtonian materials can be classified into shear-thinning materials and shear-thickening materials. Additionally, they can be classified as time-independent or time-dependent materials.

These fluids are characterized by the absence of a linear relationship between shear stresses and deformations. For these types of fluids, viscosity depends on the shear rate. They are also characterized by the variation in the ratio of shear stress to velocity gradient for the same temperature and pressure.

Numerous materials fall into this category, such as fresh concrete, plastics, hot bitumen, and toothpaste, to name a few.

In most cases, the following four flow classifications can describe non-Newtonian fluids:

- ✓ Behaviour without yield stress: These fluids exhibit a continuous flow without a threshold or minimum stress required to initiate flow. The viscosity decreases with increasing shear rate.
- ✓ Behaviour with yield stress: These fluids require a certain threshold or minimum stress, known as the yield stress, to initiate flow. Once the yield stress is exceeded, the viscosity decreases with increasing shear rate.
- ✓ Thixotropic behaviour: Thixotropic fluids exhibit time-dependent changes in viscosity. When subjected to shear stress, their viscosity

decreases over time. However, when the shear stress is removed, the viscosity gradually increases again.

- ✓ Viscoelastic behaviour: Viscoelastic fluids exhibit both viscous and elastic properties. They can deform and flow like a viscous fluid under certain conditions, but they also exhibit elastic behaviour and can recover their original shape after deformation.

These classifications help describe and understand the complex behaviours exhibited by non-Newtonian fluids in various applications and industries.

### ***Behaviour without threshold stress***

This characteristic of non-Newtonian fluid behaviour without a threshold stress is composed of pseudoplastic and shear-thickening fluids.

#### **Pseudoplastic fluids (shear-thinning fluids)**

These fluids show a decrease in resistance as the shear rate increases. This means that the viscosity of the fluid decreases when subjected to higher shear rates. Some paints exhibit this property to facilitate their application. The application of shear stress causes the material's structure to break down, leading to a decrease in viscosity. The viscosity of the fluid can vary over time, and if the material is left undisturbed, it gradually returns to its original structure and viscosity. Pseudoplastic fluids are characterized by the relationship between shear rate ( $\dot{\gamma}$ ) and shear stress ( $\tau$ ), which varies:

$$\frac{\tau}{\dot{\gamma}} = \eta_{app} \quad (\text{Apparent viscosity})$$

This relationship is typically represented by a decreasing curve on rheological graphs. It is important to note that this description is based on the general behaviour of pseudoplastic fluids, but specific variations can occur depending on the properties and composition on each material, his viscosity is constant for lower imposed shear rates. However, it gradually decreases with increasing velocity gradient until it reaches a limiting value, hence the name shear-thinning fluids. The most suitable rheological model to describe the behaviour of this category of pseudoplastic fluids is the Ostwald de Waele model:

$$\tau = K \left( \dot{\gamma} \right)^n$$

K is the consistency index of the fluid, and n represents the flow behaviour index. The apparent viscosity is given by the following equation:

$$\eta_{app} = K \left( \dot{\gamma} \right)^{n-1}$$

In this equation, the apparent viscosity is proportional to the shear rate raised to the power of (n-1). The values of K and n depend on the specific characteristics of the pseudoplastic fluid and can be determined experimentally through rheological tests.

### Dilatant Fluids (Shear-Thickening)

Shear-thickening fluids are characterized by an increase in viscosity as the shear stress or velocity gradient increases. The Ostwald model is the most reliable for describing this behaviour; provided that the flow behaviour index (n) is greater than 1. Figure II-3 below shows the different types of fluids described. Each curve plotted in rheology is called a rheogram.

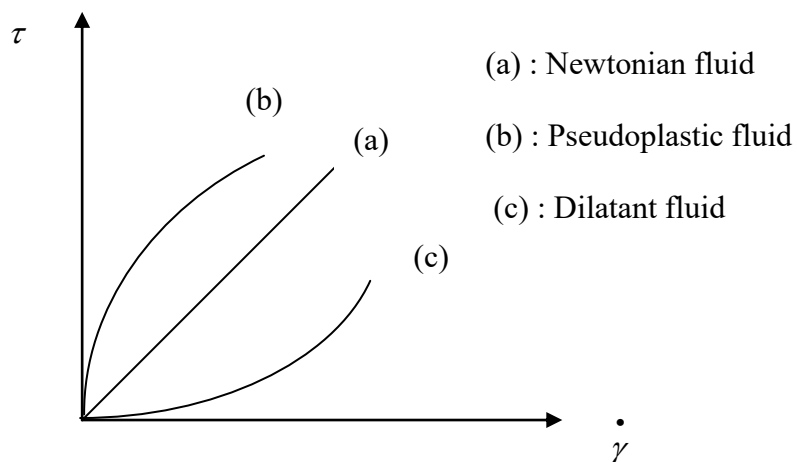


Figure II-2: Rheogram of fluids without a threshold stress.

Table II-1 : The rheological laws of fluids without threshold stress

<i>Model</i>	<i>Rheological laws</i>
<i>Ostwald de Waele</i>	$\tau = K \left( \dot{\gamma} \right)^n$
<i>Ellis</i>	$\tau = \left[ \eta_0 / \left( 1 + \left( \tau / \tau_1 \right)^{\alpha-1} \right) \right] \cdot \dot{\gamma}$
<i>Sisko</i>	$\tau = \eta_0 \cdot \dot{\gamma} + K \cdot \dot{\gamma}^n$
<i>Reiner Phillipoff</i>	$\tau = \left[ \eta_\infty + \left( \eta_0 - \eta_\infty \right) / \left( 1 + \left( \tau / \tau_s \right)^2 \right) \right] \cdot \dot{\gamma}$
<i>Carreau</i>	$\tau = \left[ \eta_\infty + \left( \eta_0 - \eta_\infty \right) \cdot \left( 1 + \left( \lambda \dot{\gamma} \right)^2 \right)^{\left( n-1/2 \right)} \right]$
<i>Prandtl-Eyring</i>	$\tau = \tau_0 sh^{-1} \left( \tau_0 \cdot \dot{\gamma} \right)$
<i>Cross</i>	$\tau = \left[ \eta_\infty - \left( \eta_0 - \eta_\infty \right) / \left( 1 + \gamma \cdot \dot{t}_1 \right)^p \right] \cdot \dot{\gamma}^n$

### ***Behavior with threshold stress***

Fluids that exhibit threshold stress behaviour are called plastic fluids. The plastic behaviour is characterized by the presence of a yield shear stress below which flow is not favoured (see Figure II-3).

Two types of fluids fall into this criterion: the ideal plastic behaviour fluid known as **Bingham fluid**, and another type of non-ideal plasticity fluid known as **Casson fluid** with a fluidizing plastic behaviour.

In the case of **Bingham fluids**, they require a minimum shear stress, called the yield stress, to initiate flow. Once the yield stress is exceeded, the fluid exhibits a linear relationship between shear stress and shear rate, similar to a Newtonian fluid. The yield stress can be thought of as the minimum force required overcoming the internal friction of the fluid and initiating flow. In the Newtonian region, the fluid's viscosity remains constant regardless of the applied shear stress or shear rate. This behaviour allows for a

predictable and linear relationship between shear stress and shear rate, making it easier to model and analyze the flow properties of Bingham fluids. In the context of Bingham fluids, the term "plastic viscosity" is used to describe the viscosity of the fluid above the yield stress. It represents the resistance to flow exhibited by the fluid when it is in its flowing state, beyond the yield stress. By measuring the plastic viscosity, one can assess the fluid's ability to flow under stress and determine its suitability for specific applications. It plays a significant role in engineering calculations, such as pressure drop calculations in pipelines or the design of pumping systems, where knowledge of the fluid's flow properties is essential for efficient and safe operations.

On the other hand, **Casson fluids** exhibit a non-linear relationship between shear stress and shear rate, characterized by a curved rheogram. These fluids have a yield stress, but their viscosity gradually decreases with increasing shear rate, resulting in shear-thinning behaviour, where the fluid becomes less resistant to flow with increasing shear rate.

The Casson model is commonly used to describe the rheological behaviour of such fluids.

Understanding the non-linear behaviour of **Casson fluids** is crucial in industries that deal with materials exhibiting similar characteristics, such as certain food products, suspensions, and cosmetic formulations. By studying the rheological properties and flow behaviour of Casson fluids, engineers and scientists can optimize processes, enhance product performance, and ensure product stability and consistency.

