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Viscosity Measurement by using Melt flow Index for Thermoplastic polymers

قياس اللزوجه بإستخدام جهاز معامل انصهار البوليمر للبلاستيك الحراري

A thesis Submitted in Partial Fulfillment of the Requirements for the degree of
Masters of Science (in Plastic Engineering)

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Dedication

Dedicated

To our parents with all our love

To our sisters

, our brothers

& our teachers who help us…

To our colleagues….

ACKNOWLEDGEMENT

I would like to express my gratitude to Dr. Mohamed Deen Hussien, my advisor, for his constant guidance and constructive criticism which have helped me to accomplish this work and improve my technical abilities.

This is an opportunity to express my sincere thanks also to the each teacher in the Department of Plastic Engineering for the help and advice I received over the years and to all my friends for their continuous support here at Sudan University and Science technology – Engineering Collage. I would also like to especially thank my colleagues Arman Mohammed Abdalla Ahmed and Muhab Slah Shalby to help me during every stage of this work.

Finally, I would like to thank my dear mother for their priceless support, love and never failing faith in me.

ABSTRACT

In plastics manufacturing, the melt flow index (MFI) is used as a routine indicator of rheological behavior when more expensive and laborious determinations of well-defined material functions are impractical. The MFI is the mass flow rate in a pressure driven flow through a standardized abrupt cylindrical contraction into a short tube performed under a standardized combination of pressure drop and temperature. In this research, we used Polyethylene low density grade of extrusion and poly propylene grade of injection to explore the connections between rheological properties and melt index.

We explore the role of shear by modeling the flow through the melt indexer using the different applied loads from 1.2 to 5 kg at different temperature to the same material to give us different value of MFI, and then calculate the mass flow rate, volume flow rate, pressure difference, viscosity, maximum shear stress and maximum shear rate. It was found that the viscosity is affected by temperature, changing the temperature change of the value of viscosity, and this change is inversely.

We present our results in different charts designed to help plastics engineers specify the MFI of a plastic for an industrial manufacturing process of known material functions.

المستخلص

يستخدم معامل انسياب المصهور في عمليات تصنيع البلاستيك كمؤشر روتيني للسلوك الريولوجي حيث تحديد هذا السلوك بالإختبارات المعمليه الاخرى مكلف وشاق ويحتاج الى زمن طويل وبعبارة اخرى غير عملي . معامل انسياب المصهور هو معدل انسياب الكتله المعرضه لضغط محدد من خلال قالب قياسي ذو مجرى اسطواني وعند درجة حرارة محدده. في هذا البحث يستخدم البولي ايثلين منخفض الكثافه والبولي بروبلين لأيجاد العلاقات ما بين الخواص الريولوجيه ومعامل انسياب المصهور. لأيجاد تأثير القص، بأستخدام جهاز قياس معامل انسياب المصهور بتطبيق احمال مختلفه من 1.2 الى 5 كيلوجرام عند درجات حرارة مختلفة لنفس الماده للحصول على قراءات مختلفة لمعامل انسياب المصهور ومن ثم نحسب معدل انسياب الكتله , معدل الانسياب الحجمي ,الضغط , اللزوجة , والقيم القصوى لأجهاد القص ومعدل إنفعال القص. وجد ان اللزوجة تتأثر بدرجة الحرارة , وتغيير درجات الحرارة يغير من قيمة اللزوجة ,وهذا التغيير يكون عكسياً .

عرضت النتائج في مخططات لمساعدة مهندسي البلاستيك لتحديد العمليه الصناعيه وفقاً لخصائص المادة.

TABLE OF CONTENTS

LIST OF FLOW CHARTS & TABLES

LIST OF FIGURES AND PLATES

List of abbreviation

Symbol	Description	Units
η	The Viscosity	$Pa.S$
F	The force	N
A	The area	cm^2
dv	different velocities	cm/s
dx	distance	cm
τ	The shear stress	Pa
$\dot{\gamma}$	The shear strain	S^{-1}
η_0	zero –shear viscosity	$Pa.S$
$\bar{K}$	consistency index of the polymer	
n	Power law- index for polymer	–
$\acute{\alpha} - 1$	the slope of the viscosity	–
Λ	Time constant (relaxation time)	S
$\acute{c}_1, \acute{c}_2$	Constant	–
T_s	the standard reference temperature taken asT_g + 50°K	^{0}C
T_g	'glass-transition temperature	^{0}C
A_0	the energy of activation for viscous flow	
R	the gas constant	
$\acute{A}_0, \acute{B}_0$	constants	–
P	pressure	N
$\bar{M}_w$	critical weight average molecular weight	
MFI	Melt Flow Index	$[g/10\,min]$
MFR	Melt Flow Rate	$[g/10\,min]$
MI	Melt Index	$[g/10\,min]$
G	shear elasticity modulus	
Δp	pressure difference between the inlet (A_{in}) and outlet (A_{out})	
l	length of the capillary	m
r	radius cylindrical	m
$\dot{V}$	volume flow	[m^3/s]
T	Test temperature	[$°C$]

s	Factor of standard time	$10\ minutes$ $= 600\ s)$
m	Amount of material flowing through the capillary under t time	$[g]$
MVR	volume rate	$[cm^3$ $/10\ min]$
t	Time needed for V amount of material to flow through the capillary	$[s]$
V	Amount of material flowing through the capillary under t time	$[cm^3]$
$\dot{G}$	mass flow	[kg/s]
P	density of the melt flow rate	[kg/m3]
L	piston moved distance or average distance of the measurement	$[cm]$
$\dot{\gamma}_{max}$	Maximum shear strain	S^{-1}
τ_{max}	Maximum shear stress	Pa

CHAPTER ONE

INTRODUCTION

1.1. Introduction:

Anyone beginning the process of learning to think Rheo-Logically must first ask the question, "Why should I make a viscosity measurement?" The answer lies in the experiences of thousands of people who have made such measurements, showing that much useful behavioral and predictive information for various products can be obtained, as well as knowledge of the effects of processing, formulation changes, aging phenomena, etc.

A frequent reason for the measurement of rheological properties can be found in the area of quality control, where raw materials must be consistent from batch to batch. For this purpose, flow behavior is an indirect measure of product consistency and quality.

Another reason for making flow behavior studies is that a direct assessment of process ability can be obtained. For example, a high viscosity liquid requires more power to pump than a low viscosity one. Knowing its rheological behavior, therefore, it is useful when designing pumping and piping systems.

It has been suggested that rheology is the most sensitive method for material characterization because flow behavior is responsive to properties such as molecular weight and molecular weight distribution. Rheological measurements are also useful in following the course of a chemical reaction. Such measurements can be employed as a quality check during production or to monitor and/or control a process. Rheological measurements allow the study of chemical, mechanical, and thermal treatments, the effects of additives, or the course of a curing reaction. They are also a way to predict and control a host of product properties, end use performance and material behavior.

Viscosity is the most important flow property, and it is the resistance to shearing, there are many different techniques for measuring viscosity. In capillary viscometers like (Melt Flow Index Tester), the shear stress is determined from the pressure applied by a piston. The shear rate is determined from the flow rate. Significance and use the melt flow index:

1- This test method is particularly useful for quality control tests on thermoplastics.
2- This test method serves to indicate the uniformity of the flow rate of polymer as made by an individual process and, in this case, may be indicative of uniformity of other properties. However, uniformity of flow rate among various polymers as made by various processes does not, in the absence of other tests, indicate uniformity of other properties.
3- The flow rate obtained with the extrusion plastometer is not a fundamental polymer property. It is an empirically defined parameter critically influenced by the physical properties and molecular structure of the polymer and the conditions of measurement. The rheological characteristics of polymer melts depend on a number of variables. Since the values of these variables occurring in this test may differ substantially from those in large-scale processes, test results may not correlate directly with processing behavior [8].

1.2. BACKGROND

1.2.1 Polymers:

Polymers are a large class of materials consisting of many small molecules (called monomers) that can be linked together to form long chains, thus they are known as macromolecules. A typical polymer may include tens of thousands of monomers. Because of their large size, polymers are classified as macromolecules [9].

Polymer Classification:

A. Plastics – Greek word plastikos (**Thermoplastics**, **Thermosets**)

The polymer chains can be free to slide past one another (thermoplastic) or they can be connected to each other with cross links (thermoset). Thermoplastics (including thermoplastic elastomers) can be reformed and recycled, while thermosets (including cross linked elastomers) are not re workable.

1- Thermoplastics:

Polymers that flow when heated; thus, easily reshaped and recycled. This property is due to presence of long chains with limited or no cross links. In a thermoplastic material the very long chain-like molecules are held together by relatively weak Vander Waals forces. When the material is heated the intermolecular forces are weakened so that it becomes soft and flexible and eventually, at high temperatures, it is a viscous melt (it flows). When the material is allowed to cool it solidifies again.

For example: polyethylene (PE), polypropylene (PP), poly (vinyl chloride) (PVC), polystyrene (PS), poly (ethylene terephthalate) (PET), nylon (polyamide), & unvulcanized natural rubber (polyisoprene).

2- Thermosets:

Decompose when heated; thus, cannot be reformed or recycled. Presence of extensive cross links between long chains induces decomposition upon heating and renders thermosetting polymers brittle.

A thermosetting polymer is produced by a chemical reaction which has two stages. The first stage results in the formation of long chain-like molecules similar to those present in thermoplastics, but still capable of further reaction. The second stage of the reaction (cross linking of chains) takes place during moulding, usually under the application of heat and pressure. During the second stage, the long molecular chains have been interlinked by strong covalent bonds so that the material cannot be softened again by the application of heat. If excess heat is applied to these materials they will char and degrade.

For example: epoxy, unsaturated polyesters, phenol-formaldehyde resins, vulcanized rubber.

B. Elastomers:

The polymer chains in elastomers are above their glass transition at room temperature, making them rubbery. Can undergo extensive elastic deformation.

Elastomeric polymer chains can be cross linked, or connected by covalent bonds. Cross linking in elastomers is called vulcanization, and is achieved by irreversible chemical reaction, usually requiring high temperatures.

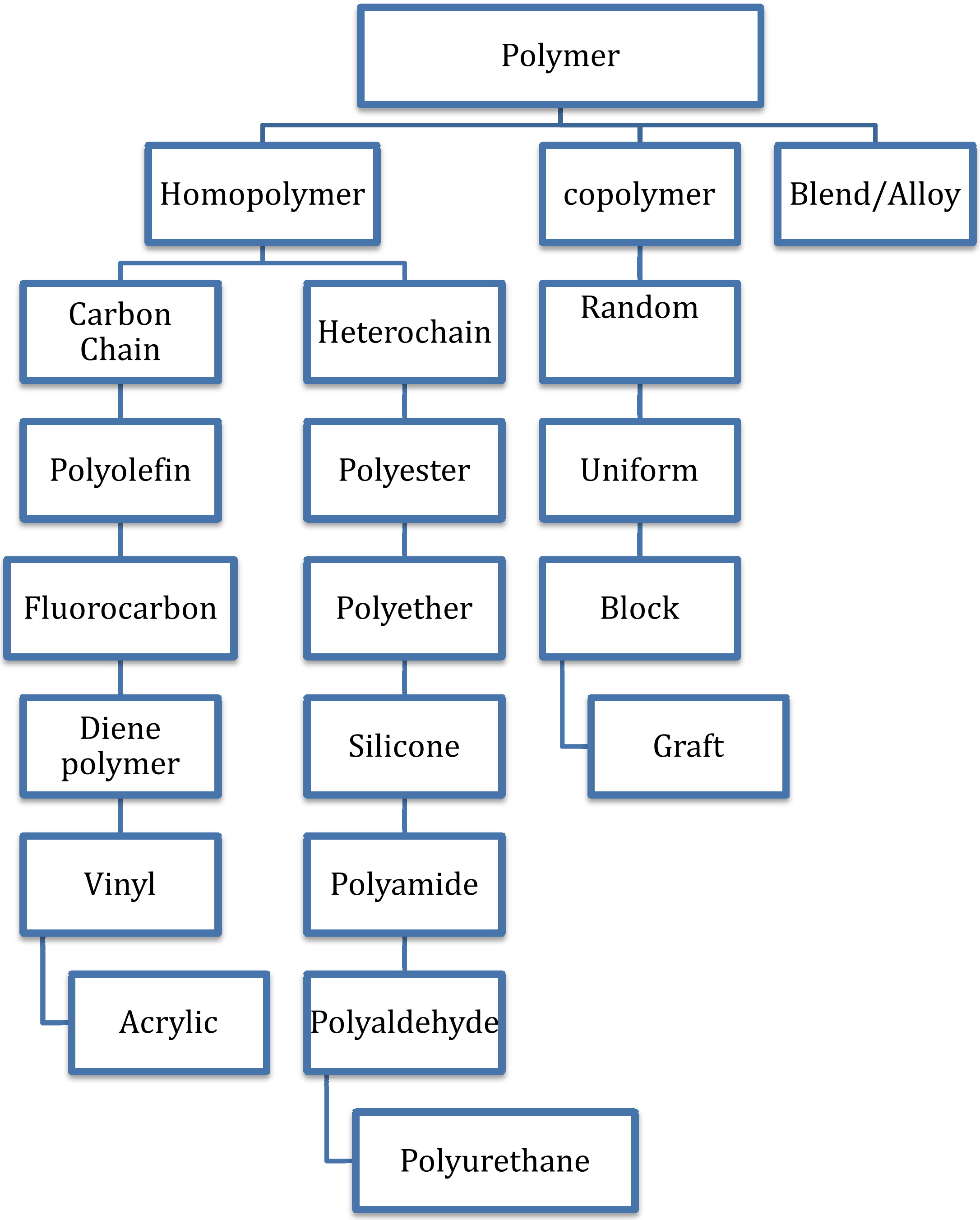
UN vulcanized natural rubber (polyisoprene) is a thermoplastic and in hot weather becomes soft and sticky and in cold weather hard and brittle. It is poorly resistant to wear. Sulfur compounds are added to form chains that bond adjacent polymer backbone chains and cross links them. The vulcanized rubber is a thermosetting polymer.

Cross linking makes elastomers reversibly stretchable for small deformations. When stretched, the polymer chains become elongated and ordered along the deformation direction. This is entropically unfavorable. When no longer stretched, the chains randomize again. The cross links guide the elastomer back to its original shape.

For example: natural rubber (polyisoprene), polybutadiene (used in shoe soles and golf balls), polyisobutylene (used in automobile tires), butyl rubber (pond and landfill linings), styrene butadiene rubber – SBR (used in automobile tires) and silicone.