Table II-2 : Rheological models of ideal and non-ideal plasticity fluids

Ideal plasticity fluids	Non-ideal plasticity fluids
Bingham fluids	Casson fluids
$\tau = \tau_c + \eta_p \cdot \dot{\gamma}$	$\tau^{\frac{1}{2}} = (\tau_c)^{\frac{1}{2}} + (\eta_p)^{\frac{1}{2}} \cdot \dot{\gamma}^{\frac{1}{2}}$

Where  $\eta_p$  is the plastic viscosity and  $\tau_c$  is the threshold stress.

Understanding the behaviour with yield stress is crucial for industries that deal with materials such as paints, slurries, and certain food products, where the ability to flow under applied stress is important for processing and application. Rheological

testing and modelling techniques are employed to determine the yield stress and study the flow behaviour of these plastic fluids.

The figure II-3 displays the various rheogram curves of non-Newtonian fluids.

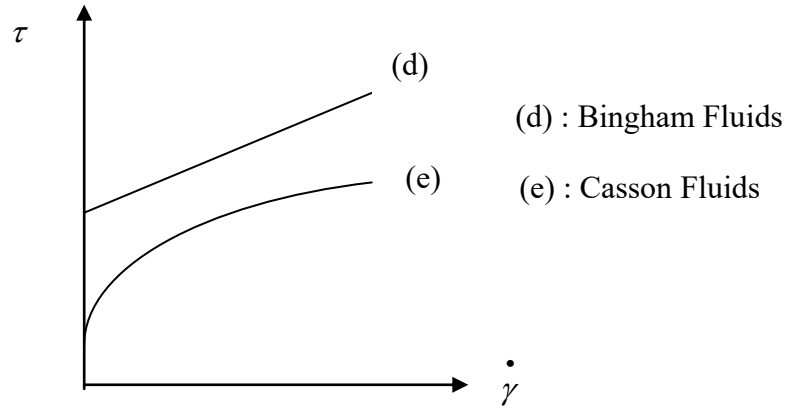


Figure II-3: Rheogram of fluids with yield stress.

Table II-3 Rheological models describing the flow of fluids with yield stress

Rheological model	Law
Bingham	$\tau = \tau_c + \eta_p \cdot \dot{\gamma}$
Herschel-Bulkley	$\tau = \tau_c + K \dot{\gamma}^n$
Casson général	$\tau^n = (\tau_c)^n + (\eta_p)^n \cdot \dot{\gamma}^n$
Casson	$\tau^{\frac{1}{2}} = (\tau_c)^{\frac{1}{2}} + (\eta_p)^{\frac{1}{2}} \cdot \dot{\gamma}^{\frac{1}{2}}$

### ***Thixotropic behaviour***

When a material has been at rest for an extended period, and a sudden load (stress) is applied and maintained constant, a fascinating phenomenon known as thixotropy occurs. Thixotropic materials exhibit a decrease in viscosity over time during the application of the load. This behaviour can be attributed to the disruption of the material's initial structure as a result of flow, causing the breaking of interatomic bonds.

As a consequence, energy is developed between molecules, which significantly impact the viscosity of the material. What makes thixotropic behaviour particularly intriguing is that, after the stress is removed and the material is allowed to rest for a sufficiently long period, it often demonstrates the ability to regain its initial properties. This phenomenon indicates the remarkable ability of the material to reconstitute its microstructure, restoring its original characteristics.

Thixotropic materials are encountered in various applications, including paints, inks, certain gels, and many other substances where their time-dependent flow behaviour plays a crucial role. Understanding and studying thixotropic behaviour are essential for optimizing the performance and stability of such materials in practical applications.

In this case, we refer to the phenomenon as thixotropy, which is illustrated in Figures II-4 and II-5.

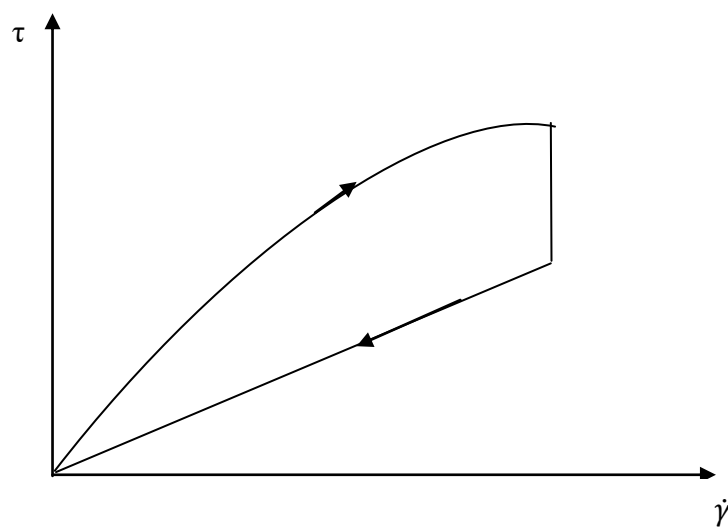


Figure II-4: Rheogram recorded by a rheometer with imposed shear rate

Figure II-4 demonstrates the time-dependent decrease in viscosity as the material is subjected to shear stress over a specific duration. The viscosity gradually decreases, indicating the fluidity of the material and its ability to flow more easily with continued agitation.

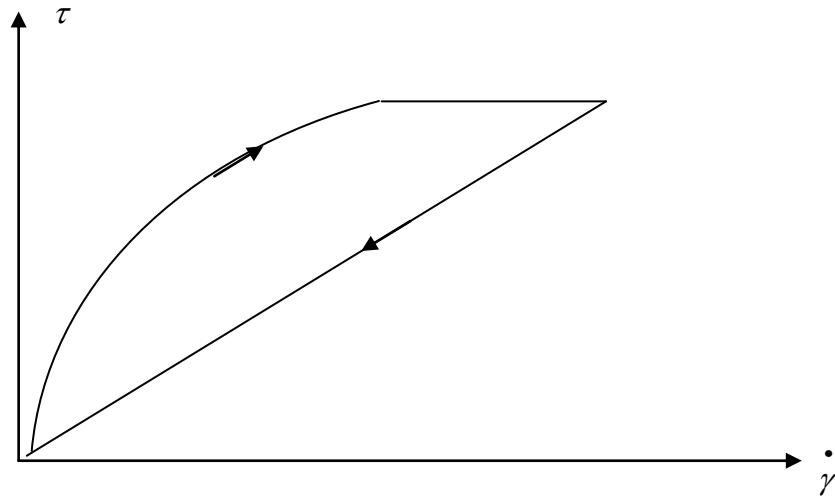


Figure II-5: Rheogram recorded by a rheometer with imposed stress

Figure II-5 further highlights the thixotropic behaviour by depicting the recovery of viscosity after the stress or agitation is removed. The material, upon being allowed to rest for a sufficient period, gradually restores its original viscosity, indicating the reformation of its internal structure.

## Chapter III : Rheology of Fresh Concrete

### III.1 Introduction

The rheology of fresh concrete is a relatively young science. Indeed, concrete that falls under such an approach, that is, concrete with sufficient fluidity, has only been used in recent times. Moreover, for a long time, a technological approach was sufficient, which involved subjecting a concrete sample to more or less controlled stress and deriving an index (slump height, flow time, compactness, etc.) to classify mixtures in terms of workability. The fact that the rankings obtained from different tests do not coincide with each other demonstrates the limitations of such an approach.

However, in recent years, there has been a growing recognition of the need for a more comprehensive understanding of the rheological behaviour of fresh concrete. This involves studying the flow properties, deformability, and interaction of its constituents, such as cement, aggregates, water, and admixtures. By delving into the rheological aspects, a deeper insight into the material's workability, pumping ability, and resistance to segregation can be gained.

The development of sophisticated measurement techniques, such as rheometers and slump flow tests, has allowed researchers to explore the rheological properties of fresh concrete in a more systematic and quantitative manner. This has paved the way for the formulation of rheological models and parameters that can better characterize and predict the flow behaviour of concrete.