Polymer classification can be done in a number of different ways, as shown in flow charts 1.1 – 1.2

Flow chart 1.1: polymer Classification Based on Chemical



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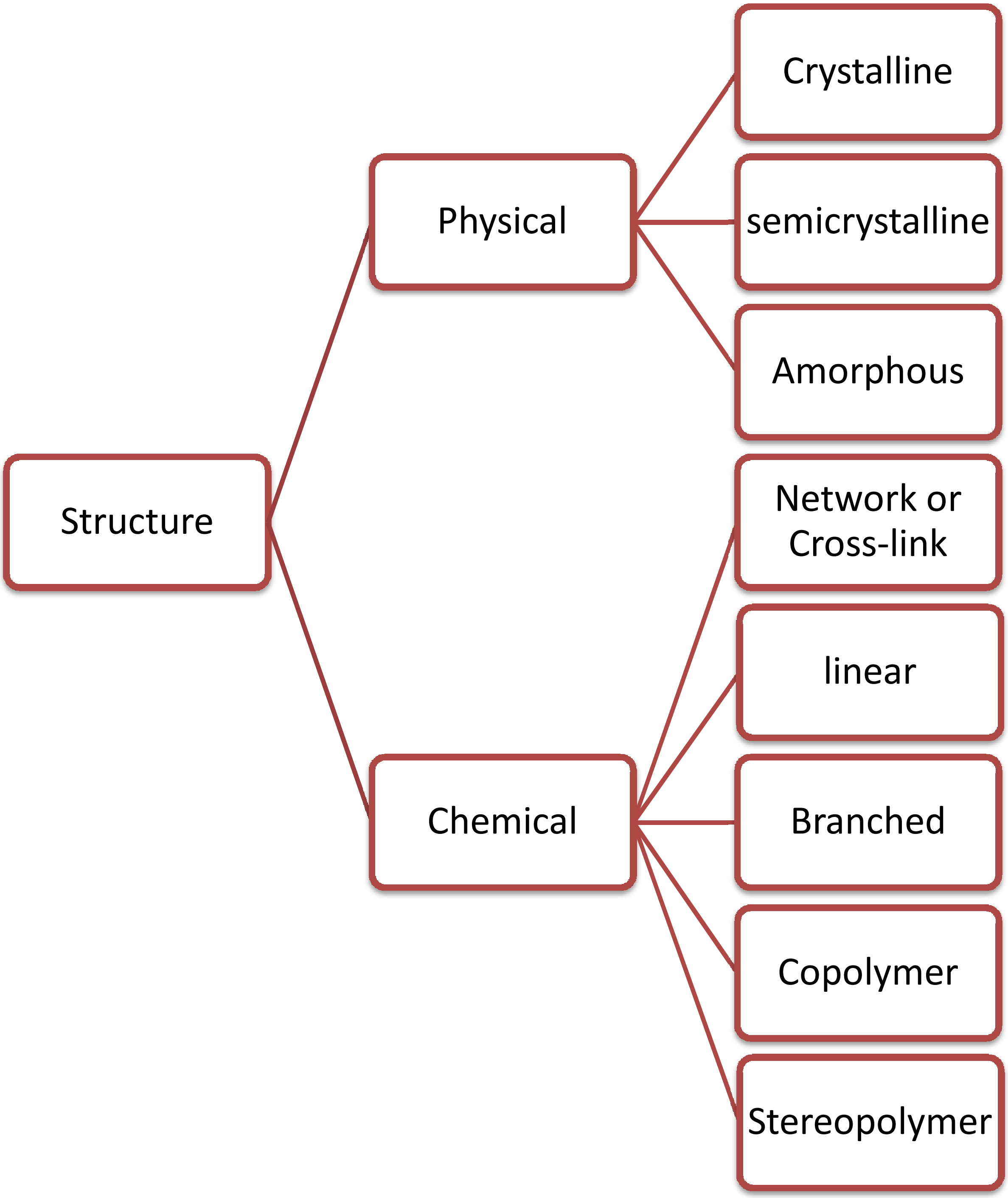
Flow chart 1.2: polymer Classification Based on Structure



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1.3. Objectives of present study:

In this research used two types of materials are polyethylene low density and polypropylene by using melt flow index testing, it is then recorded readings of MFI in different temperature, and then used the only two laws for Viscosity [Power law and Carreau law].

This study aim to:

1- Measurement of Melt flow index (MFI) and Melt volume rate (MVR) of LDPE with capillary Plastometer at a different temperature and with different weight at 2.16, 2.4, 2.84, 3.36, 3.36and 5kg, also for polypropylene at different temperature and with different weight at 1.2, 1.285, 1.525, 1.965, 2.16, 2.4, 2.84 and 3.36kg.

2- Calculation of mass flow$\dot{G}$, volume flow$\dot{V}$, viscosityη,maximum shear stress τ_f and maximum shear rate $\dot{\gamma}_f$.

3- Drawing of viscosity curve between viscosity versus shear rate and flow curve between shear stress via shear rate in different temperature.

4- Determination of activation energy For LDPE only.

5- Drawing the Experimantal data of viscosity with power law for LDPE &PP.

6- Drawing the Experimantal data of viscosity with Carreau model for LDPE & PP.

7- Drawing the relationship between the Melt flow index and loads for LDPE &PP.

Thesis out lines:

This thesis is divided into five chapters

- Chapter one gives relevant information on Classification of polymer.
- Chapter two presents introduction of Reology including viscosity and types of viscosity, Other Relationships for Shear Viscosity Function, Rheometers for polymer melt characterization, polymer processing, a literature review and objectives of present study.
- Chapter three Review the way the device used with a description of its parts and the method of operation of the device, Techniques used to calculate mass flow $\dot{G}$, volume flow $\dot{V}$, viscosity η, maximum shear stress τ_f and maximum shear rate $\dot{\gamma}_f$, Evaluation of standard test
- Chapter four present all of charts of low density polyethylene and polypropylene.
- Chapter five the conclusion and recommendations based on this study are summarized in this chapter.

CHAPTER TWO

LITERTURE REVIEW

2.1. Introduction:

Numerous publications have been reported on the rheological characterization of polymers. However, most of these publications pertain either to pure rheological characterization, with emphasis on finding possible explanations for certain observed polymer behaviors, or to the mathematical modeling of polymer behavior during the processing. Very few attempts have been made to characterize the rheological properties of resin, with the effect of rheology on resin process ability as the ultimate objective. In this chapter, some of publications related to this work are reviewed. But before displaying previous research, we explain some of the concepts related to the polymers behavior.

2.2. Rheology:

Rheology is the science of deformation and flow of materials. The Society of Rheology has a Greek motto "Panta Rei" translates as "All things flow." Actually, all materials do flow, given sufficient time. What makes polymeric materials interesting in this context is the fact that their time constants for flow are of the same order of magnitude as their processing times for extrusion, injection molding and blow molding [3]. In very short processing times, the polymer may behave as a solid, while in long processing times the material may behave as a fluid. This dual nature (fluid-solid) is referred to as viscoelastic behavior [3].

2.2.1 Viscosity:

Viscosity is the measure of the internal friction of a fluid. This friction becomes apparent when a layer of fluid is made to move in relation to another layer. The greater the friction, the greater the amount of force required to cause this movement, which is called shear. Shearing occurs whenever the fluid is physically moved or distributed, as in pouring, spreading, spraying, mixing, etc. Highly viscous fluids, therefore, require more force to move than less viscous materials.

Viscosity is the most important flow property. It represents the resistance to flow. Strictly speaking, it is the resistance to shearing, i.e., flow of imaginary slices of a fluid like the motion of a deck of cards. Referring to Figure 2.1, we can define viscosity as the ratio of the imposed shear stress (force F, applied tangentially, divided by the area A), and the shear rate (different velocities V1 and V2, divided by distance dx) [3].

$$\eta = \frac{shear\ stress}{shear\ rate} = \frac{F/A}{dv/dx} = \frac{\tau}{\dot{\gamma}} \quad \text{...} \quad (2\text{-}1)$$

For flow through a round tube or between two flat plates, the shear stress varies linearly from zero along the central axis to a maximum value along the wall. The shear rate varies nonlinearly from zero along the central axis to a maximum along the wall. The velocity profile is quasi-parabolic with a maximum at the plane of symmetry and zero at the wall as shown in Figure 2.2, for flow between two flat plates [3].

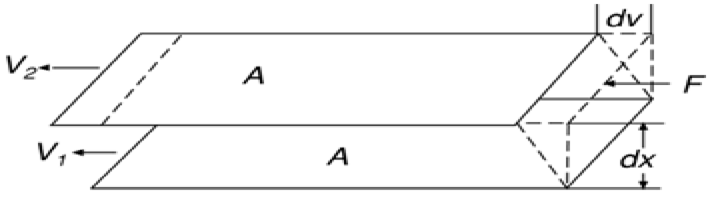


Figure: 2.1 Simple shear flows, Source Ref: [3].

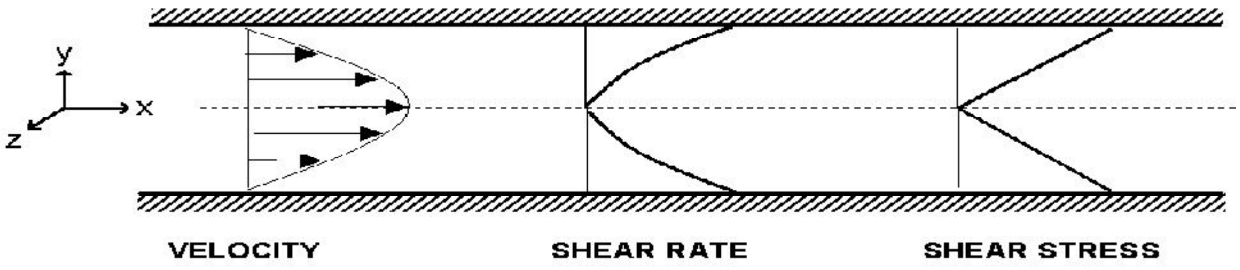


Figure: 2.2 Velocity, shear rate and shear stress profiles for flow between two flat plates

Source Ref: [3].

Fluid mechanics of polymers are modeled as steady flow in shear flow. Shear flow can be measured with a pressure in the fluid and a resulting shear stress. Shear flow is defined as flow caused by tangential movement. This imparts a shear stressτ, on the fluid. Shear rate is a ratio of velocity and distance and has units sec^{-1} Shear stress is proportional to shear rate with a viscosity constant or viscosity function.

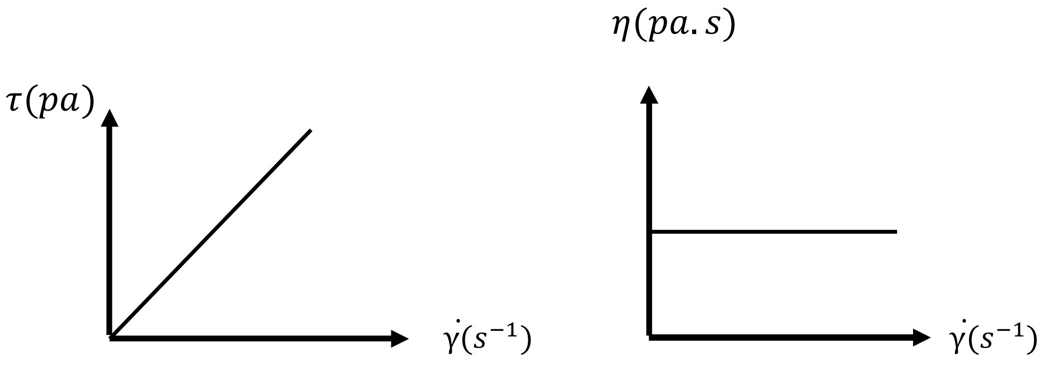
$$\tau_{yx} = \eta \frac{du}{dy} = \eta\dot{\gamma} \qquad (2\text{-}2)$$

The types of flow behavior are:

(A) Newtonian fluids :

Newtonian fluids are those having a constant viscosity dependent on temperature but independent of the applied shear rate. One can also say that Newtonian fluids have direct proportionality between shear stress and shear rate in laminar flow [5].

A Newtonian fluid is represented graphically in the figure 2.3 below. The Graph A shows that the relationship between shear stress (τ) and shear rate ($\dot{\gamma}$) is a straight line (Flow curve). Graph B shows that the fluid's viscosity remains constant as the shear rate is varied (Viscosity curve). Typical Newtonian fluids include water and thin motor oils [4].



Graph (A) Flow curve
Graph (B) Viscosity curve

Figure: 2.3 (Newtonian fluid) relationships between shear rate, shear stress and viscosity, Source Ref [4].

(B) non-Newtonian fluids (plastics) :

A non-Newtonian fluid is broadly defined as one for which the relationship $\tau / \dot{\gamma}$ is not a constant. In other words, when the shear rate is varied, the shear stress doesn't vary in the same proportion (or even necessarily in the same direction). The viscosity of such fluids will therefore change as the shear rate is varied. Thus, the experimental parameters of Viscometer model, spindle and speed all have an effect on the measured viscosity of a non-Newtonian fluid. This measured viscosity is called the apparent viscosity of the fluid and is accurate only when explicit experimental parameters are furnished and adhered to [6].

There are several types of non-Newtonian flow behavior, characterized by the way a fluid's viscosity changes in response to variations in shear rate. The most common types of non-Newtonian fluids you may encounter include:

1. Psuedoplastic:

   This type of fluid will display a decreasing viscosity with an increasing shear rate, as shown in the figure 2.4 below.

2. Dilatant:

   Increasing viscosity with an increase in shear rate characterizes the dilatant fluid; see the figure 2.4 below.

3. Plastic:

   This type of fluid will behave as a solid under static conditions. A certain amount of force applied to the fluid before any flow is induced; this force is called the yield stress (f'). Once the yield value is exceeded the flow begins, plastic fluids may display Newtonian, pseudoplastic, or dilatant flow characteristics. See the figure 2.4 below.

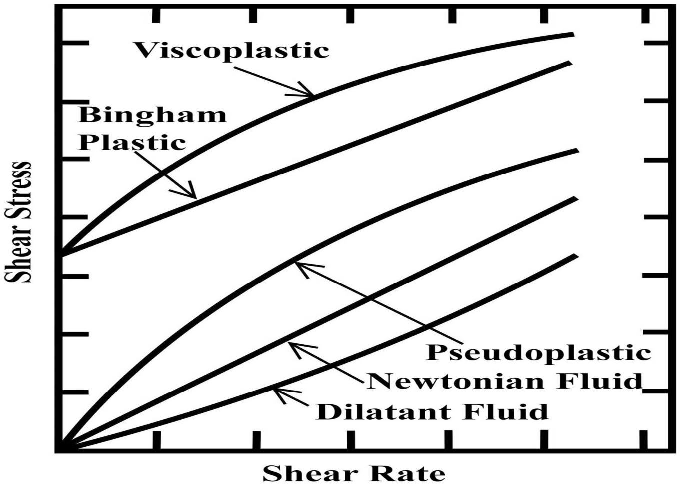


Figure .2.4. Qualitative flow curves for different types of non-Newtonian fluids.

So far we have only discussed the effect of shear rate on non-Newtonian fluids. What happens when the element of time is considered? This question leads us to the examination of two more types of non-Newtonian flow:

4. Thixotropy and Rheopexy

Some fluids will display a change in viscosity with time under conditions of constant shear rate. There are two categories to consider:

- Thixotropy:

  As shown in the figure 2.5 below, a thixotropic fluid undergoes a decrease in viscosity with time, while it is subjected to constant shearing.

- Rheopexy:

This is essentially the opposite of thixotropic behavior, in that the fluid's viscosity increases with time as it is sheared at a constant rate. See the figure 2.5 below.

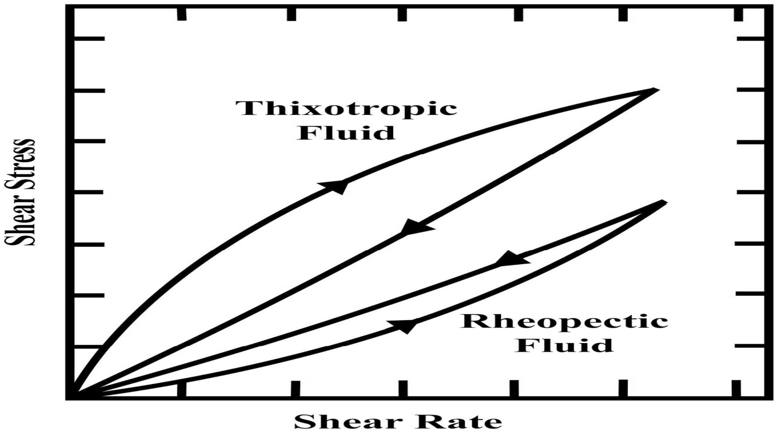


Figure: 2.5 (Thixotropy and Rheopexy) relationship between shear rates, shear stress

2.3 Other Relationships for Shear Viscosity Function

2.3.1 Viscosity-Temperature Relationships:

An understanding of the mechanism of polymer melt-flow processes in relation to the nature and composition of the material can be elucidated by a study of the temperature dependency of melt viscosity. The temperature sensitivity of the melt viscosity has a profound effect on the choice of processing conditions as well as on the quality of the end product. An increase in temperature sets up thermal motion of the molecules, resulting in their displacement based on the available free motion and the overcoming of forces of intermolecular interactions [7].

Presently, there are two commonly used expressions to evaluate the temperature dependency of the viscosity-one based on free-volume concepts, namely, the equation proposed by Williams et al. (W-L-F) , and the second, of the Arrhenius type, based on the absolute theory of rate processes as derived by Eyring [7] .