Understanding the rheology of fresh concrete is of utmost importance in the construction industry. It aids in optimizing concrete mix designs, improving construction processes, and ensuring the durability and performance of the final structures. By considering the rheological aspects, engineers and concrete technologists can make informed decisions regarding the selection of materials, mixture proportions, and construction techniques to achieve desired workability and quality.

Empirical methods and tests are commonly employed to evaluate the rheological properties of fresh concrete. These include slump tests, flow table tests, and V-funnel tests, among others. These tests provide practical indicators of workability, such as slump measurements and flowability observations. However, they do not provide a comprehensive understanding of the underlying rheological mechanisms and cannot capture the complete rheological behaviour. In recent years, efforts have been made to develop more advanced rheological measurement techniques and modelling approaches

to overcome the limitations of empirical methods. Rheometers equipped with rotational or oscillatory shear devices have been used to study the flow and deformation characteristics of fresh concrete under controlled conditions. These measurements enable the determination of rheological parameters, such as shear stress, shear rate, and viscosity, which contribute to a more quantitative and scientific understanding of concrete rheology.

Furthermore, the development of rheological models, such as the Bingham model, Herschel-Bulkley model, and various other viscoplastic models, has provided a framework to describe and simulate the complex rheological behaviour of fresh concrete. These models consider factors such as yield stress, plastic viscosity, and thixotropy, which are essential for capturing the flow and deformation characteristics of concrete.

### III.2 Fresh Concrete Behaviour

The Bingham model is the most commonly used model to simulate the rheological behaviour of fresh concrete due to its simplicity and the small number of parameters required for adjustment (yield stress  $\tau_0$  and plastic viscosity  $\mu_{pl}$ ).

According to the Bingham model, fresh concrete behaves as a yield stress fluid, meaning it does not flow until a certain stress, known as the yield stress ( $\tau_0$ ), is exceeded. Once the yield stress is surpassed, the concrete begins to flow, and its rheological behaviour can be described by a linear relationship between shear stress and shear rate.

$$\dot{\gamma} = 0 \text{ si } \tau < \tau_0$$

$$\tau = \tau_0 + \mu_{pl} \cdot \dot{\gamma} \text{ si } \tau > \tau_0$$

The yield stress ( $\tau_0$ ) corresponds to the minimum stress that must be exceeded to initiate flow. It can be interpreted physically as the presence of intergranular friction.

When the applied stress ( $\tau$ ) is below the yield stress ( $\tau_0$ ), the concrete does not flow ( $\dot{\gamma} = 0$ ). Once the stress exceeds the yield stress, the concrete starts to flow, and the relationship between the stress ( $\tau$ ) and the strain rate ( $\dot{\gamma}$ ) is described by the second equation. The plastic viscosity ( $\mu_{pl}$ ) represents the resistance to flow once the yield stress is surpassed.

By using the Bingham model, engineers and researchers can predict the flow behaviour of fresh concrete under different conditions, such as during pumping, casting,

or formwork filling. This information is valuable in optimizing concrete mix designs and ensuring proper workability and pumpability of the material.

It is important to note that the Bingham model is a simplification and does not capture all the complexities of fresh concrete behaviour. The actual rheological properties of concrete can be influenced by various factors, including the properties of the cementitious materials, water-to-cement ratio, admixtures, aggregate characteristics, and mixing procedures. Therefore, it is common to adjust the Bingham model parameters based on experimental data specific to a particular concrete mixture.

In recent years, more advanced rheological models have been developed to better capture the non-linear and time-dependent behaviour of fresh concrete. These models consider additional parameters, such as thixotropy (time-dependent recovery of viscosity) and shear thinning (decrease in viscosity with increasing shear rate), which can be observed in certain concrete mixtures.

Overall, understanding the behaviour of fresh concrete is crucial for ensuring its workability and flowability during construction operations. The Bingham model, although simplified, provides a practical framework for modelling the rheological properties of fresh concrete and serves as a valuable tool in concrete mix design and construction process optimization. Ongoing research continues to refine and expand our knowledge of fresh concrete rheology, leading to more accurate and comprehensive models in the future.

By incorporating rheological testing methods into the quality control process, concrete producers can gain a better understanding of the fresh concrete's flow properties and make more informed decisions regarding mix design, production parameters, and construction processes. This can ultimately lead to improved concrete performance, enhanced workability, and optimized construction operations.

### **III.3 Characterization and Control Tests for Fresh Concrete**

#### **III.3.1 Slump Test (Abrams Cone Test)**

The slump test, also known as the Abrams cone test, is a standardized test method (ASTM C 143 or NFP 18-451). It involves filling a truncated cone mold with three successive layers of concrete (approximately 6 liters in total), compacting each layer with 25 strokes. The cone is then removed, and the slump, or settlement, is measured at the original center of the upper surface of the sample (see Figure III-1, Figure III-2).

The slump value can range from a few millimeters or even zero for very dry concrete (such as road concrete) to about twenty centimeters for highly fluid concrete.

The simplicity and ease of the slump test have made it widely used both on construction sites and in laboratories, providing a quick assessment of workability. However, a major drawback of this test is that concrete samples with the same slump value can exhibit significantly different rheological behaviours. In other words, the relationship between slump and workability is not always straightforward. This can lead to issues of varying severity on construction sites.

Currently, the slump test is commonly used to monitor the quality of concrete from one batch to another with a fixed mix design. Any abnormal variation in slump may indicate an unexpected change in formulation or materials. However, for projects involving highly advanced techniques, it has been found that the slump measurement may not be sensitive enough. Moreover, when it comes to analyzing the causes of variation, the slump test demonstrates its limitations once again.

To overcome the limitations of the slump test and gain a deeper understanding of the rheological properties of fresh concrete, more sophisticated testing methods such as rheometry and dynamic testing have been developed. These methods provide comprehensive rheological data, including shear stress-shear rate relationships, yield stress, and thixotropic behaviour, allowing for a more accurate assessment of concrete's flow characteristics and its response to external forces.

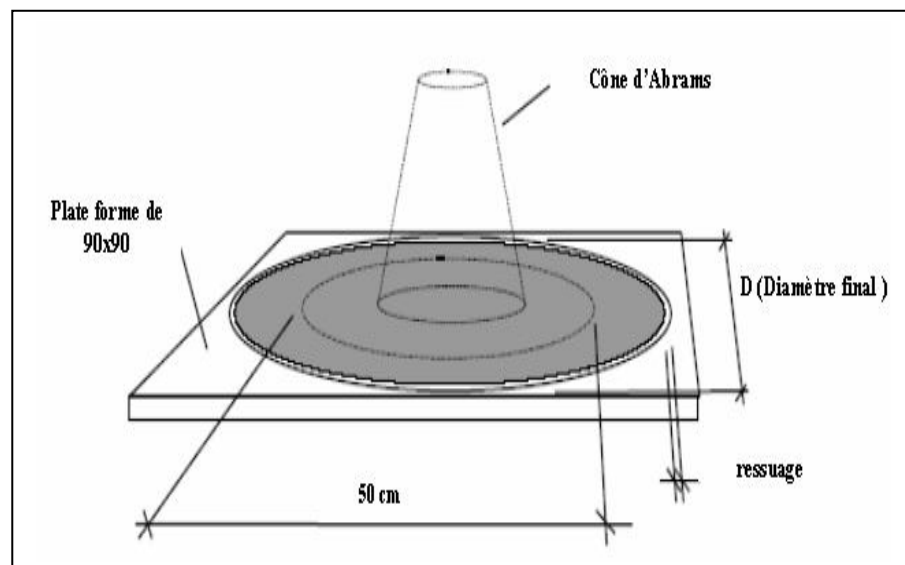


Figure III-1: Schematic representation of the spreading test.



Figure III-2: Spread test using the Abrams cone

### III.3.2 Sieve Stability Test

The sieve stability test is conducted to assess the risk of segregation in self-compacting concrete (SCC). It can be used during the formulation phase of SCC in laboratory studies or for on-site evaluation of concrete stability. This test complements other tests that evaluate the mobility of concrete in confined or non-confined conditions by specifically characterizing its stability.

The test involves using a 10-liter bucket with a lid, a 315 mm diameter sieve with a 5 mm opening, and a weighing scale with a minimum capacity of 20 kg and a precision of 20 g.