W-L-F Equation:

$$log \frac{\eta}{\eta_s} = \frac{\acute{c}_1(T-T_s)}{\acute{c_2}+(T-T_s)} \text{ ……………………………… (2-3)}$$

Where η and η_s are the viscosities at temperatures T and T_s, respectively; $\acute{c}_1$: and $\acute{c_2}$; are constants; andT_s, is the standard reference temperature taken asT_g + 50°K whereT_g, is the 'glass-transition temperature. Modifications of Eq. (2-3) using different constants and different characteristic temperatures have been proposed.

But becauseT_g, is a practical and easily available parameter, Eq. (2-3) is used preferentially.

Arrhenius-Erying Equation:

$$\eta = A_0 exp[\frac{E}{RT}] \text{ ……………………………………… (2-4)}$$

Where η is the viscosity at temperature T, R is the gas constant, A_0 is the frequency term depending on the entropy of activation of flow, and E is taken to be the energy of activation for viscous flow [7].

The temperature dependence of activation of flow process as defined by:

$$E = R\frac{ln\eta}{1/T} = \frac{2.303\acute{c}_1\acute{c}_2RT^2}{(\acute{c}_2+T-T_s)^2} \text{ ………………………………… (2-5)}$$

2.3.2 viscosity-Pressure Relationship:

Thermoplastic melt viscosity also depends on pressure. Viscosity generally increases with increasing pressure and can be correlated generally by an equation of the type [7].

$$\eta = \acute{A}_0 \exp(\acute{B}_0 p) \quad \text{(2-6)}$$

Where $\acute{A}_0$ and $\acute{B}_0$; are constants and P is the pressure. The pressure reduces free volume and, as a result, it reduces molecular mobility; however, this effect becomes noticeable only at very high pressures. High pressure raises both T_g and T_m, which also reflects an increase in viscosity. In general, during most practical situations, thermoplastic melts are assumed incompressible for ease and simplification.

2.3.3 Viscosity-Molecular Weight Relationship:

Experiments have shown that the following relationship between zero-shear viscosity and molecular weight holds:

$$\eta_0 = K_0 \bar{M}_w \quad \text{For} \quad \bar{M}_w < \bar{M}_{wc} \quad \text{(2-7)}$$

$$\eta_0 = K_0 \bar{M}_w^{3.5} \quad \text{For} \quad \bar{M}_w > \bar{M}_{wc} \quad \text{(2-8)}$$

Where $\bar{M}_{wc}$ is the critical weight average molecular weight, thought to be the point at which molecular entanglement begins to dominate the rate of slippage of molecules [7].

2.3.4 Shear-Rate-Dependent Viscosity Laws :

Several viscosity laws are available for generalized Newtonian flows. The isothermal viscosity laws will be presented in this section, and Temperature-

Dependent Viscosity Laws describes their extension to include temperature dependence in non isothermal flows [13].

1- Constant For Newtonian fluids, a constant viscosity

$$\eta = \eta_0$$

Can be specified η_0: Is referred to as the Newtonian or zero-shear-rate viscosity.

2- The power law for viscosity is:

$$\eta = K(\lambda\dot{\gamma})^{n-1} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \text{(2-9)}$$

Where: K is the consistency factor, λ is the natural time or the reciprocal of a reference shear rate, and n is the power-law index, which is a property of a given material.

3- The Bird-Carreau law for viscosity is:

$$\eta = \eta_\infty + (\eta_0 - \eta_\infty)(1 + \lambda^2\dot{\gamma}^2)^{\frac{n-1}{2}} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \text{(2-10)}$$

Where:

η_∞= infinite-shear-rate viscosity.

η_0 = zero-shear-rate viscosity.

λ=natural time (i.e., inverse of the shear rate at which the fluid changes from Newtonian to power-law behavior).

n= power-law index.

The Bird-Carreau law is commonly used when it is necessary to describe the low-shear-rate behavior of the viscosity. It differs from the Cross law primarily in the curvature of the viscosity curve in the vicinity of the transition between the plateau zone and the power law behavior [13].

4- The Cross law for viscosity is:

$$\eta = \frac{\eta_0}{1+(\lambda\dot{\gamma})^m} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \text{(2-11)}$$

Where:

η_0 = zero-shear-rate viscosity.

λ=natural time (i.e., inverse of the shear rate at which the fluid changes from Newtonian to power-law behavior).

m= Cross-law index (= 1- n for large shear rates).

Like the Bird-Carreau law, the Cross law is commonly used when it is necessary to describe the low shear- rate behavior of the viscosity. It differs from the Bird-Carreau law primarily in the curvature of the viscosity curve in the vicinity of the transition between the plateau zone and the power law behavior.

5- A modified Cross law for viscosity is also available:

$$\eta = \frac{\eta_0}{1+(\lambda\dot{\gamma})^m} \quad \text{...} \quad (2\text{-}12)$$

This law can be considered a special case of the Carreau-Yasuda viscosity law Eqn. (2-18) where the exponent a has a value of 1.

6- The Bingham law for viscosity is:

$$\eta = \eta_0 + \frac{\tau_0}{\dot{\gamma}}, \quad \dot{\gamma} \geq \dot{\gamma}_c$$

$$= \eta_0 + \tau_0 \frac{2-\frac{\dot{\gamma}}{\dot{\gamma}_c}}{\dot{\gamma}_c}, \quad \dot{\gamma} < \dot{\gamma}_c \quad \text{...} \quad (2\text{-}13)$$

Where: τ_0 is the yield stress and $\dot{\gamma}_c$ is the critical shear rate

The Bingham law is commonly used to describe materials such as concrete, mud, and toothpaste, for which a constant viscosity after a critical shear stress is a reasonable assumption, typically at rather low shear rates [13].

7- A modified Bingham law for viscosity is also available:

$$\eta = \eta_0 + \tau_0 \left[\frac{1-\exp(-m\,\dot{\gamma})}{\dot{\gamma}}\right] \quad \text{...} \quad (2\text{-}14)$$

Where $m = {}^{3}/_{\dot{\gamma}_c}$

8- The Herschel-Bulkley law for viscosity is:

$$\eta = [\tau_0 \frac{2-\frac{\dot{\gamma}}{\dot{\gamma}_c}}{\dot{\gamma}_c} + \mathrm{K} \left\lfloor (2-n) + (n-1)\frac{\dot{\gamma}}{\dot{\gamma}_c} \right\rfloor, \quad \dot{\gamma} \leq \dot{\gamma}_c$$

$$= \frac{\tau_0}{\dot{\gamma}} + K \left\lfloor \frac{\dot{\gamma}}{\dot{\gamma}_c} \right\rfloor^{n-1}, \quad \dot{\gamma} > \dot{\gamma}_c \ldots\ldots \text{(2-15)}$$

Where $\tau_0$is the yield stress, $\dot{\gamma}_c$ is the critical shear rate, K is the consistency factor and n is the power law index.

9- A modified Herschel-Bulkley law is also available:

$$\eta = \tau_0 \left[\frac{1-exp\left(\frac{-3\,\dot{\gamma}}{\dot{\gamma}_c}\right)}{\dot{\gamma}} \right] + K \left\lfloor \frac{\dot{\gamma}}{\dot{\gamma}_c} \right\rfloor^{n-1} \ldots\ldots \text{(2-16)}$$

10- The log-log law for viscosity is:

$$\eta = \eta_0 10^{a_0 + a_1 [log(\dot{\gamma}\ /\dot{\gamma}_c)] + a_{11}(log[\dot{\gamma}\ /\dot{\gamma}_c]^2)} \ldots\ldots \text{(2-17)}$$

Where: a_0, a_1 and a_{11} are the coefficients of the polynomial expression.

11- The Carreau-Yasuda law for viscosity is:

$$\eta = \eta_\infty + (\eta_0 - \eta_\infty)[1 + (\lambda\,\dot{\gamma})^a]^{\frac{n-1}{a}} \ldots\ldots \text{(2-18)}$$

Where:

η_0= zero-shear-rate viscosity.

η_∞=infinite-shear-rate viscosity.

λ=natural time (i.e., inverse of the shear rate at which the fluid changes from Newtonian to power-law behavior).

n= power-law index.

2.4 Rheometers for polymer melt characterization:

Rheometry is the measuring arm of rheology and its basic function is to quantify the rheological material parameters of practical importance [7].

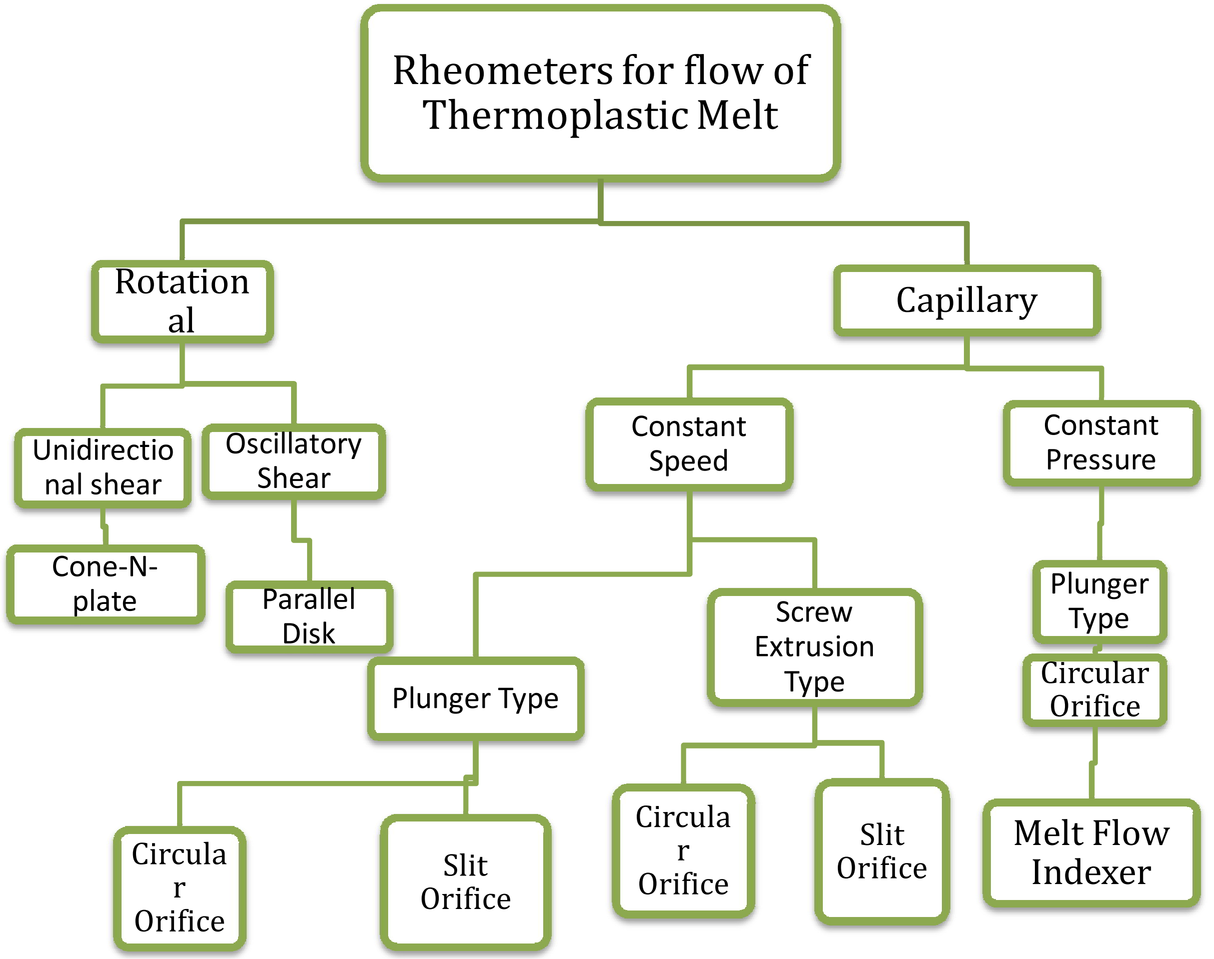
Rheometers used for determining the material functions of thermoplastic melts can be divided into two broad categories:

(1) Rotational type and

(2) Capillary type

Further, subdivisions are possible and these are shown in Table 2.1.

Flow chart 2.1: Rheometers for flow of Thermoplastic Melts



Source: Ref.7

2.5. Processing Polymers:

Polymer processing may be divided into two broad areas:

1- The processing of the polymer into some form such as pellets or powder.
2- The process of converting polymeric materials into useful articles of desired shapes [7].

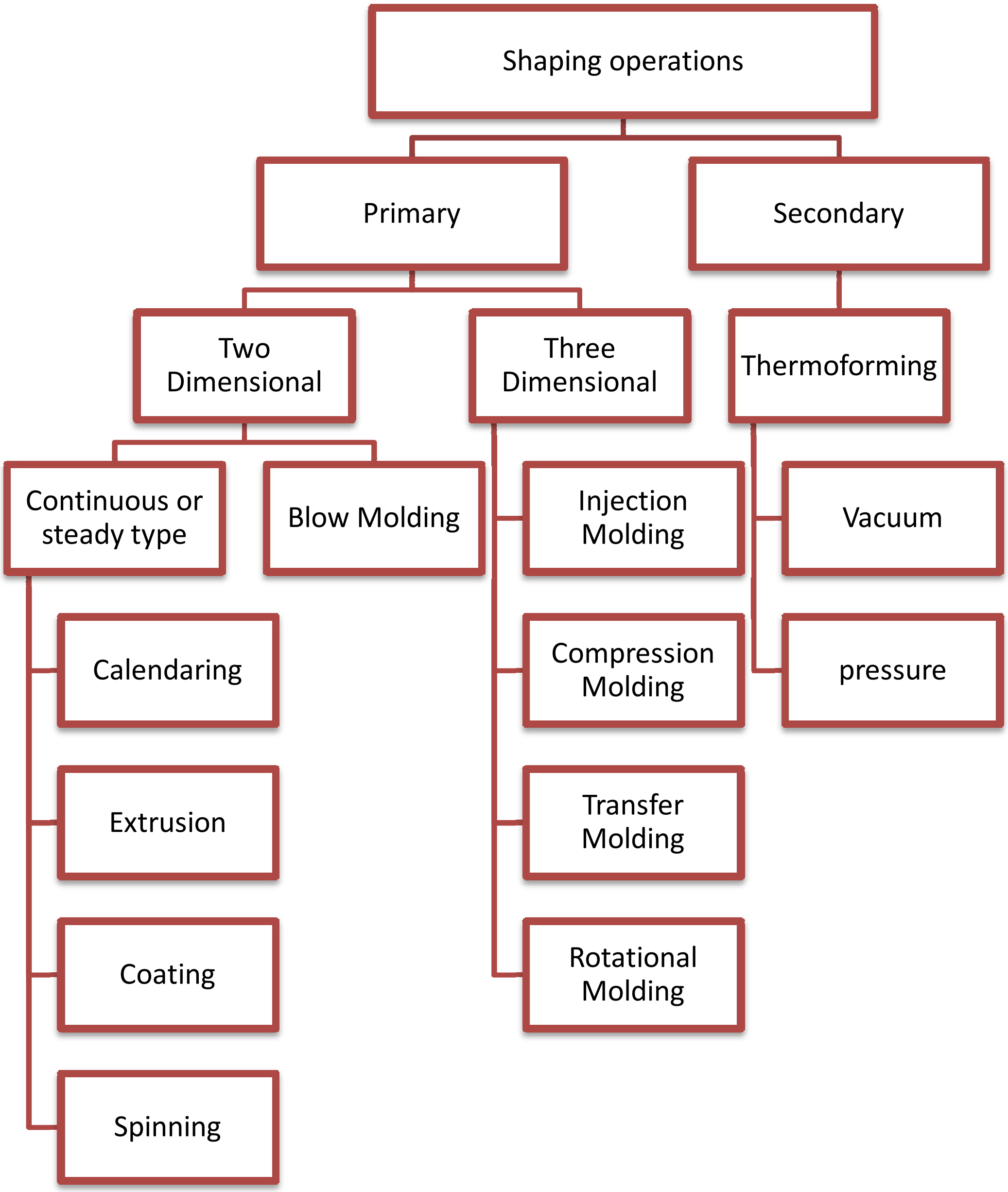
Our discussion here is restricted to the second method of polymer processing. The choice of a polymer material for a particular application is often difficult given the large number of polymer families and even larger number of individual polymers within each family. However, with a more accurate and complete specification of end-use requirements and material properties the choice becomes relatively easier. The problem is then generally reduced to the selection of a material with all the essential properties in addition to desirable properties and low unit cost. But then there is usually more than one processing technique for producing a desired item from polymeric materials or, indeed, a given polymer. For example, hollow plastic articles like bottles or toys can be fabricated from a number of materials by blow molding, thermoforming, and rotational molding. The choice of a particular processing technique is determined by part design, choice of material, production requirements, and, ultimately, cost–performance considerations.