According to the recommendations of the French Association for Civil Engineering (AFGC), the procedure is as follows: After the mixing process, ten liters of concrete are poured into the bucket. After fifteen minutes, a 4.8 kg sample is poured from the bucket onto the sieve. Two minutes later, the amount of mortar (bleed water) that has passed through the sieve is weighed. The percentage by weight of the bleed

water relative to the sample weight gives the segregation index ( $\pi$ ), which is used to classify SCC mixtures as follows:

$$\pi = (\text{bleed water weight} \times 100) / \text{sample weight}$$

Where:

Bleed water weight = (weight of sieve with bleed water) - (weight of sieve)

Sample weight = (weight of sieve with bleed water) - (weight of empty sieve)

$0\% \leq \pi \leq 15\%$	Satisfactory stability
$15\% < \pi \leq 30\%$	Critical stability, retesting required on-site
$\pi > 30\%$	Very poor stability, concrete unusable

Table III-1 Recommended Values for AFGC Tests

Test	Recommended Range
Slump Test	60 cm to 75 cm
H <sub>2</sub> /H <sub>1</sub> Ratio	≥ 0,8
Sieve Stability Test (segregation index $\pi$ )	≤ 15%



Figure III-3: Sieve Stability Test

The sieve stability test is conducted to assess the risk of segregation in self-compacting concrete (SCC). It can be used during the formulation phase of SCC in laboratory studies or for on-site evaluation of concrete stability. This test complements other tests that evaluate the mobility of concrete in confined or non-confined conditions by specifically characterizing its stability. The test involves using a 10-liter bucket with a lid, a 315 mm diameter sieve with a 5 mm opening, and a weighing scale with a minimum capacity of 20 kg and a precision of 20 g.

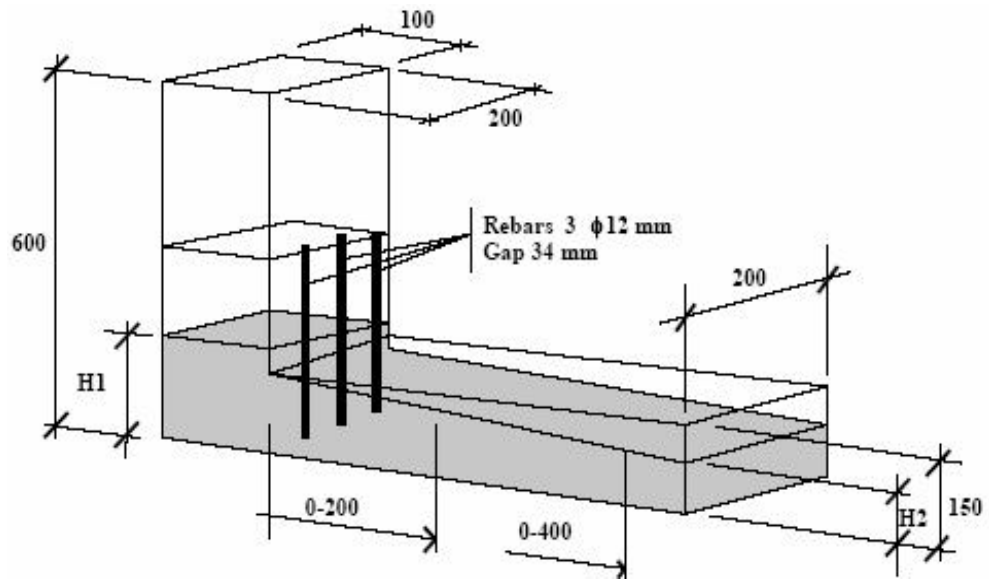
### **III.3.3 L-Box Test**

The L-Box test is used to assess the flowability of concrete in confined spaces and to verify that the concrete placement will not be hindered by undesirable blocking phenomena.

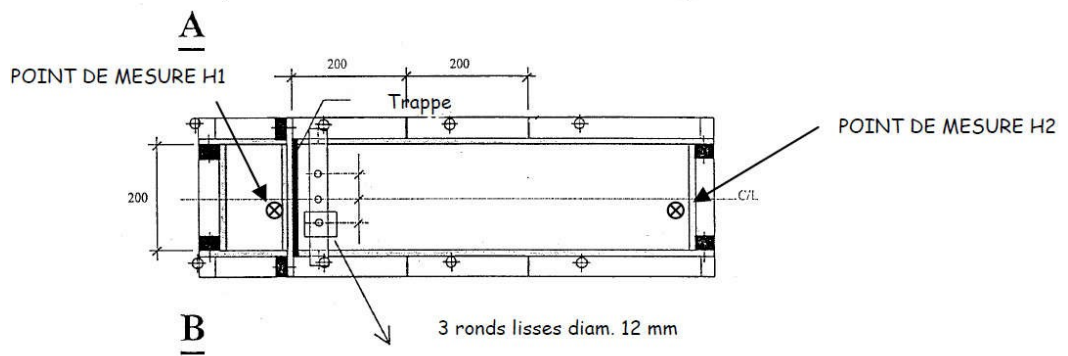
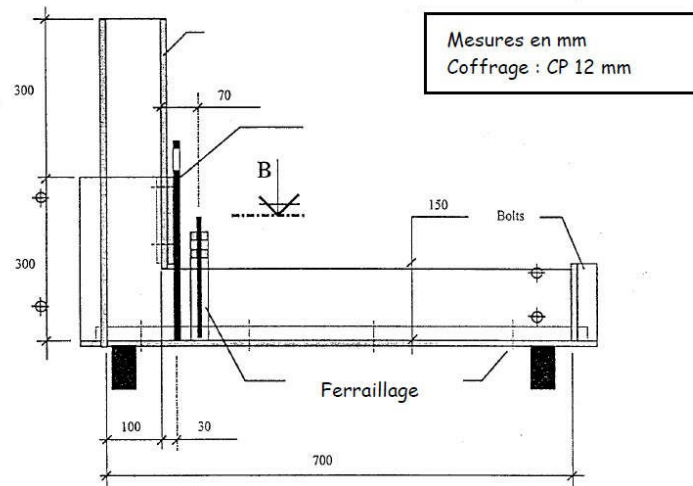
The procedure, as outlined by the [AFGC], is as follows:

- ❖ The vertical section of the L-Box is completely filled with concrete (approximately 13 liters in volume).
- ❖ After levelling the surface, the concrete is left undisturbed for one minute.
- ❖ The trap door is then lifted, allowing the concrete to flow into the horizontal section of the box through the reinforcement bars. The clear spacing between the bars is 39 mm.
- ❖ Once the concrete stops flowing, the heights H1 and H2 are measured, and the result is expressed as the filling ratio  $H2/H1$ .
- ❖ When the concrete flows poorly through the reinforcement bars and there is accumulation of aggregates downstream of the grid, it indicates a blocking or segregation issue. (see Figure III-4 a – b and Figure III-5)

The L-Box test provides insights into the ability of the concrete to flow through restricted spaces and helps identify potential issues such as blockage or excessive segregation. If the concrete exhibits poor flowability and aggregates accumulate downstream of the reinforcement bars, it indicates the need for adjustments in the mix design or placement technique to ensure proper concrete placement.



(a)



(b)

Figure III-4: Schematic representation of L-box test (a) and (b)



Figure III-5: Realisation of L-box test

### III.3.4 V-Funnel Test

The V-Funnel test is used to assess the viscosity of concrete by measuring the flow time of a concrete sample passing through the opening of a funnel. The flow time provides an indication of the concrete's viscosity. A longer flow time suggests a higher viscosity, indicating a more viscous concrete, while a shorter flow time suggests a lower viscosity, indicating a less viscous concrete.

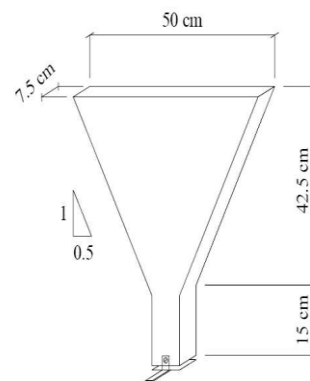


Figure III-6: V-funnel Test

The procedure involves the following steps:

- ❖ The V-Funnel apparatus is prepared, consisting of a funnel with a specific opening diameter and a cylindrical container at the bottom.
- ❖ The funnel is filled with a specified volume of concrete.
- ❖ The flow time is measured by releasing the concrete from the funnel and timing how long it takes for the entire sample to flow out through the opening.
- ❖ The measured flow time is then used to evaluate the flowability and viscosity of the concrete.