The most common polymer processing unit operations, but only extrusion and injection molding, the two predominant polymer processing methods, are treated in fairly great detail. Our discussion is restricted to general process descriptions only, with emphasis on the relation between process operating conditions and final product quality. The subject of polymer processing has been traditionally and continuously as unite –shaping operations in Table 2.2.

2.5.1 Extrusion:

Extrusion is a processing technique for converting thermoplastic materials in powdered or granular form into a continuous uniform melt, which is shaped into items of uniform cross-sectional area by forcing it through a die. As shown in Table 2.2. Extrusion is perhaps the most important plastics processing method today.

Flow chart 2.2. Shaping Operations



Source: Ref.7

A simplified sketch of the extrusion line is shown in Figure 2.6. It consists of an extruder into which is poured the polymer as granules or pellets and where it is melted and pumped through the die of desired shape. The molten polymer then enters a sizing and cooling trough or rolls where the correct size and shape are developed. From the trough, the product enters the motor-driven, rubber-covered rolls (puller), which essentially pull the molten resin from the die through the sizer into the cutter or coiler where final product handling takes place.

extrusion end products include pipes for water, gas, drains, and vents; tubing for garden hose, automobiles, control cable housings, soda straws; profiles for construction, automobile, and appliance industries; film for packaging; insulated wire for homes, automobiles, appliances, telephones and electric power distribution; filaments for brush bristles, rope and twine, fishing line, tennis racket; parisons for blow molding.

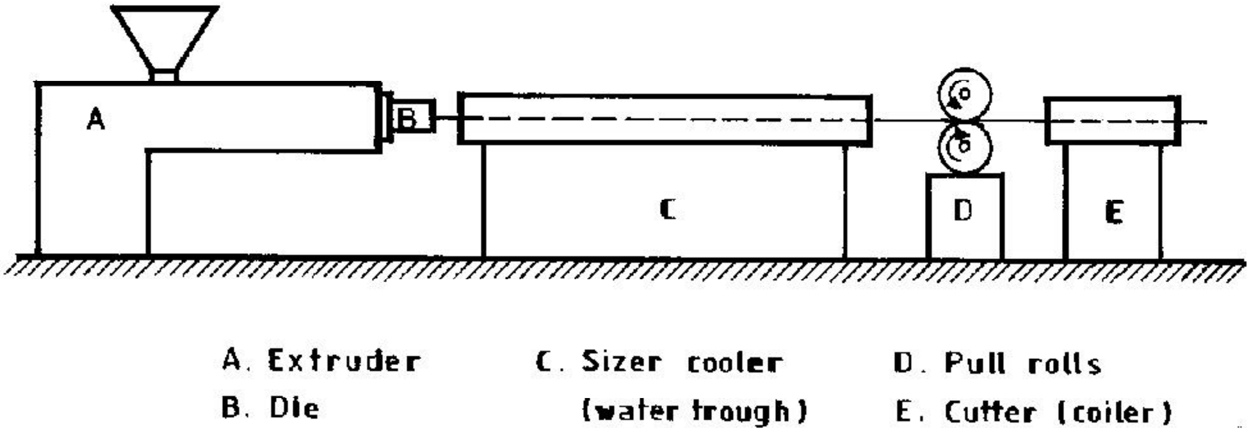


Figure 2.6: Sketch of an extrusion line.

2.5.2 Injection molding:

Injection molding is one of the processing techniques for converting thermoplastics, and recently, thermosetting materials, from the pellet or powder form into a variety of useful products. Forks, spoons, computer, television, and radio cabinets, to mention just a few, are some of these products. Simply, injection molding consists of heating the pellet or powder until it melts. The melt is then injected into and held in a cooled mold under pressure until the material solidifies. The mold opens and the product is ejected. The injection molding machine must, therefore, perform essentially three functions:

1. Melt the plastic so that it can flow under pressure.
2. Inject the molten material into the mold.
3. Hold the melt in the cold mold while it solidifies and then eject the solid plastic.

These functions must be performed automatically under conditions that ideally should result in a high quality and cost-effective part. Injection molding machines have two principal components to perform the cyclical steps in the injection molding process. These are the injection unit and the clamp unit (Figure 2.7). We now describe the operation of the various units of the injection molding machine that perform these functions.

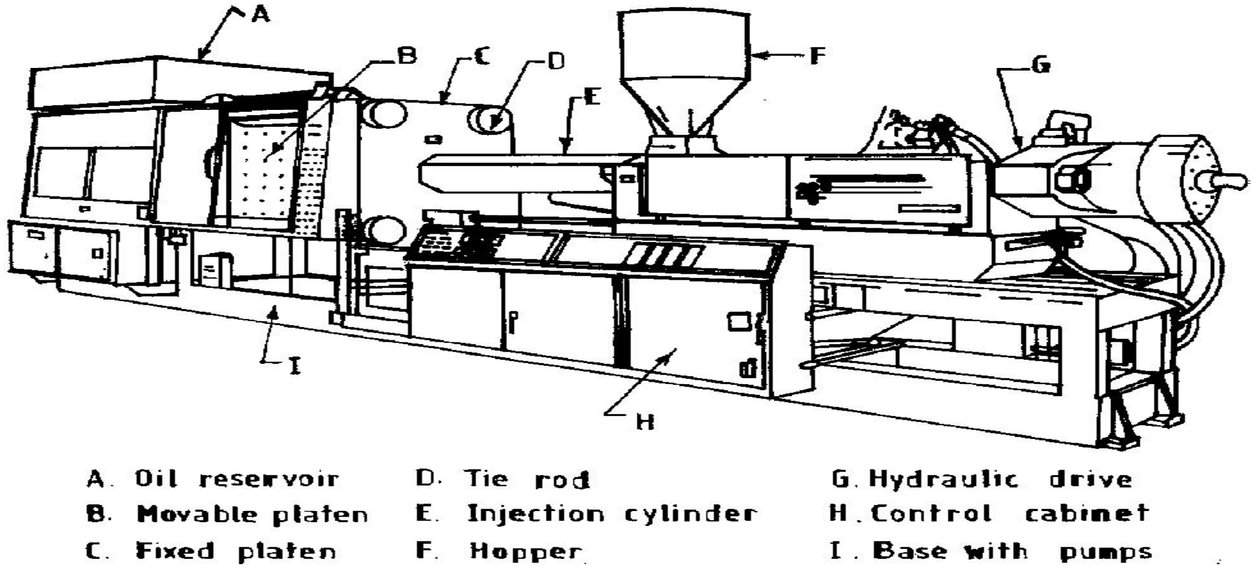


Figure 2.7: Major parts of a typical injection-molding machine.

2.6. Review of literature:

Throughout the years, research has been intensively going on which various publications on the melt flow index of plastics materials such as Understanding Melt Index and ASTM D1238, (Mertz et al, 2013) They use a finite element model to explore the connections between rheological properties and melt index. We explore the role of shear thinning by modeling the flow through the melt indexer using the Bird-Carreau model. They then explore the role of melt viscoelasticity in the MFI using the co rotational Maxwell model. They presented their results in dimensionless charts designed to help plastics engineers specify the MFI of a plastic for an industrial manufacturing process of known material functions [11].

Another study, (S.S. Jikan1 et al, 2013) studied the fabrication and characterization of polypropylene filled with recycled plaster of paris as filler had been carried out. Six different percentage levels of filler content were designed with PP/virgin plaster of paris composite and unfilled PP as reference samples. The sample characterizations were conducted by using melt density, melt flow index, tensile test and hardness test. The results demonstrate that the weight percent of filler content greatly influence the tensile property with decreasing values of maximum load and elongation at break as well as the melt flow index. However, the increase in melt density with increasing filler content leads to improve of hardness values of all samples [13].

There have been a number of rheological models proposed for representing the flow behavior of polymer melts. The constitutive equations, which relate shear stress or apparent viscosity with shear rate, involve the use of two to five parameters. Many of these constitutive equations are quite cumbersome to use in engineering analyses and hence only a few models are often popular [15]. For example Use of Least Square Procedures and Ansys Polyflow Software to Select Best Viscosity Model for Polypropylene (Arman Mohamed, Ahmed Ibtahim, 2013) .This work was intended to select the best viscosity model of polypropylene (PP) data using the percentage root-mean-square error function (PRMSE) and ansys package. Eleven samples of polypropylene (KPC - PP113) were tested at different loads and constant temperature 230^{o}C using melt flow index tester (SUST plastic laboratory) and the results for each sample were recorded. Different viscosity models (viscosity versus shear rate) were checked using polyflow and the best of them was selected using PRMSE function. It was found that PP 113 shear stress versus shear rate was Non-Newtonian and PRMSE beside easy to apply get an accurate model to quote viscosity [1]. Finally viscosity model Carreau-Yasuda was best one from them.

CHAPTER THREE

MATERIAL AND METHOD

3.1. Introduction:

In this study used Thermoplastic materials were:

1- LDPE (low density polyethylene) (HP 2022: no slip & No Anti block) for blown film from SABIC (**Saudi Arabia Basic Industries corporation**).

Typical Applications: Thin shrink film, lamination film, produce bags, textile packing, soft goods packing and general purpose bags with good optics and carrier bags.

Typical data:

Table 3-1: The basic characteristics of the used material

Properties	unit	Value
Resin properties		
Melt flow rate @190^0 C&2.16 kg load	$g/10\ min.$	2
Density @ 23^0 C	kg/m^3	922
Mechanical Properties		
Tensile Strength @ break	MPa	21
Tensile Elongation @ break	%	290
Tensile strength @ yield	MPa	8
1% Secant Modulus	MPa	160
Dart Impact Strength	g	60
Elmendorf Tear strength	g	180
Optical Properties		
Haze	%	7
Gloss @ 45^0	-	80
Thermal softening point		
Vicat softening point	^0C	92

Source: Saudi Arabia Basic Industries Corporation (Sabic)

2- Polypropylene (PP) injection grade, MFI = 20 g/10 min, from national petrochemical industrial company.

3-2 Method:

Melt Flow rate Index Testing instrument was used. It consists of a heated barrel and piston assembly to contain a sample of resin. A specified load (weight) is applied to the piston, and the melted polymer is extruded through a capillary die of specific dimensions as shown in figure 3-1. The mass of resin, in grams, that is extruded in 10 minutes equals the MFR; expressed in units of g/10 min. Some instruments can also calculate the shear rate, shear stress, and viscosity.

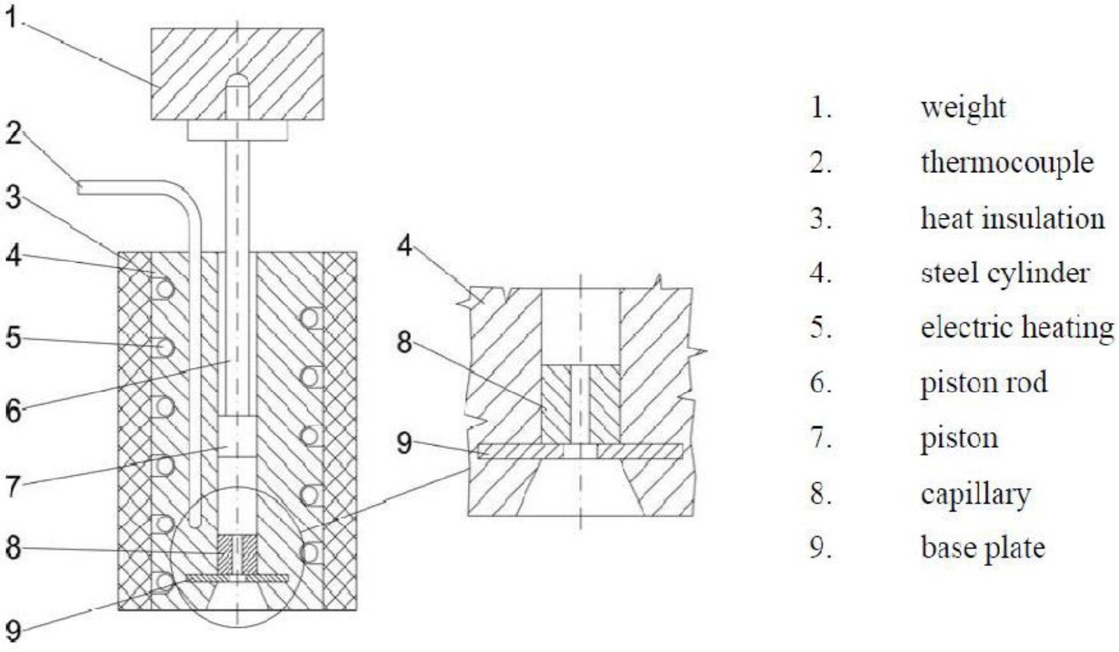


Figure 3.1Typical apparatus for determining melt flow rate (showing one of the possible methods of retaining the die and one type of piston), source Ref: [2].

Testing Procedure:

A small amount of the polymer sample 4grams for (LDPE) and 6 grams for (PP) is taken in the specially designed MFI apparatus. The apparatus consists of a small die inserted into the apparatus, with the outside diameter is 9.475mm, inner diameter is 2.095 mm and the length of die is 8.000mm.The material is packed properly inside the barrel to avoid formation of air pockets. A piston is introduced which acts as the medium that causes extrusion of the molten polymer. The length of piston bar is 210mm; piston diameter is 9.475mm.

The sample is preheated for a specified amount of time: 5 min at 190°C for polyethylene and 6 min at 230°C for polypropylene. After the preheating a specified weight is introduced into the piston. Examples of standard weights are 2.16 kg to 5 kg. The weight exerts a force on the molten polymer and it immediately starts flowing through the die. A sample of the melt is taken after desired period of time and is weighed accurately.

MFI is expressed as grams of polymer/10 minutes of total time of the test.

{Synonyms of Melt Flow Index are Melt Flow Rate and Melt Index. More commonly used are their abbreviations: MFI, MFR and MI}.

Confusingly, MFR may also indicate "melt flow ratio", the ratio between two melt flow rates at different gravimetric weights. More accurately, this should be reported as FRR (flow rate ratio), or simply flow ratio. FRR is commonly used as an indication of the way in which rheological behavior is influenced by the molecular mass distribution of the material.

MFI is often used to determine how a polymer will process. However MFI takes no account of the shear, shear rate or shear history and as such is not a good measure of the processing window of a polymer. The MFI device is not an extruder in the conventional polymer processing sense in that there is no screw to compress heat and shear the polymer. MFI additionally does not take account of long chain branching nor the differences between shear and elongational rheology. Therefore two polymers with the same MFI will not behave the same under any given processing conditions.

3.3. Techniques used to calculate mass flow$\dot{G}$, volume flow$\dot{V}$, viscosityη, maximum shear stress τ_f and maximum shear rate $\dot{\gamma}_f$:

Most processing technologies of thermoplastic polymers have a phase when the material is in a fluidic state before formation. This makes possible the completion of formation with relatively small force and pressure. The knowledge of behavior and characteristics of melts and the basics of melt rheology are essential for plastic processing and manufacturing of polymer products [3].

3.3.1 Basics, viscosity law of Newton:

What is the difference between solids and fluids? Fig.3.2. shows a solid body fixed between two plane plates. To the right a fluid layer can be seen placed between two similar plates. The cross section of the solid body and fluid parallel with the plates is marked with A [m^2] [3].