The V-Funnel test provides valuable information about the flow characteristics of concrete and helps in assessing its workability and ease of placement. By measuring the flow time, it allows for the determination of the concrete's viscosity, which is an important parameter in various construction applications.

The European guide on SCC (Self-Compacting Concrete) provides a classification of SCC based on the flow time in the V-Funnel test and the intended application.

Two classes are defined:

VF1 Class: Flow time  $\leq 8$  s

VF2 Class: Flow time  $\geq 9$  s and  $\leq 25$  s

In this classification, a longer flow time or flow discontinuity indicates blockage and segregation issues in the concrete. These phenomena can occur in two different cases: when the concrete is highly viscous or when the concrete exhibits segregation. In the case of segregation, a bridge of coarse aggregates forms at the lower opening of the funnel, blocking the flow of concrete.

The V-Funnel test, along with the classification system, provides valuable guidance for assessing the flow properties and segregation resistance of SCC. It helps in selecting the appropriate SCC mix design for specific applications, ensuring that the concrete can flow easily and uniformly without blockages or segregation issues.

### **III.4 Rheology and Construction Processes**

The rheology of fresh concrete has implications for various construction processes, including pumping, formwork filling, and casting. Understanding the flow behaviour and rheological properties of concrete is crucial for designing appropriate pumping systems, ensuring uniform filling of formwork, and preventing segregation. Rheological measurements, such as those obtained from slump tests or rheometers, aid in quality control and process optimization during concrete placement.

### **III.5 Challenges and Future Directions**

Despite advancements in fresh concrete rheology, challenges remain, particularly in understanding and controlling the flow behaviour of complex concrete mixtures and specialized applications. Developing more accurate rheological models and measurement techniques, as well as studying the influence of supplementary cementitious materials and fibers, are areas of ongoing research. Improving the compatibility between fresh and hardened concrete properties is another important aspect for future development.

The rheology of fresh concrete is a crucial factor in achieving desired workability, homogeneity, and performance of the hardened material. Proper measurement techniques and rheological models help in characterizing and predicting the flow behaviour of fresh concrete.

## Chapter IV : Rheology of Granular Media and Polymers

### IV.1 Rheology of Granular Media

#### IV.1.1 Introduction

Granular materials are of major interest in various industrial sectors such as cosmetics, pharmaceuticals, food processing, and construction materials. Depending on the circumstances, their behaviour can resemble that of a solid, liquid, or gas. Therefore, their handling and processing are strongly influenced by their rheological properties, which primarily depend on the physicochemical and geometric characteristics of the grain surfaces, the volumetric fraction of the samples, the distribution of free volume, the intergranular pressure, and the mode of stress transmission (elastic, frictional, or collisional) that results from these factors. These properties are closely related to the spatial configuration adopted by the grains when they are arranged within characterization devices. As a result, there is significant variability in measurements due to the multitude of possible configurations. Consequently, the intrinsic characterization of a powder is a challenging task that requires averaging over all accessible configurations. Hence, only a few techniques are currently available to establish relationships between the global rheological properties of powders and the local structural characteristics of the constituent grains.

Efforts to understand and control the rheology of granular materials are vital for optimizing their performance in various applications. However, further research is needed to develop more advanced techniques and methodologies for accurately characterizing and predicting the rheological behaviour of granular media based on their microstructural properties. Such advancements would enable better design and processing strategies, leading to enhanced performance and efficiency in industrial processes involving granular materials.

#### IV.1.2 Granular Material

A granular material is a collection of macroscopic solid particles, typically larger than 100 $\mu\text{m}$ , that interact through inelastic collisions and frictional contacts. This commonly accepted definition excludes soft and fragile objects, making granular materials a distinct subset within the broader category of divided media. The size criterion is related to the dominant interactions. When thermal agitation and surface

forces (such as van der Waals or electrostatic forces) become significant, the term "fine powders" or "colloids" is more appropriate.

Granular materials are typically composed of a large number of particles (e.g., around 600,000 particles in a teaspoon of sugar). They are also characterized by a wide size distribution that can span several orders of magnitude. For example, in a rockslide along a mountainside, large rock blocks coexist with numerous small debris. Additionally, granular particles exhibit a variety of shapes and surface properties. They can be angular or elongated to different degrees, and their surfaces can be rough or smooth. The mechanical behaviour of an individual particle can be complex due to its internal structure. However, in this context, we will consider only rigid particles, and their interactions will be simplified to dry friction and impenetrability.

The mechanical behaviour of granular materials is governed by interactions between individual particles. These interactions are influenced by factors such as particle size, shape, and surface characteristics, as well as external forces and boundary conditions. When subjected to external loading, granular materials can display behaviours reminiscent of solids, liquids, or even gases, depending on the applied stress and the overall arrangement of particles.

Understanding the rheology of granular materials involves studying their flow and deformation characteristics under different conditions. The behaviour of granular materials can exhibit non-linearities, discontinuities, and complex transitions between solid-like and fluid-like states. These phenomena arise from a combination of interparticle friction, contact forces, and the collective response of the particle assembly.

Characterizing the rheological properties of granular materials presents challenges due to the inherent variability in particle arrangements and interactions. The behaviour of a granular material is highly dependent on factors such as particle size distribution, packing density, compaction history, and external constraints. Therefore, studying granular materials requires careful experimental techniques, numerical simulations, and theoretical models to capture their complex behaviour.

Granular materials find applications in numerous industries, including civil engineering, geotechnical engineering, pharmaceuticals, food processing, and many others. Understanding their rheological properties is crucial for optimizing processes involving handling, transport, mixing, and storage of granular materials, as well as predicting their behaviour in various industrial and natural contexts.

Two glasses, one filled with water and the other with semolina, are sufficient to illustrate the particularity of this state of matter. When pouring semolina into a glass, it takes the shape of the container. At first glance, it may seem that granular matter behaves like a liquid, or at least possesses analogous properties. However, upon closer observation, it becomes apparent that unlike the surface of water, the surface formed by the grains is not flat. If the semolina is then poured onto a surface, it flows and forms a pile, with grains rolling, some dispersing, but eventually settling into a distinct angled heap. This ability of granular materials to form slopes is familiar to us. We can observe it in nature through debris deposits, dunes, and ripples on beaches. It is an essential property that distinguishes granular materials from fluids, as they can bear deviatoric loads that tend to deform the material without undergoing deformation themselves, akin to solids. Thus, sand, which we enjoy treading upon, resists easy classification into the traditional states of matter. Due to the richness of their behaviour and their response to various stimuli, can we say that granular materials behave like solids, liquids, or gases?

The complex nature of granular materials' behaviour defies a rigid classification as a solid, liquid, or gas. When examining the characteristics of granular materials such as semolina, we can observe aspects that resemble properties of liquids, solids, and even gases.

When semolina is poured into a glass, it takes the shape of the container, giving the impression of liquid-like behaviour. However, unlike the flat surface of water, the surface formed by the semolina grains is irregular. When semolina is poured onto a surface, it flows and forms a heap with a characteristic angle. This ability of granular materials to form slopes is a distinct property from fluids, yet it differs from solid behaviour as there is no permanent deformation of the material.

Granular materials can also exhibit behaviours similar to gases. When subjected to external forces, semolina grains can disperse and roll, much like gas molecules in random motion. Additionally, granular materials may display dilatancy or contraction effects when compressed or released, resembling the compressibility of gases.

Due to the richness of behaviours and responses to different stimuli, it is challenging to reduce granular materials to a single category. They possess unique characteristics that are not fully captured by the properties of solids, liquids, or gases. Understanding granular materials requires a specific approach that considers their complex nature and unique particle interactions.

In recent years, extensive research has shed further light on the complex behaviour of granular materials, revealing their unique and fascinating characteristics. Granular materials are not easily classified within traditional states of matter such as solids, liquids, or gases. Instead, they exhibit a rich variety of behaviours that defy simple categorization.

These materials are prevalent in numerous industrial sectors, playing a crucial role in processes ranging from mineral extraction and construction to chemical manufacturing and pharmaceutical production. The manipulation of granular substances requires an understanding of their flow properties, packing behaviour, and response to external forces.

Moreover, granular materials are also integral to many geophysical phenomena, providing insights into natural processes such as dune formation, landslides, and volcanic flows. Their study contributes to the prediction and mitigation of natural hazards, ensuring the safety of human settlements in landslide-prone areas and enhancing our understanding of planetary dynamics.

Despite their ubiquity and significance, granular materials pose unique challenges for characterization and modelling. The behaviour of individual grains, their shape, size distribution, and interparticle interactions all influence the collective behaviour of the material. This complexity demands innovative approaches and interdisciplinary research to unravel the fundamental principles governing granular systems.

Advances in experimental techniques, numerical simulations, and theoretical modelling have led to significant progress in understanding granular materials. However, many questions remain unanswered, and ongoing research continues to unveil new insights into their behaviour.

By studying the rheology of granular materials, researchers aim to establish connections between their microstructural properties and macroscopic behaviour. This interdisciplinary field combines principles from physics, materials science, engineering, and geophysics to unlock the secrets of these intriguing materials and harness their potential in various applications.

In summary, granular materials defy conventional classification and exhibit a wide range of behaviours that bridge the gap between solids, liquids, and gases. They play vital roles in industrial processes and geophysical phenomena, offering both opportunities and challenges for further exploration. Understanding the unique

properties of granular materials is crucial for advancing our knowledge across diverse scientific disciplines and improving practical applications in various industries.

Another field where granular media are ubiquitous is geophysics, as soil is primarily composed of grains. Among the situations involving divided media are:

- ✓ Dunes
- ✓ Rock avalanches
- ✓ Landslides
- ✓ Pyroclastic flows during volcanic eruptions (Fig. 1.1b)
- ✓ Snow avalanches (snow being a granular medium with unique phase-change characteristics)
- ✓ Saturn's rings, which are formed by large blocks of ice.

Describing these events and predicting disasters such as landslides requires a good understanding of the flow properties of these particle-based media.

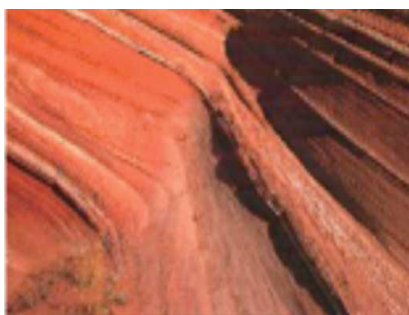


Figure IV-1: Overview of the diversity of granular media

## **IV.1.3 Rheology of Granular Media**

### ***Introduction:***

Granular materials, composed of discrete solid particles, are abundant in nature and play a crucial role in various industrial processes. Understanding their mechanical behaviour, particularly their rheology, is essential for predicting their flow properties, stability, and designing efficient processing techniques. This paper provides an overview of the rheological characteristics of granular media, exploring the interparticle interactions, stress-strain relationships, and flow regimes. Additionally, it discusses key experimental and theoretical approaches used to study granular rheology and highlights their significance in practical applications.

### ***Rheological behaviour of granular media***

**Stress-Strain Relationships** Rheological behaviour in granular media is often described by stress-strain relationships, which relate the applied stress to the resulting deformation. The stress distribution within granular assemblies is typically non-uniform, leading to complex force chains and localized regions of high stress. The response of granular materials under different loading conditions, such as compression, shear, and extension, exhibits distinct characteristics that contribute to their overall rheological properties.

**Flow Regimes** Granular materials can flow in various regimes, including dense flow, shear flow, and dilute flow, each exhibiting unique characteristics. Dense flow refers to the movement of tightly packed particles, while shear flow involves the sliding of particles along each other. Dilute flow occurs when particles are widely dispersed, resembling a fluid-like behaviour. Understanding these flow regimes and their transitions is crucial for predicting the flow behaviour of granular materials in different applications.

### ***Experimental Techniques***

A wide range of experimental techniques has been developed to investigate the rheology of granular materials. These techniques include direct shear tests, triaxial tests, and rotational rheometry, among others. Each method offers unique insights into the mechanical behaviour of granular systems, enabling the measurement of parameters such as shear strength, flowability, and bulk density.

Theoretical Models In addition to experimental approaches, various theoretical models have been proposed to describe granular rheology. These models, such as the Mohr-Coulomb and Drucker-Prager models, provide mathematical frameworks to predict the stress-strain behaviour and flow characteristics of granular materials. Understanding these theoretical models enhances our ability to analyze and engineer granular systems in practical applications.

### ***Practical Applications***

The rheological properties of granular materials have significant implications in numerous industries, including pharmaceuticals, agriculture, civil engineering, and mining. By understanding and controlling granular rheology, engineers and scientists can optimize processes, improve product quality, and enhance safety in a wide range of applications.

Rheology of granular media, covering the interparticle interactions, stress-strain relationships, flow regimes, experimental techniques, theoretical models, and practical applications. A comprehensive understanding of granular rheology is crucial for effectively dealing with the challenges and opportunities presented by granular materials in various fields.

### ***Mechanisms of Blockage***

Granular media often exhibit complex flow behaviour due to the presence of blockages within the material. Blockages occur when particles become trapped or jammed, impeding the flow of neighboring particles. Several mechanisms contribute to blockage formation, including arching, clogging, and segregation. Arching happens when particles form stable bridges or arches, redistributing the applied stress and reducing the flow. Clogging occurs when particles accumulate and obstruct the flow path, leading to localized regions of high resistance. Segregation refers to the separation of particles based on size or density, which can result in the formation of regions with different flow properties.

Correlations of Particle Movements Understanding the correlations between particle movements are crucial for predicting and analyzing the flow behaviour of granular media. Granular flows often exhibit spatial and temporal correlations, meaning that the movement of one particle affects the behaviour of its neighboring particles. These correlations can arise due to various factors, including interparticle forces, contact dynamics, and collective particle interactions. The study of particle

correlations provides insights into the development of flow patterns, velocity fluctuations, and the overall flow dynamics of granular systems.

### ***Experimental Techniques for Blockage Analysis***

To study the mechanisms of blockage and the role of boundaries in granular media, several experimental techniques are commonly employed. These include flow visualization methods, particle tracking techniques, and imaging technologies such as X-ray tomography. These techniques allow researchers to observe and analyze the formation and evolution of blockages, particle movements, and interactions near boundaries. Experimental data combined with theoretical models provide valuable insights into the fundamental understanding of granular rheology in the presence of blockages and confinement.

The understanding of the rheology of granular media, including mechanisms of blockage, correlations of particle movements, and the role of boundaries, has significant practical implications. It enables engineers and scientists to develop more efficient processes for handling and transporting granular materials in industries such as mining, agriculture, and construction. Furthermore, further research in this field is essential to advance our understanding of granular systems, refine existing models, and develop new approaches for controlling and manipulating granular flows.

The rheology of granular media is influenced by various factors, including mechanisms of blockage, correlations of particle movements, and the role of boundaries. These aspects contribute to the complex flow behaviour observed in granular systems. By studying and comprehending these phenomena, we can enhance our ability to predict and control the flow properties of granular materials, leading to improved industrial processes and applications.

### **IV.1.4 Granular Plasticity and Internal Variables**

Granular materials exemplify the challenge of identifying a constitutive law that can capture the majority of observed phenomena. Sand, or any granular material, exhibits a pronounced plastic behaviour. The flow of sand results solely from the relative displacements of grains in response to external forces. The mechanical behaviour of the material emerges from the exchange of momentum and dissipation of energy at the contacts, which mark the mutual exclusion of particles. Despite this simple and accurate depiction of plasticity in granular media, no model has yet succeeded in proposing a constitutive law that fully captures it, due to

the extraordinarily rich behaviour that emerges from the structure of the particle packing, known generally as texture.

The difficulty lies in the fact that the macroscopic constitutive law lacks an equivalent at the microscopic scale where individual particles are considered. To bridge the gap between scales, one must smooth out behaviours that are not only heterogeneous and non-stationary but also contact laws between rigid grains that have the characteristic of being non-regular.

The impenetrability of particles and the disordered nature of grain packings result in significant heterogeneity within the material. This heterogeneity is characterized firstly by variations in stress within the packing. Chains of force are observed numerically and experimentally, revealing the presence of force transmission pathways through the particle network.

Additionally, the heterogeneous distribution of contacts and interparticle forces leads to non-uniform deformation and strain localization within the granular material. This localization can manifest as shear bands, where the material undergoes intense shearing and deformation while the surrounding regions remain relatively undisturbed.