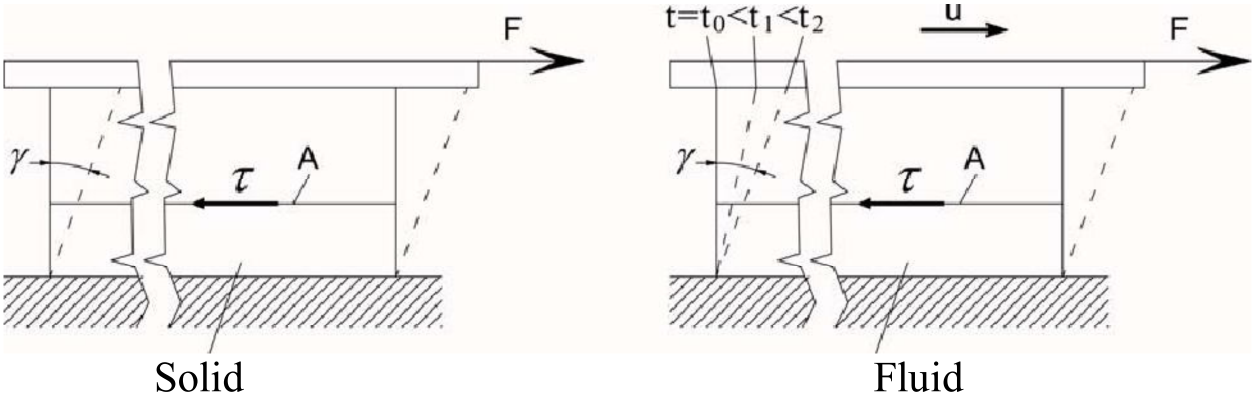


Figure 3.2.Deformation of solid body and fluid layer, source Ref: [3].

The lower plate is immobile while the upper one can be moved in parallel. If an F [N] force is applied on the upper plate, a τ=F/A [Pa] shear stress is developed and deformation occurs in the solid body. The γ angle featuring the deformation is directly proportional with the τ [Pa] shear stress to a certain limit (Hooke law, Chapter two section {2-2} Figure 2-3A). Thus the deformation is proportional with the shear stress developing in the solid body and the proportionality coefficient is the G shear elasticity modulus.

$$G = \frac{\tau}{\gamma} \qquad (3\text{-}1)$$

If there is fluid between the plates, the upper plate moves with a u velocity induced by the F force, the fluid is under a continuous deformation. Thus in case of fluids shear rate $(d\gamma/dt)$ is examined instead of γ deformation.

The basics of fluid mechanics according to Newton model is introduced below. The Newton model used for describing the behavior of real fluids is the basic model of melt rheology.

In case of Newtonian fluids the velocity distribution is linear between the two plates (Fig. 3.3). The velocity of fluid particles near to the stationary plate is $vx = 0$, while near to the upper plate the flow velocity is equal with the u velocity of the plate. Let's determine the $d\gamma$ rotatation of the M line during a dt time period! The upper end of the line is moving with a velocity of $vx + (dvx/dy)dy$, while the lower end is moving with a velocity of vx. We can get the rotation $(d\gamma)$ belonging to dt duration by dividing the difference of length of path with dy.

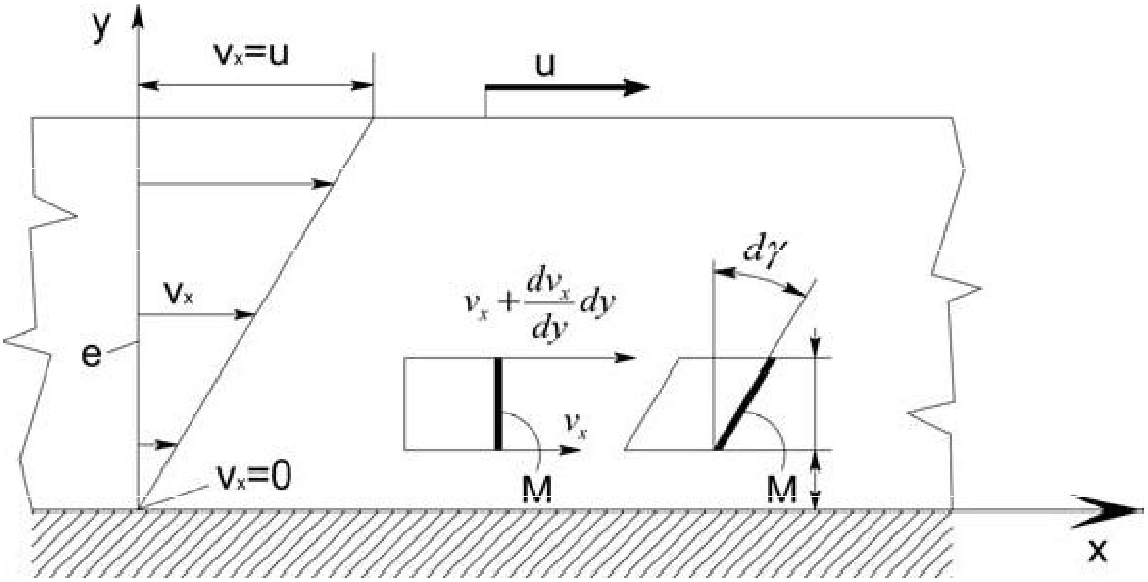


Figure: 3.3 Velocity distributions in fluid layer, source Ref: [3].

The time specific angular rotation, i.e. the shear rate is given by dividing with dt:

$$\dot{\gamma} = \frac{d\gamma}{dt} = \frac{dv_x}{dy} \qquad \text{(3-2)}$$

We can determine the linear relationship between shear rate and shear stress, the Newton equation, where η $[Pa \cdot s]$ is a factor depending on fluid properties called dynamic viscosity:

$$\tau = \eta\dot{\gamma} = \eta\frac{d\gamma}{dt} = \eta\frac{dv_x}{dy} \qquad \text{(3-3)}$$

The value of η depends on the shear stress required for maintaining a certain shear rate in case of a given fluid. It should be noted that if $\dot{\gamma}$& shear rate converges to zero, shear stress also converges to zero. This means that the static friction of fluids – in spite of solid materials – is zero.

Another difference is that fluids can be deformed endlessly without the alteration of their structure.

The flow velocity of fluids near to a wall is equal with the velocity of the wall. This phenomenon is called the law of adherence.

3.3.2 Flow in capillary:

The following chapter is dealing with flow of Newtonian fluids in a small diameter tube, i.e. capillary, because in the viscosimeter the tested material must pass through a capillary. Figure 3-4 shows the schema of capillary [3]. Equation (3-3) and the fact that $\dot{\gamma}$ shear rate can be expressed with the derivative function of flow velocity:

$$\tau = \eta\dot{\gamma} = \eta\frac{dv(r)}{dr} \quad \text{......} (3\text{-}4)$$

Where $v(r)$ $[m/s]$ is the flow velocity of the melt in the function of radial location r $[m]$ is the radial coordinate of capillary, $(0 \leq r \leq R)$.

From equation (3-4):

$$\frac{dv_r}{d_r} = \frac{\tau}{\eta} \quad \text{......} (3\text{-}5)$$

In order to continue the development we have to determine the distribution of τ shear stress along the cross section of the capillary. The following equation describes the force equilibrium of a fluid element in the function of r radius of the capillary:

$$2\pi r l\tau = r^2\pi\Delta p \quad \text{......} (3\text{-}6)$$

Where Δp $[Pa]$ is the pressure difference between the inlet (A_{in}) and outlet (A_{out}) cross section of the capillary, l (m) is the length of the capillary. The force developing on the surface of r radius cylindrical shell is in equilibrium with the pressure on the r radius base circle of the cylinder $(0 \leq r \leq R)$.

Furthermore we assume that the pressure decrease along the capillary axis (Figure 3-4) is linear, thusτ, v and $\dot{\gamma}$& are functions of the radius but they do not change along the capillary axis.

From equation (3.6) we get the function describing the distribution of τ in the cross section, which is independent on the material and depends only on the loading and the dimensions of the capillary:

$$\tau = \frac{\Delta \mathrm{p}}{2l}\,\mathrm{r} \ \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \ (3\text{-}7)$$

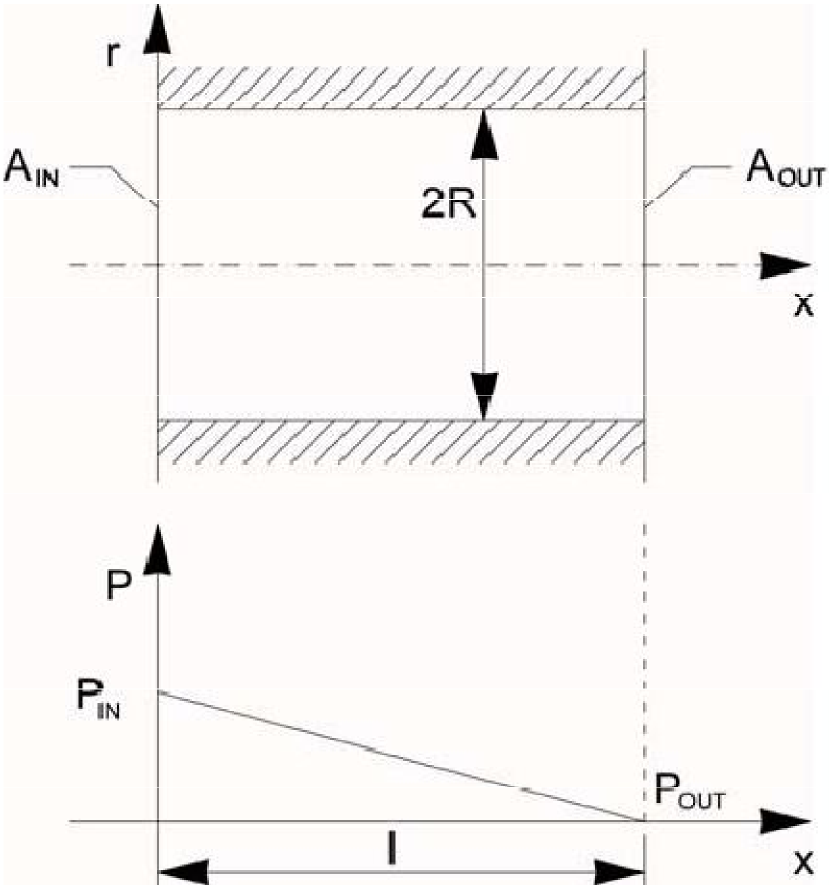


Figure 3-4 Pressure distribution along the capillary

The distribution has a conical shape; it is stress-free in the axis and reaches its maximum value at$r = R$. The τ shear stress is directly proportional with the radius, thus it reaches its maximum value at the wall of the capillary, at $r = R$ (Figure 3-5).

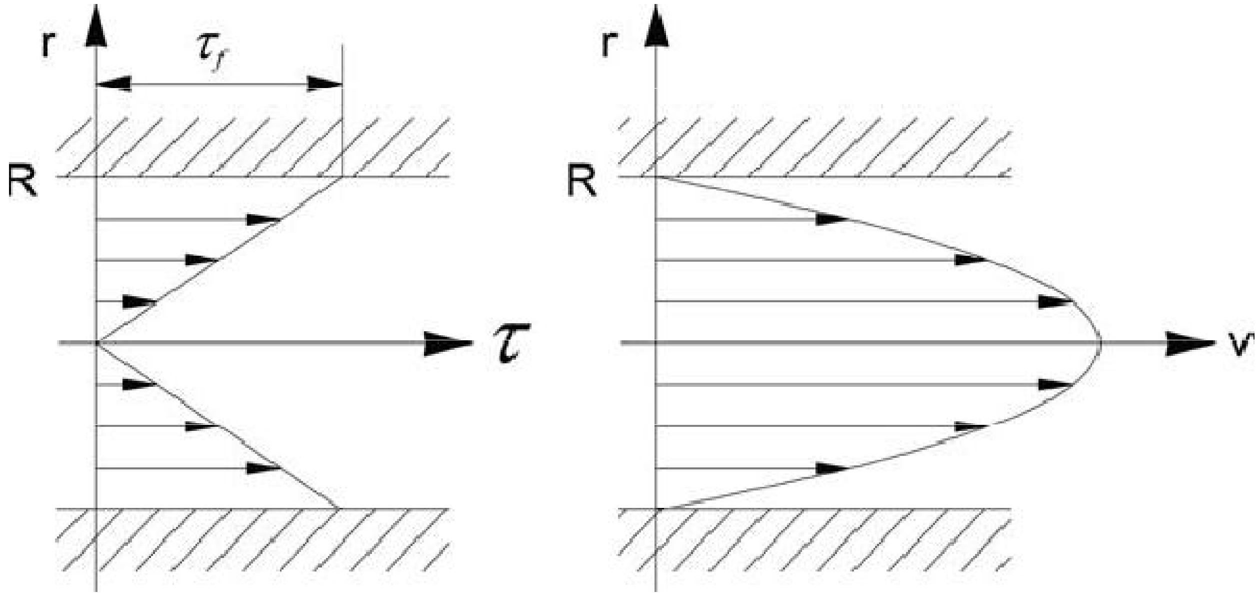


Figure 3-5 Distribution of τ shear stress and flow velocity in the cross section of the capillary.

$$\tau = \tau_{r=R} = \tau_f = \frac{\Delta p}{2l} R \quad \text{(3-8)}$$

By substituting equation (3.7) into (3.5) we get the following differential equation:

$$\frac{dv}{dr} = \frac{\Delta p}{2l\eta} r \quad \text{(3-9)}$$

From differential equation (3.9):

$$dv = \frac{\Delta p}{2l\eta} r\, d_r \quad \text{(3-10)}$$

From (3.10) by integration:

$$v = \frac{\Delta p}{2l\eta} \frac{r^2}{2} + c \quad \text{(3-11)}$$

In order to determine constant c we shall use the following boundary condition:

$$\text{At } r = R: v = 0 \quad \text{……} \quad (3\text{-}12)$$

By substituting the above equation into (3.11):

$$0 = \frac{\Delta \mathrm{p}}{2\, l \eta} \frac{\mathrm{R}^2}{2} + \mathrm{c} \quad \text{……} \quad (3\text{-}13)$$

From equation (3.13) we get the value of c:

$$\mathrm{c} = -\frac{\Delta \mathrm{p} \mathrm{R}^2}{4\, l \eta} \quad \text{……} \quad (3\text{-}14)$$

By substituting the value of constant c into equation (3.11) we get the function describing the distribution of flow velocity in the cross section (Figure 3-5):

$$\mathrm{v} = \frac{\Delta \mathrm{p}}{4\, l \eta} (\mathrm{r}^2 - \mathrm{R}^2) \leq 0 \quad \text{……} \quad (3\text{-}15)$$

This is a parabolic velocity distribution. The negative sign originates from the fact that the flow direction and the direction of pressure growth are opposite. In practice the velocity distribution is calculated from the following equation:

$$v = \frac{\Delta p}{4\, l\, \eta} (R^2 - r^2) \quad \text{……} \quad (3\text{-}16)$$

3.4 Evaluation of standard test:

The instrument is able to determine Melt Flow Index (MFI) by measuring the mass of melt which can be calculated by the following equation [2]:

$$\mathrm{MFI}_{(\mathrm{T,F})} = \frac{\mathrm{m.S}}{\mathrm{t}} \quad \text{……} \quad (3\text{-}17)$$

Where:

MFI $[g/10\, min]$; melt flow index,

T $[°C]$; Test temperature,

F $[N]$; Weight force,

s $[-]$; Factor of standard time $(10\ minutes = 600\ s), s = 600$

t $[s]$; Time needed for V amount of material to flow through the capillary,

m $[g]$; Amount of material flowing through the capillary under t time.

To determine MVR (Melt Volume Rate) volume rate, this can be calculated by the following equation:

$$\mathrm{MVR}_{(\mathrm{T,F})} = \frac{\mathrm{V.S}}{\mathrm{t}} \quad \text{…………………………………………………….. (3-18)}$$

Where: MVR $[cm^3/10\ min]$; volume rate,

T $[°C]$; Test temperature,

F $[N]$; Weight force,

S [-]; factor of standard time $(10\ minutes = 600\ s), s = 600$

t $[s]$; Time needed for V amount of material to flow through the capillary,

V $[cm^3]$; Amount of material flowing through the capillary under t time.

3.4.1Evaluation of MFI test:

In case of MFI tests, we apply a simplification, namely we neglect the pseudoplastic behavior of the polymer melt and we treat it as a Newtonian fluid. The data required for drawing the viscosity curve can be calculated the following way [4]:

$\dot{V}$ & $[m^3/s]$ volume flow can be calculated from the MVR by the following equation:

$$\dot{\mathrm{V}} = \frac{\mathrm{MVR}}{\mathrm{S.10^6}} \quad \text{……………………………………………………………….. (3-19)}$$

Where:

$\dot{\mathrm{V}}$ $[m^3/\mathrm{s}]$; volume flow,

MVR $[cm^3/10\ \mathrm{min}]$; volume flow measured by instrument,

s [-]; factor of standard time $(10\ minutes = 600\ s), s = 600$

Division with $10^6$is needed to convert $[cm^3]$ to $[m^3]$.

MFI can be calculated by equation (3-17).

$\dot{G}$ [kg/s] mass flow can be calculated from the MFI by the following equation:

$$\dot{G} = \frac{MFI}{S.10^3} \quad \text{...} \quad (3\text{-}20)$$

Where:

$\dot{G}$ [kg/s]; mass flow,

MFI [g/10 min]; melt flow index,

s [-]; factor of standard time $(10\ minutes = 600\ s), s = 600$

Division with 10^3 is needed to convert $[g]$ to$[kg]$.

ρ [kg/m3] density of the melt flow rate can be calculated from $\dot{V}\&\dot{G}$ volume flow and mass flow:

$$\rho = \frac{m}{0.711\ L} = \frac{\dot{G}}{\dot{V}} \quad \text{...} \quad (3\text{-}21)$$

Where:

L [cm]: piston moved distance or average distance of the measurement,

m [g}: the sample quality of extrusion when the piston moved L [cm}.

Pressure at inlet cross section of the capillary can be approximated with the pressure Calculated from D diameter of piston and F force of weight:

$$\Delta p = \frac{4}{\pi D^2} F \quad \text{...} \quad (3\text{-}22)$$

Furthermore we assume that according to Figure 3-4 Pressure decreasing is linear along the capillary axis, thus τ and $\dot{\gamma}$ depends on radius but is constant along the capillary axis.

$$\tau_{max} = \tau_{r=R} = \tau_f = \frac{\Delta p}{2l} R \quad \text{...} \quad (3\text{-}23)$$

Dynamic viscosity (η) can be calculated by:

$$\mu = \frac{\pi.\Delta p.R^4}{8l\dot{V}} \quad \text{...} \quad (3\text{-}24)$$

The distribution of $\dot{\gamma}$ shear rate in the cross section:

$$\dot{\gamma} = \frac{\tau}{\mu} = \frac{\Delta p}{2\mu l} r \quad \text{......} \quad (3\text{-}25)$$

The distribution of $\dot{\gamma}$ has a shape similar to the τ, because they differ only in a constant factor. $\dot{\gamma}$ Reaches its maximum value at the capillary wall.

$$\dot{\gamma}_{max} = \dot{\gamma}_{r=R} = \frac{\Delta p}{2\mu l} R = \frac{\tau_f}{\mu} = \dot{\gamma}_f \quad \text{......} \quad (3\text{-}29)$$

CHAPTER FOUR

RESULT AND DISSCUSION

4.1. Introduction:

In this chapter we present our experimental data in many different charts, In this research used Excel program to draw the data in the form of curves and extract trend line to draw a curve of power law, activation energy and carreau model.

4.1.1 Low density Polyethylene (LDPE):

a- The result obtained are given in figure 4-1, 4-2 , 4-3& 4-4 and the data is given in table 4.1,4.2 ,4.3 ,4.4 ,4.5 and 4.6 in Appendix (A).Drawing of viscosity curve between viscosity versus shear rate and flow curve between shear stress versus shear rate at different temperatures. The chart shows the variation of the viscosity and temperature .The higher the temperature the viscosity decreases and the shear strain rate increases shown in figure 4.1 & 4.2.

b- Drawing the experimantal data of viscosity with power law shown in figuer 4-3 from figure 4.1Was extracted trendline power law type , the index n for LDPE at 170^0C: n=o.61 , 190^0C: n= 0.61 . 210^0C: n=0.58.See appendices (A) table 4.8.

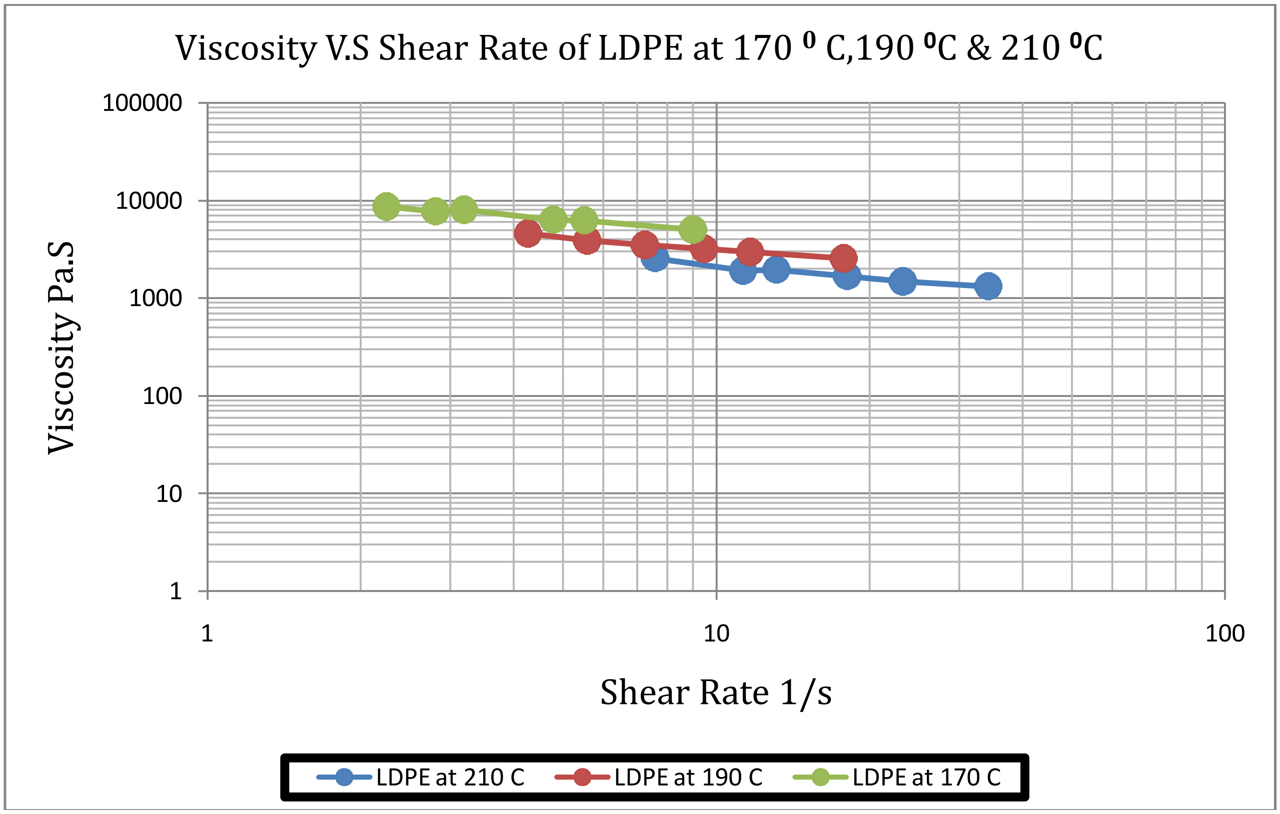


Figure: 4-1.Viscosity V.S shear rate curve for (LDPE at 210^{0}C, 190^{0}C & 170^{0}C).

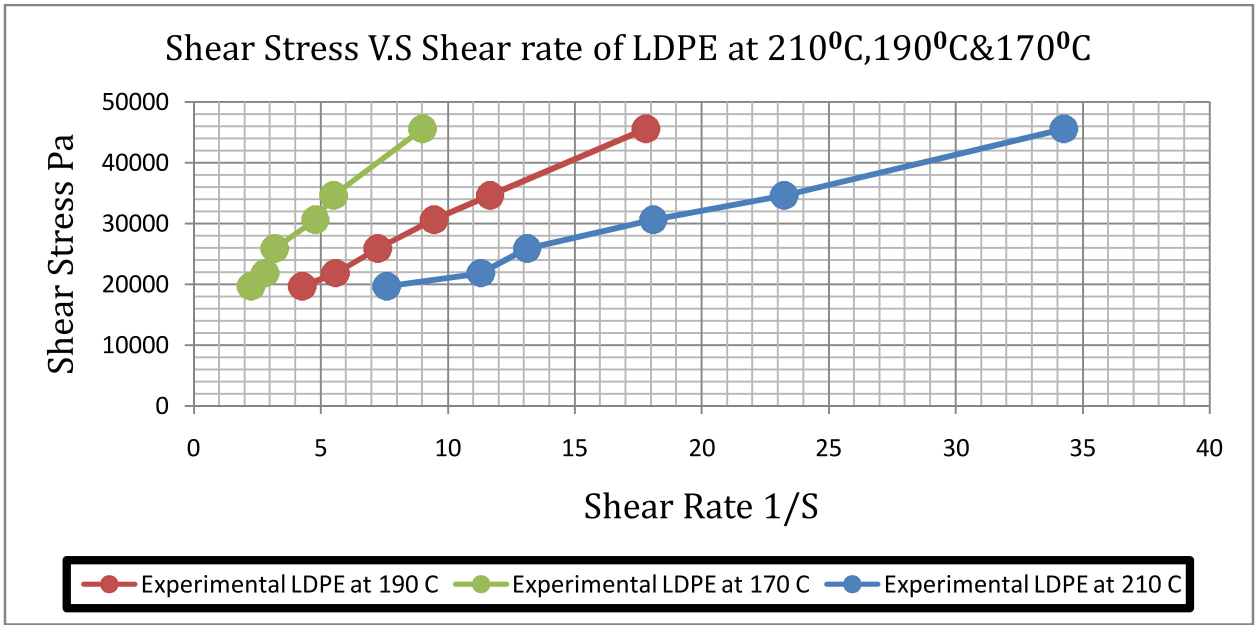


Figure: 4-2. Relation between Shear Stress and Shear Rate for (LDPE at 210^{0}C ,190^{0}C & 170^{0}C).

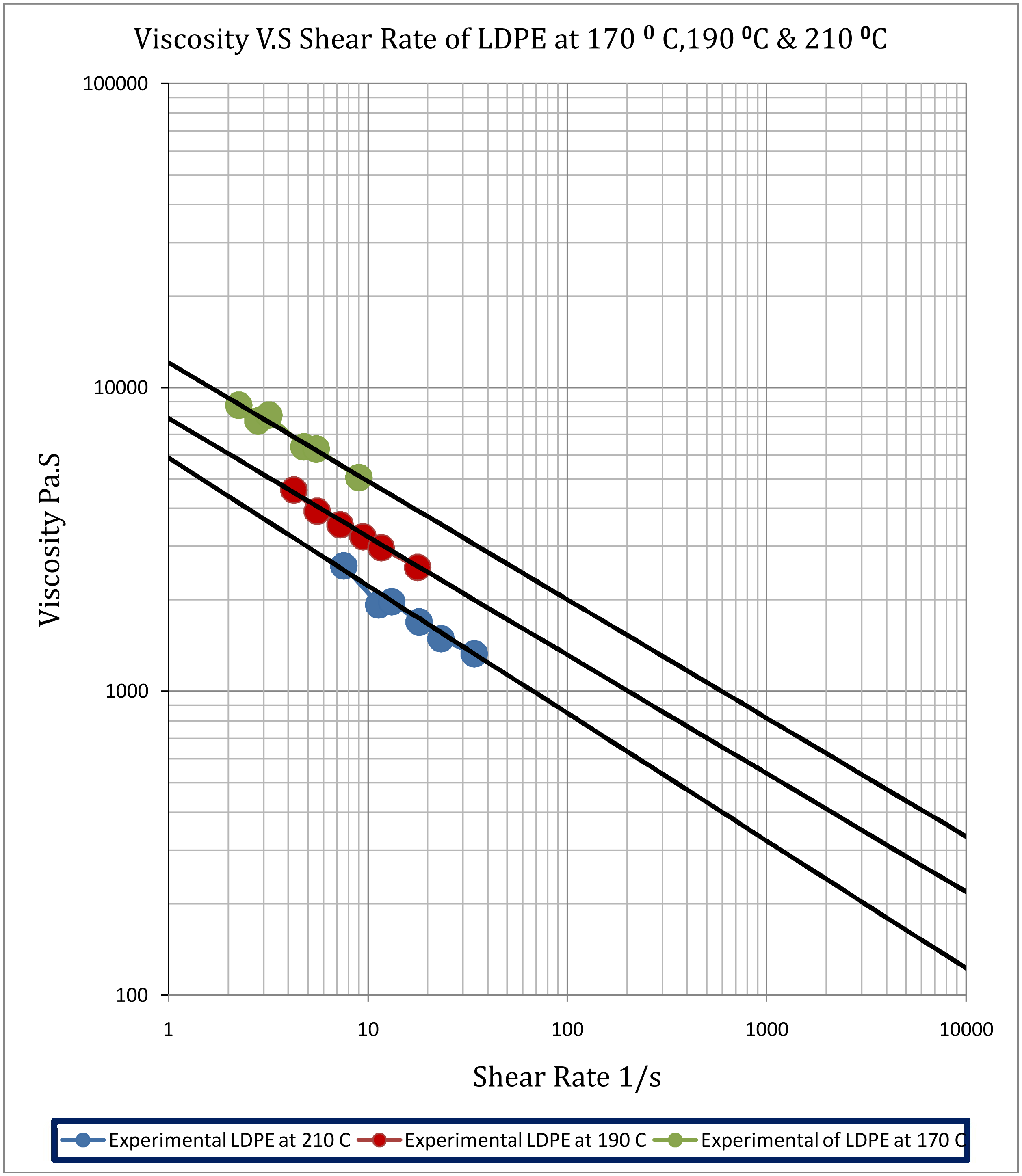


Figure: 4-3 Experimental viscosity with power law model c- Drawing the value of MFI experimental of LDPE at 170^{0}C, 190^{0}C &210^{0}C with applied load .

Figure 4-4 shows the implication of MFI on shear viscosity. The plot shows that increasing MFI causes a decrease in shear viscosity, which is expected. A faster flowing polymer shoud have a higher MFI and lower viscosity.