The existence of internal variables, such as fabric anisotropy and stress history, further complicates the plastic behaviour of granular materials. These internal variables influence the material's response to external loading and deformation, introducing additional complexity into the constitutive modelling.

Overall, the plasticity of granular materials arises from the intricate interplay between contact interactions, particle rearrangements, and the collective behaviour of the particle assembly. Capturing the complete range of behaviours exhibited by granular materials remains a formidable challenge, necessitating ongoing research and the development of advanced constitutive models that can effectively capture the rich and complex behaviour observed in these materials.

Understanding the behaviour of granular materials and developing accurate constitutive models is crucial for various engineering and geophysical applications. Ongoing research focuses on elucidating the microstructural origins of plasticity, exploring the role of particle shape and size distribution, and developing more sophisticated constitutive models that can capture the rich behaviour and heterogeneity observed in granular materials.

## **IV.2 Rheology of Polymers**

### **IV.2.1 Introduction**

Polymers, with their unique molecular structure and diverse applications, exhibit complex rheological behaviour. Understanding the rheology of polymers is crucial for designing and optimizing processes in industries such as plastics, rubber, coatings, and biomedical materials. This paper provides an overview of polymer rheology, including the fundamental principles, rheological characterization techniques, and the influence of molecular structure on polymer flow behaviour. Additionally, it explores the significance of polymer rheology in practical applications and highlights the challenges and future directions in the field.

### **IV.2.2 Molecular Structure and Rheological Behaviour**

The rheological properties of polymers are strongly influenced by their molecular structure. Factors such as polymer chain length, branching, molecular weight distribution and the presence of additives or fillers affect their flow behaviour. Understanding the relationships between molecular structure and rheological behaviour is essential for predicting and manipulating the flow characteristics of polymer materials.

**Viscoelasticity of Polymers** Polymers exhibit both viscous and elastic behaviour, known as viscoelasticity, due to the molecular relaxation processes occurring during deformation. Viscoelastic behaviour is characterized by time-dependent responses, such as stress relaxation, creep, and stress overshoots. The understanding of viscoelasticity is vital for various polymer processing operations, such as extrusion, injection molding, and film casting.

### **IV.2.3 Rheological Characterization Techniques**

A variety of experimental techniques are used to characterize the rheological behaviour of polymers. These include shear rheology, oscillatory rheology, extensional rheology, and dynamic mechanical analysis. Each technique provides unique insights into different aspects of polymer flow, such as viscosity, shear thinning behaviour, storage and loss moduli, and melt elasticity. The choice of characterization technique depends on the specific application and the desired rheological parameters.

#### **IV.2.4 Polymer Rheology in Applications**

The rheology of polymers has significant implications in various industries. In the plastics industry, polymer rheology affects the processability, mechanical properties, and final product performance. In the rubber industry, rheology influences the processing of elastomers and the properties of rubber products. Additionally, polymer rheology plays a crucial role in the formulation and performance of coatings, adhesives, and biomedical materials.

The rheology of polymers is a multidisciplinary field that combines principles from polymer science, materials engineering, and fluid mechanics. A thorough understanding of polymer rheology is essential for designing and optimizing polymer processing operations and tailoring the properties of polymer-based materials for specific applications.

Chapter V : **Methods and measurement instruments:**  
**Viscometers and Rheometers**

**V.1 Measurement Methods: Rheometers**

Rheometers are advanced instruments that can measure both the viscosity and viscoelastic properties of polymer materials. They apply controlled shear or extensional deformation to the sample and measure the resulting stress, strain, and deformation behaviour. Rheometers can perform a range of tests, including shear flow, oscillatory shear, and extensional flow. They provide information on parameters such as shear viscosity, viscoelastic modulo, yield stress, and relaxation behaviour. Rheometers are widely used for characterizing the rheological properties of polymer melts, solutions, and gels. Common types of rheometers include rotational rheometers, capillary rheometers, and extensional rheometers.

**V.1.1 Different types of rheometers**

*Coaxial cylinder rheometers*

The operating principle is simple. The sample to be studied is placed between two coaxial cylinders, one fixed and the other rotating. Laminar motion is imposed by rotating one of the cylinders at a speed  $\Omega$ . The substance to be analyzed decomposes into laminar layers, each with its own velocity. The velocities range from zero (the laminar layer attached to the fixed cylinder) to  $\Omega$  (the layer attached to the cylinder rotating at a speed  $\Omega$ ). The shear velocity and shear stress depend on the specific layer being considered and its position relative to the axis of the cylinders. (see figure V-1)

We denote  $r$  as the distance or position of a layer, and we will have  $\dot{\gamma}(r)$  and  $\tau(r)$  to denote the rate at a distance  $r$  and the shear stress at a distance  $r$  from the central axis, which leads to:

$$\dot{\gamma} = r \frac{d\Omega}{dr}$$
$$\tau(r) = \frac{M}{2\pi r^2 h}$$

The shear rate is equal to:

$$r \frac{d\Omega}{dr} = \frac{1}{\eta} \frac{M}{2\pi r^2 h}$$

And by integration, the viscosity is given by:

$$\eta = \frac{M}{2\pi h\Omega} \left[ \frac{1}{R_1^2} - \frac{1}{R_2^2} \right]$$

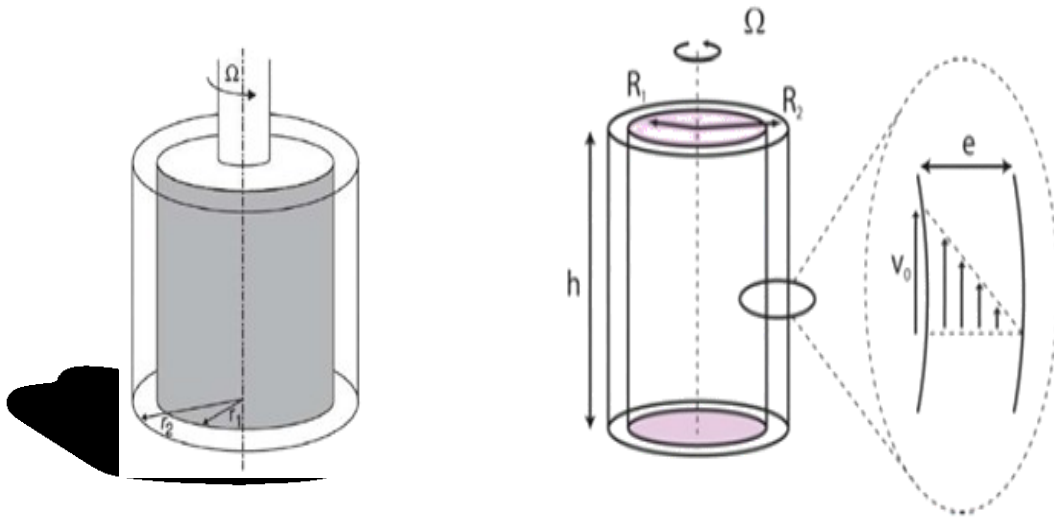


Figure V-1: Principle of coaxial cylinder rheometers

### ***Planar rotational rheometer***

This device was developed at LCPC Paris, and its geometry is of the disk-disk type. The apparatus is fully automated. It allows for the measurement of dilatancy during testing and also enables the study of behaviour under vibration. It is designed for use with high-flow concrete (slump greater than 100 mm). The sealing joint appears to be a relatively sensitive part during friction measurement. Wall effects are negligible, and the development of mathematical equations is possible to obtain results in fundamental units. The major advantage of this device is its portability. (See figure V-2)



Figure V-2: Rheometer BT-RHEO

***Mixer-agitator rheometer: The TATTERSALL "two point test" apparatus***

The first one, the MK I, was a HOBART mortar mixer equipped with a wattmeter as a torque indicator. It featured a hook-shaped rod that rotated in a planetary motion within the concrete. The mixer had three rotational speeds. Subsequently, the apparatus was modified. A cylinder with interrupted helical blades rotated in the center of the concrete, known as the MK II. Furthermore, to measure the torque, a pressure gauge installed on the hydraulic transmission indicates the force exerted by the blades. (See figure V-3).

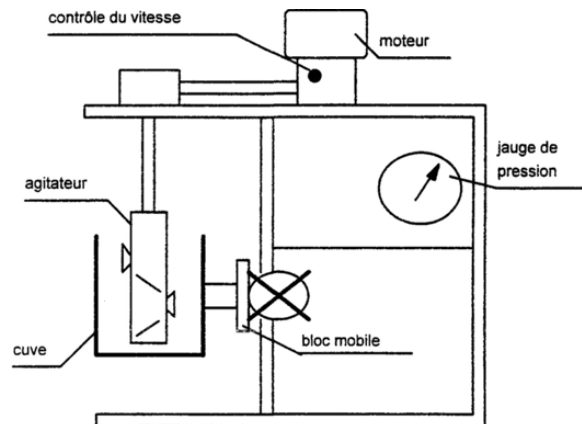


Figure V-3: Schematic representation of "two point test" apparatus

***BML - Rheometer***

Based on the MK II, this apparatus closely resembles a coaxial cylinder viscometer. Blades installed on the cylinders limit slip at the surface. This device is fully computer-controlled. Its geometry allows for the straightforward development of equations to determine rheological parameters in fundamental units. Piston flow occurs during tests for sufficiently plastic concrete sample.

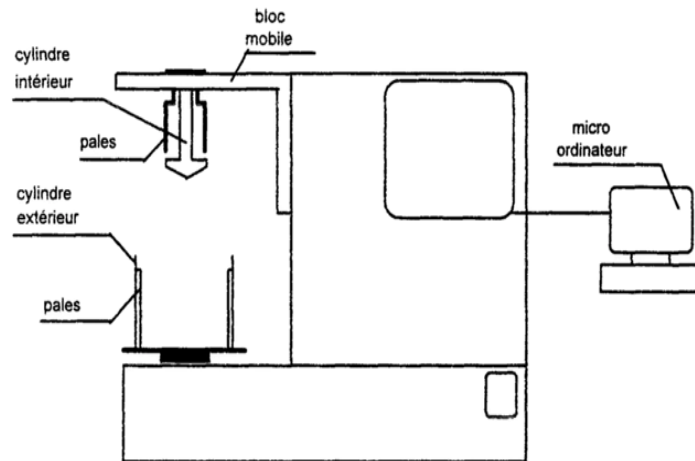


Figure V-4: Schematic representation of BML - rheometer

### ***IBB Rheometer***

The principle of this rheometer involves an agitator in the shape of an H, which rotates with a planetary motion within the concrete. A speed sensor automatically records the velocities on the computer, as well as the measured torques using a torque sensor placed on the main shaft. The test is automatically controlled by the computer. This rheometer is capable of measuring rheological parameters of low-workability concrete. (See figure V-5 and figure V-6)

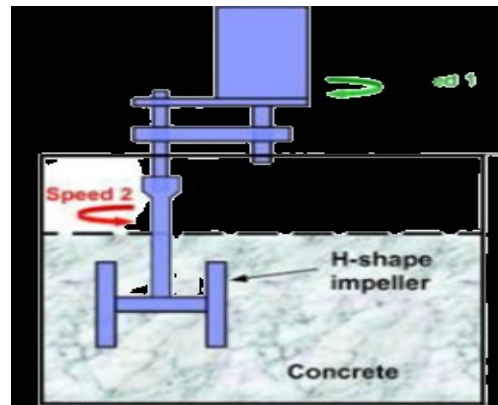


Figure V-5: Schematic representation of IBB rheometer



Figure V-6: IBB - rheometers

## V.2 Measurement Methods: Viscosimeters

Viscosimeters are commonly used instruments for measuring the viscosity of polymer melts or solutions. There are several types of viscosimeters, including capillary viscosimeters, rotational viscosimeters, and falling ball viscosimeters. Capillary viscosimeters, such as the Ostwald viscometers, determine viscosity by measuring the flow of a sample through a capillary tube under gravity or pressure. Rotational viscosimeters, such as the cone-and-plate and parallel-plate rheometers, apply shear stress to the sample and measure the resulting shear rate and viscosity. Falling ball viscosimeters use the time it takes for a ball to fall through a sample to determine its viscosity. These viscosimeters provide valuable data for understanding the flow behaviour and rheology of polymer fluids.

### V.2.1 Different types of viscosimeters

#### *Capillary viscosimeter:*

Capillary viscometers are instruments used for measuring the viscosity of fluids based on the flow rate through a capillary tube. The principle behind capillary viscometry is that the viscosity of a fluid is inversely proportional to its flow rate through a capillary under constant pressure. The capillary tube has a known length and diameter, and the time taken for the fluid to flow through the capillary is measured. By applying the Hagen-Poiseuille equation or other relevant mathematical models, the viscosity of the fluid can be determined. Capillary viscometers are commonly used in industries such as petroleum, paint, and chemical manufacturing, where precise viscosity measurements are crucial for quality control and process optimization. They provide a simple yet

effective method for characterizing the flow properties of various fluids, aiding in product development, formulation adjustments, and ensuring product consistency.

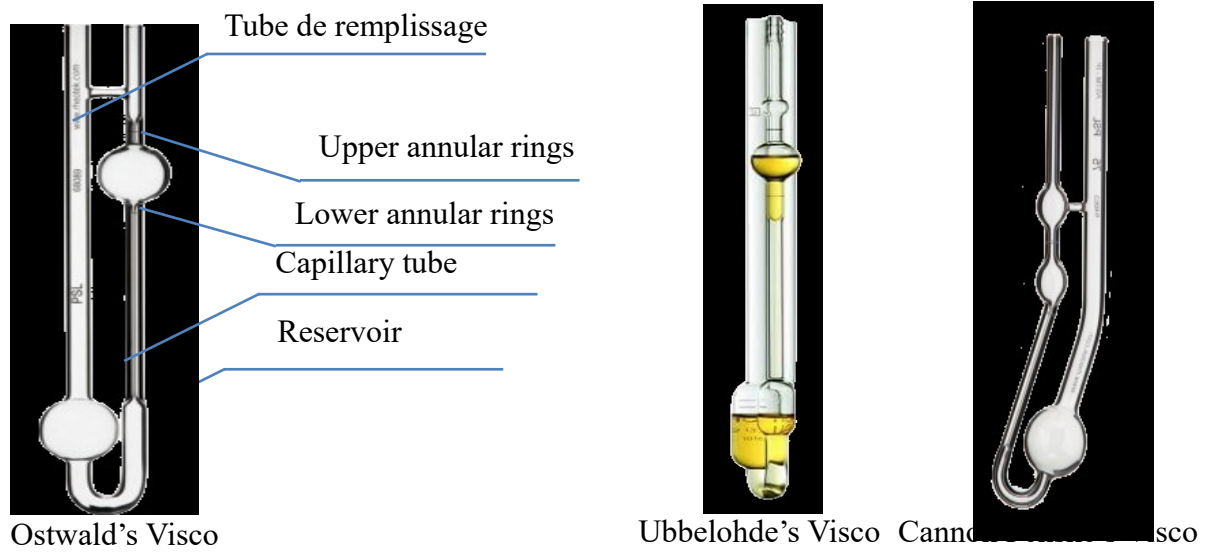


Figure V-7: Types of Capillary viscosimeters

***Rotational viscometer of cone-plate type***

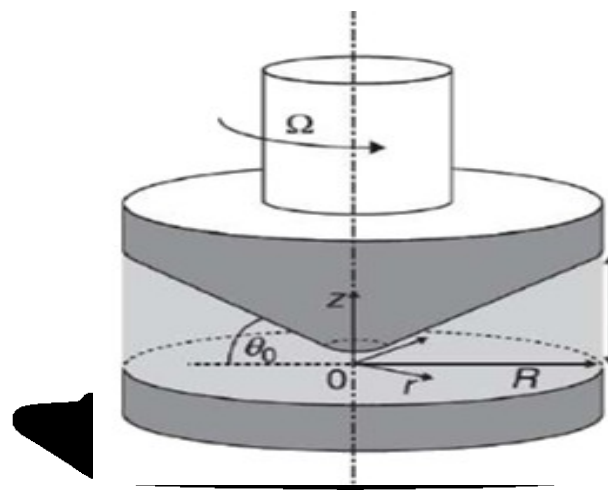


Figure V-8: Rotational viscometer of cone-plate type

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