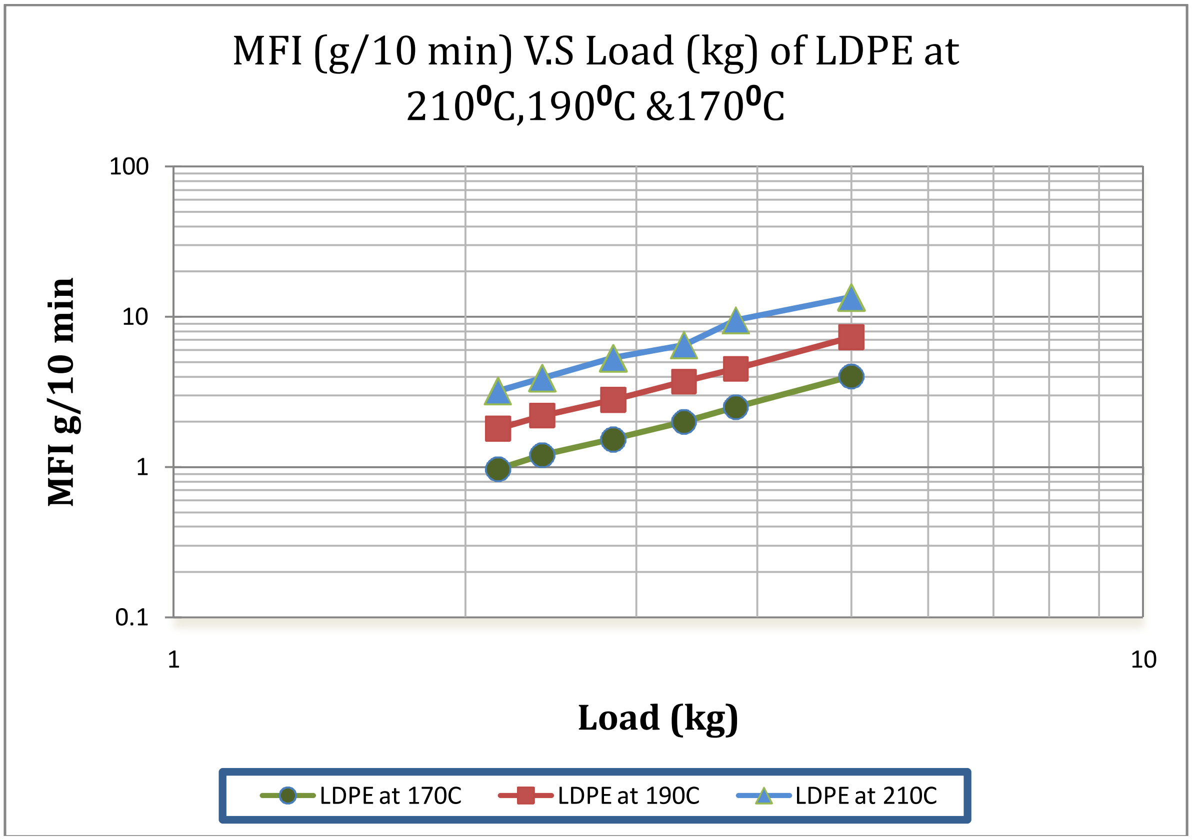


Figure: 4-4. Relation between Melt flow index MFI (g/10min) and load (kg)

d- Activation Energy :

An accurate determination of activation energy is very important . The value of activation energy changes with temperature and hence some preaution are necessary in its evalation . Melt viscosity measurement have been made at three different temperature 170^{0}C,190^{0}C &210^{0}C using the Equation 2-4in chapter two section 2.3.1.

Zero shear rate viscosity or the limiting viscosity has been obtained in the region where Q is proportinal to Δp. The logarithm of limiting viscosity is plotted aginast $1000/T$ shown in figure 4.5 and the value of activation energy obtained from the slope of the plot which is equal to $E/_R$.See appendices (A) table 4.7.

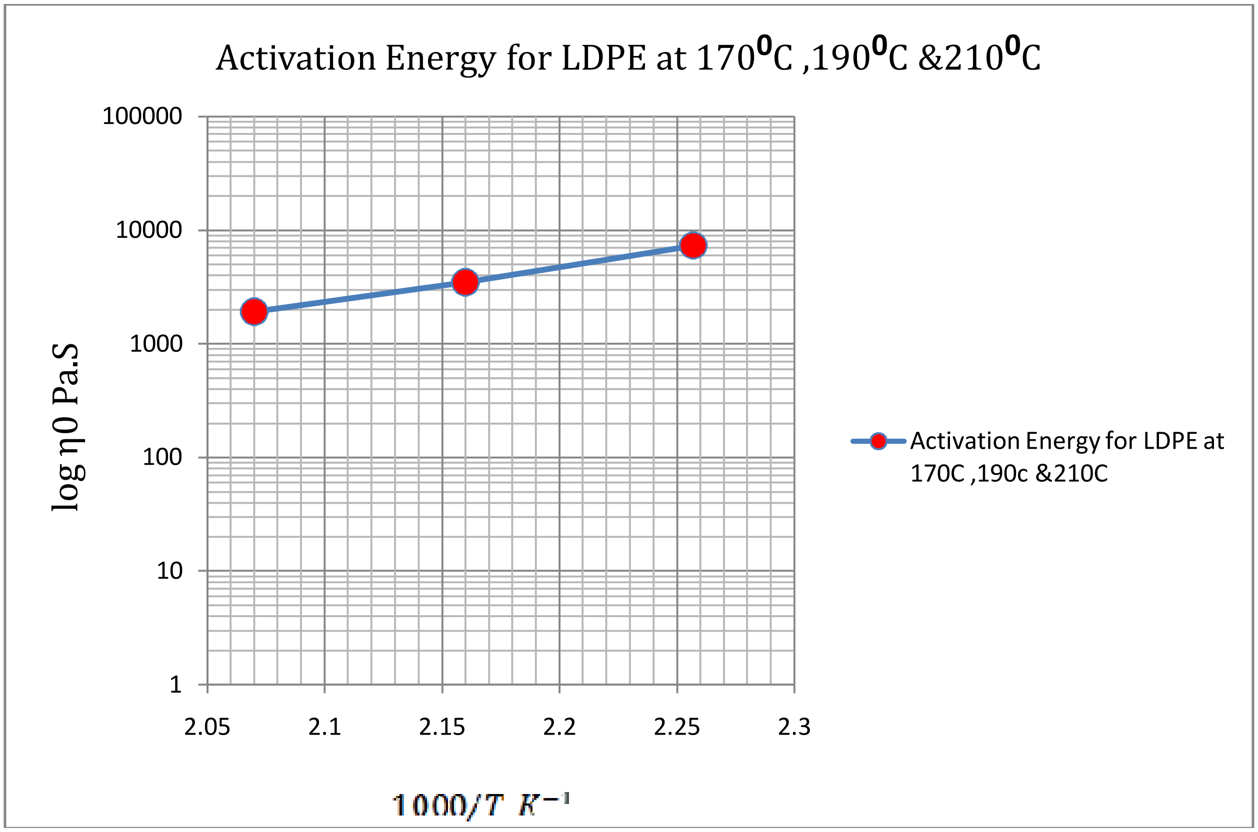


Figure .4-5. Logarithm of zero shear rate viscosity VS. $1000/T$

- From figure 4.3. the activation energy is egual Slope of plot

$$\frac{E}{8.3145} = \quad 26.315$$

$$E = 218.8\ k\ cal/mol$$

e- Drawing the experimantal data of viscosity with Carreau Law shown in figuer 4.6.

Note when the values of Shear strain rates between 0.1 to 1 the viscosity very regular or almost equal at the same temperature, but at shear rates greater than the one, the values of viscosity decreasing that curves up to the extent of interproximal.See appendices (A) table 4.9.

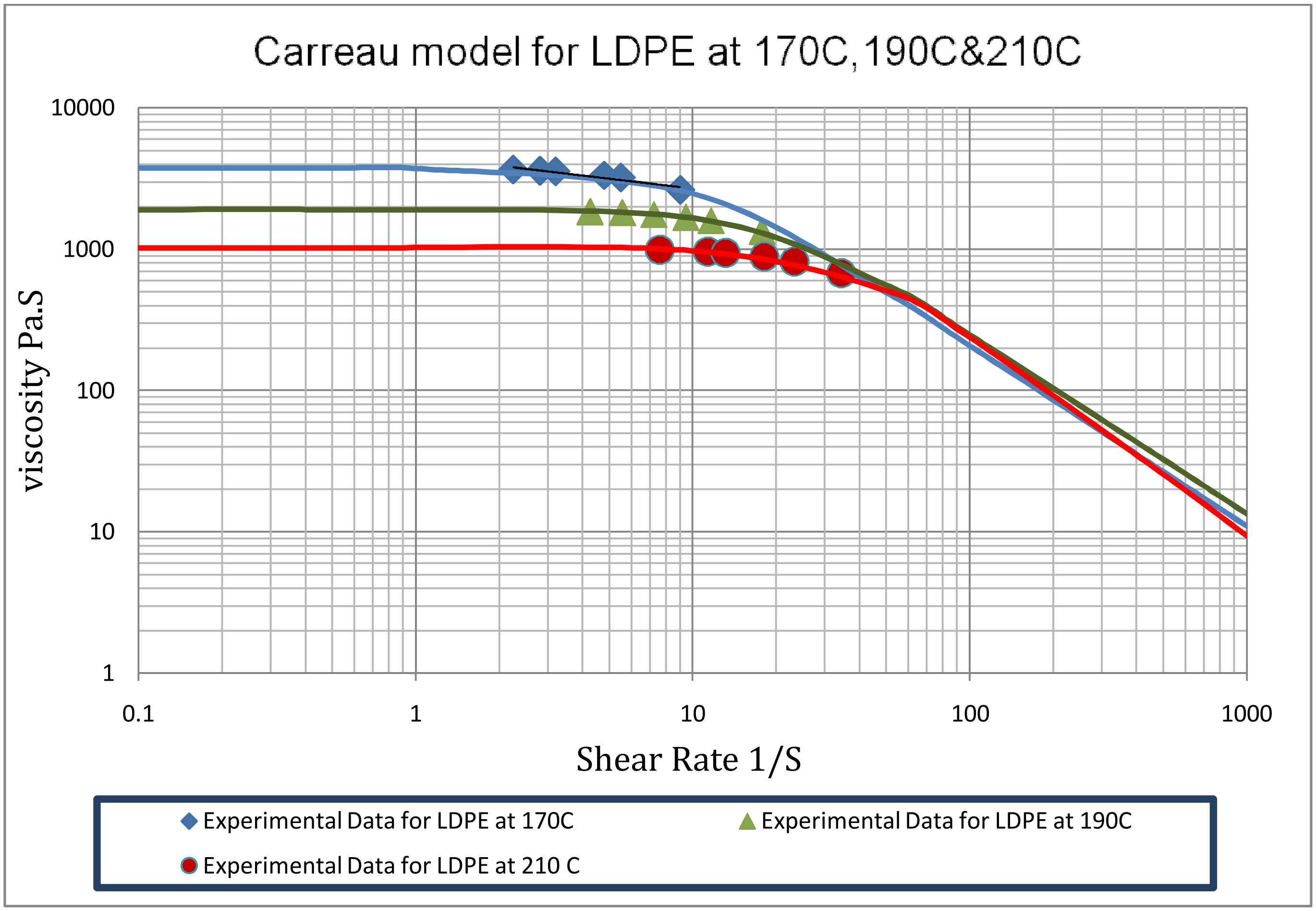


Figure 4-6 Experimental viscosity with carreau model.

4.1.2 Polypropylene (PP):

All the charts were drawn to raw polypropylene and bearing the same description that was mentioned previously in schemes polyethylene. The result obtained are given in figure 4-7, 4-8 , 4-9, 4-10 &4-11 and the data is given in table 4.10,4.11 ,4.12 ,4.13 ,4.14 and 4.15 in Appendix (B).

a- Drawing of viscosity curve between viscosity versus shear rate and flow curve between shear stress versus shear rate at different temperatures. The chart shows the variation of the viscosity and temperature .The higher the temperature the viscosity decreases and the shear strain rate increases shown in figure 4.7 &4.8.

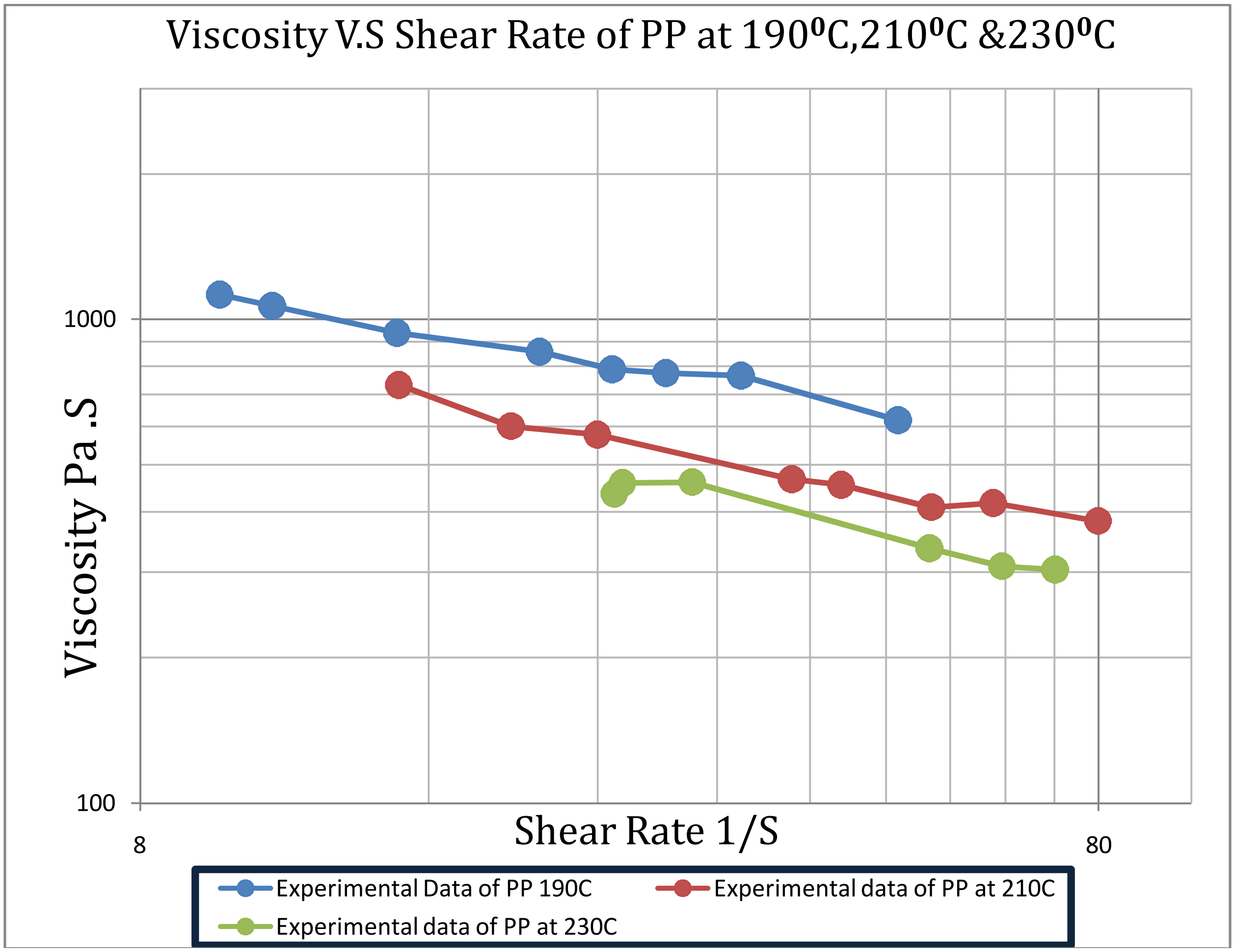


Figure: 4-7.Viscosity V.S shear rate curve for (PP at 190^0C, 210^0C & 230^0C).

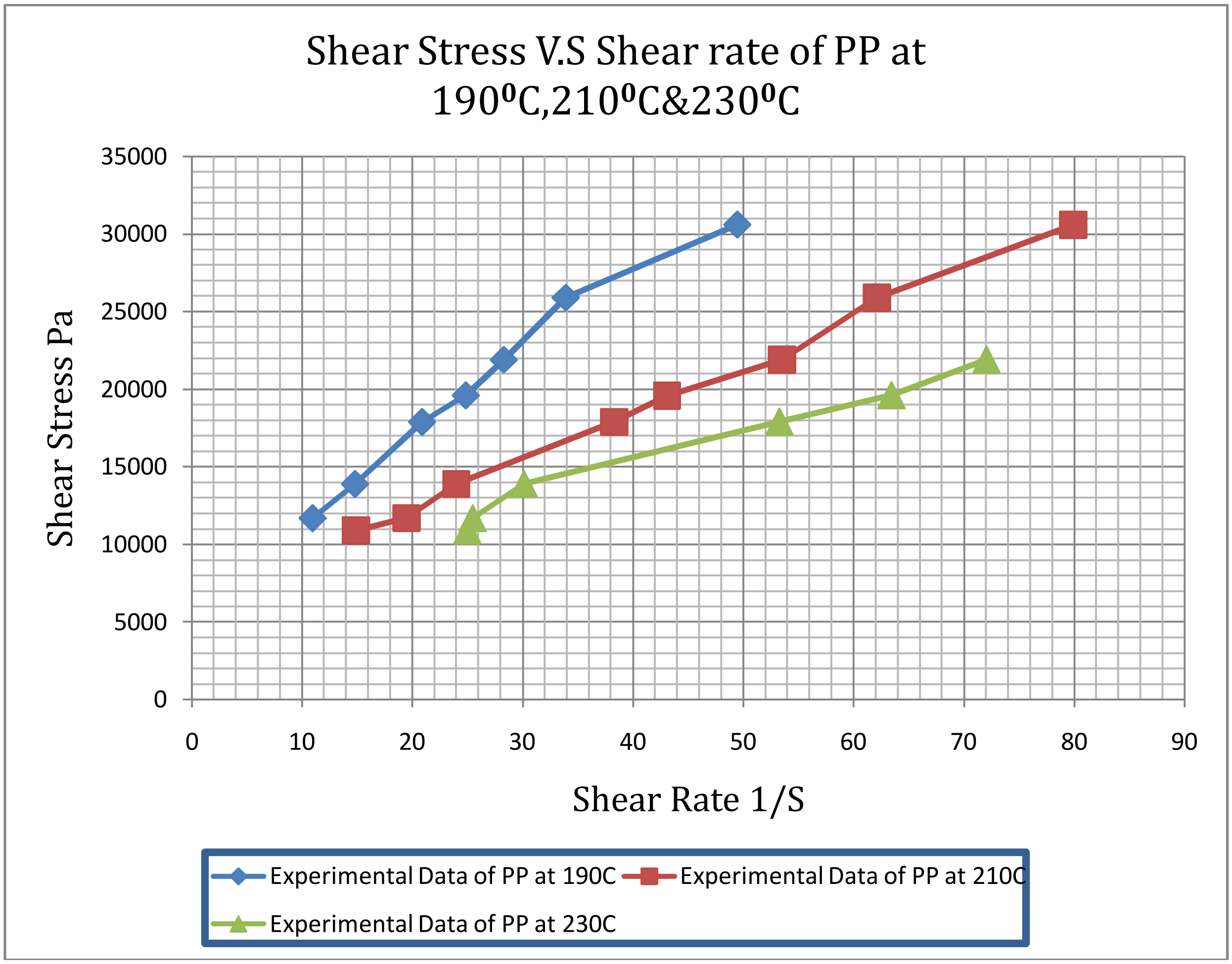


Figure: 4-8. Relation between Shear Stress and Shear Rate for (PP at 190^{0}C ,210^{0}C & 230^{0}C).

b- Drawing the experimantal data of viscosity with power law shown in figuer4-9. from figure 4-7Was extracted trendline power law type , the index n for PP at 190^0C: n=o.66 , 210^0C: n= 0.63 . 230^0C: n=0.60.See appendices (B) table 4.17.

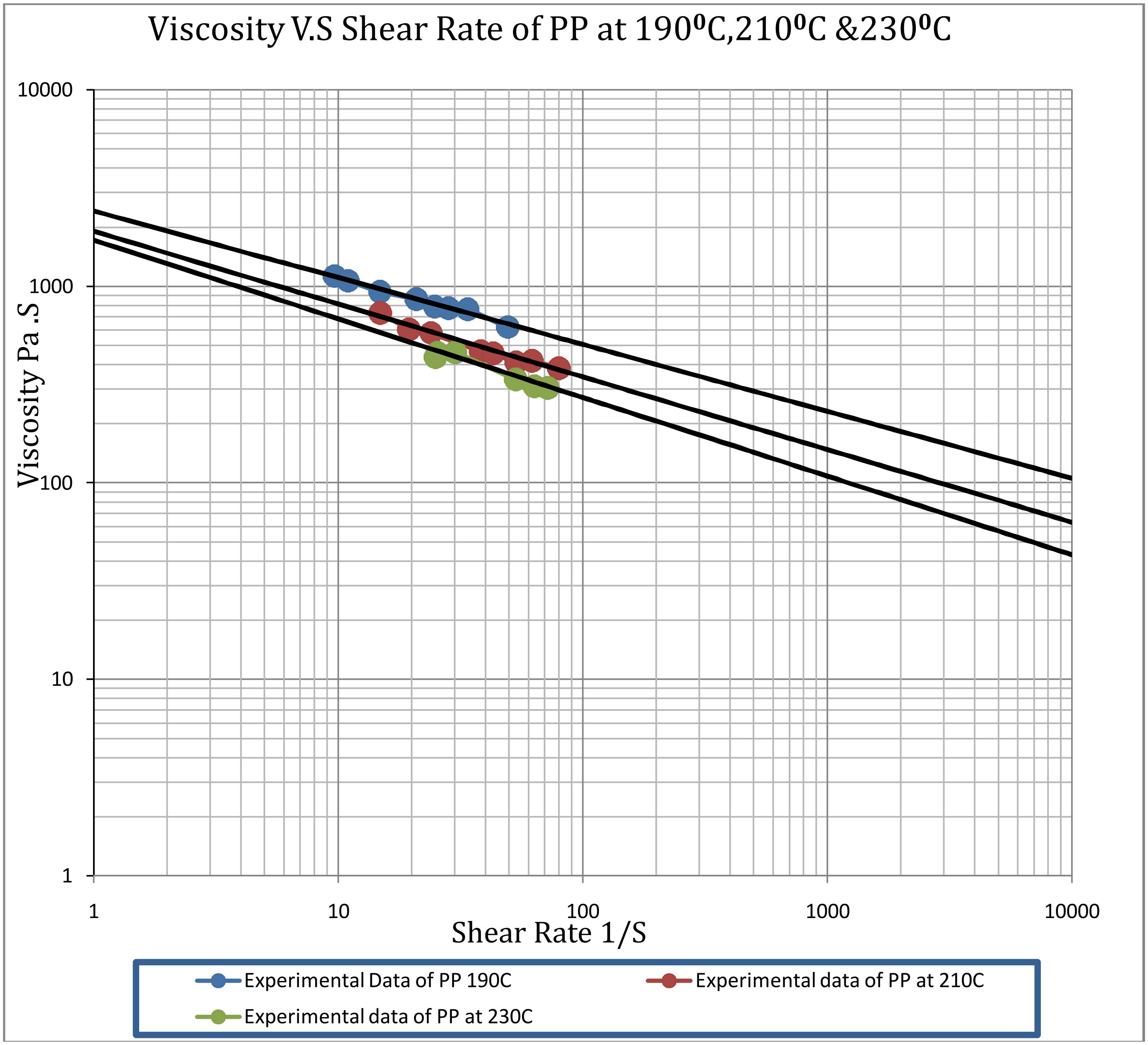


Figure 4-9 Experimental viscosity for PP with power law model

c- Drawing the value of MFI experimental of PP at 190^{0}C, 210^{0}C &230^{0}C with applied load .

Figure 4-10 shows the implication of MFI on shear viscosity. The plot shows that increasing MFI causes a decrease in shear viscosity, which is expected. A faster flowing polymer shoud have a higher MFI and lower viscosity.

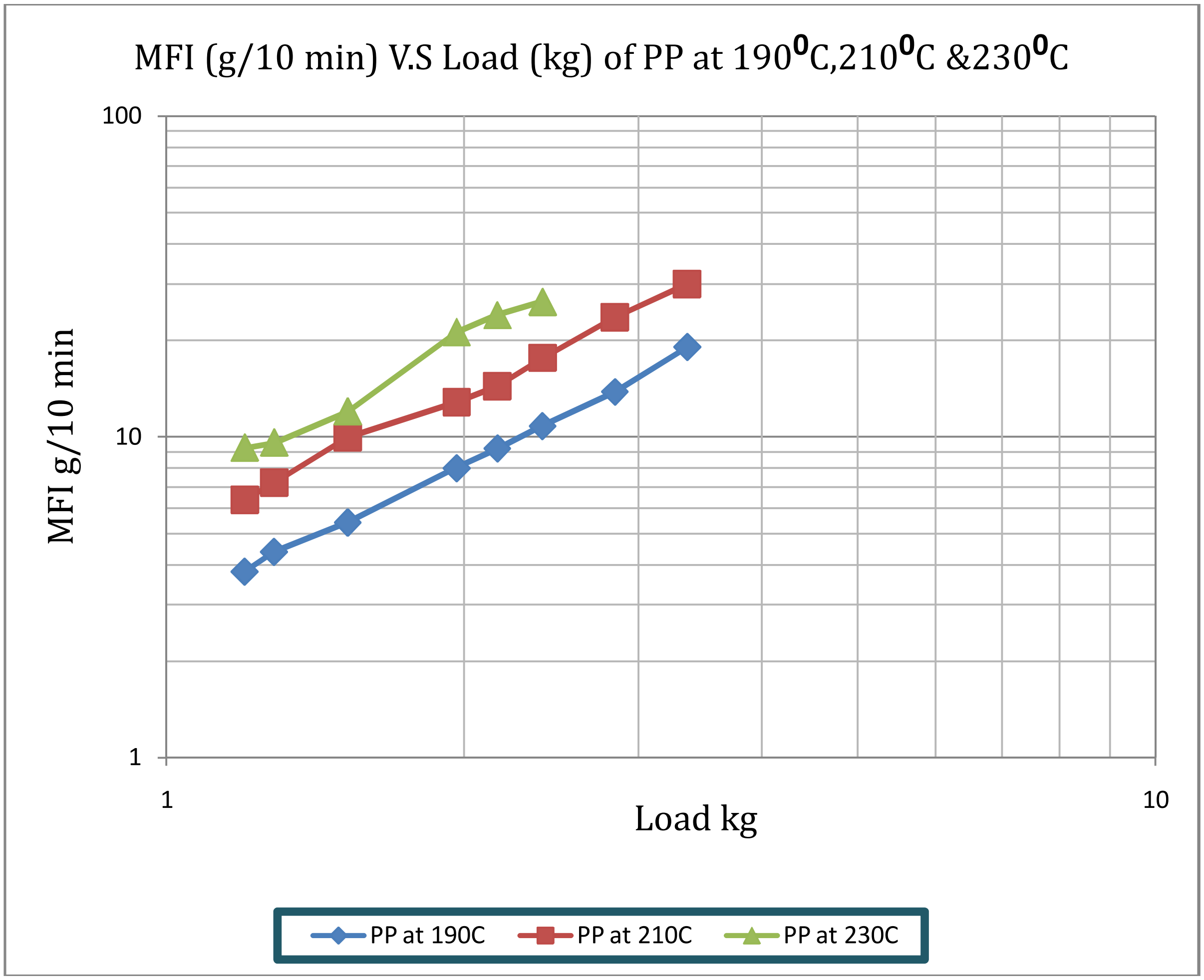


Figure: 4-10. Relation between Melt flow index MFI (g/10min) and load (kg) for pp

d- Drawing the Experimantal data of viscosity with Carreau Law shown in figuer 4-11.

Note when the values of Shear strain rates between 1 to 10 the viscosity very regular or almost equal at the same temperature, but at shear rates greater than the 10 S^{-1}, the values of viscosity decreasing that curves up to the extent of Interproximal.See appendices (B) table 4.18.

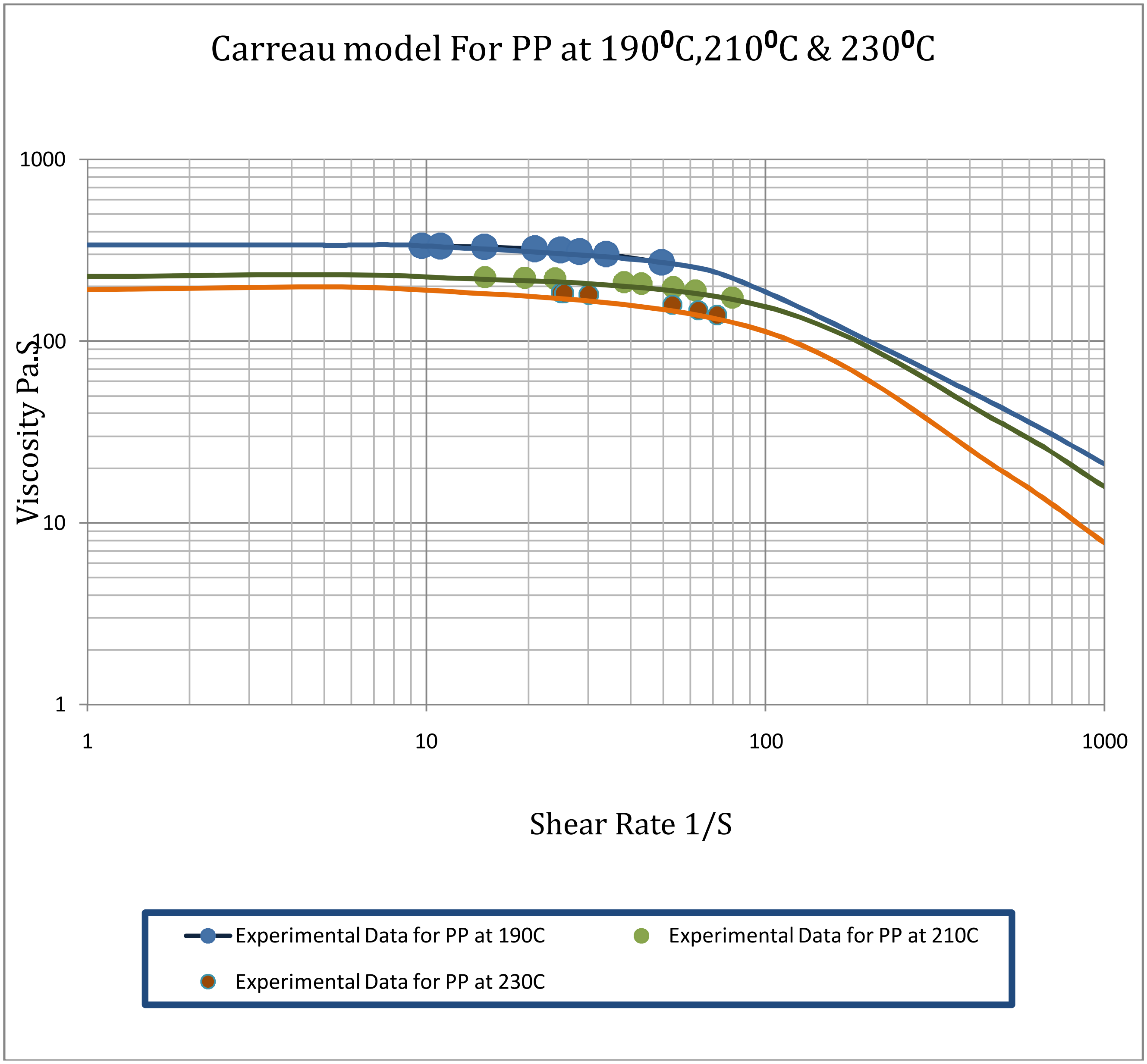


Figure 4-11 Experimental viscosity for PP with carreau model

CHAPTER FIVE

CONCLUSIONS AND RECOMMENDATIONS

The Conclusions of this study are summarized as follows:

The melt flow index test is widely used in the industry for quality control purposes and it is not used for the viscosity study. In this research I tried to study the viscosity of thermoplastics using MFI.

The melt flow index testing device was used to measure viscosity of thermoplastics: Polyethylene (PE) and poly propylene (PP) where the MFI is indirect relation with the shear stress and strain.

In order to get the relation between shear strain rate and viscosity six different weights are used that is: 2.16, 2.4, 2.84, 3.36, 3.80 and 5 kg for LDPE and eight different weights are used for PP that is: 1.2, 1.285, 1.525, 1.965, 2.16, 2.40, 2.84 and 3.36kg.

For PE six curves were drawn for the temperature 170^0C, 190^0C and 210^0C the data for these curves appear in the appendices(A) tables 4.1, 4.2, 4.3, 4.4, 4.5 and 4.6. Similarly for PP six curves were drawn for the temperatures 190^0C, 210^0C and 230^0C the data for these curves appear in the appendices (B) table 4.10, 4.11, 4.12, 4.13, 4.14 and 4.15.

The above data were analyzed and studied to get the parameters in each rheological model for these:

1. Power law model
2. Carreau model
3. Arrhenius model.

These parameters vary with temperature variation. The variations in these parameters are slight ones.

Further studies are recommended for these parameters to determine whether it is a fixed value or there is variation that should be considered.

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APPENDICES
A
(Low density polyethylene)

Table 4.1Measured melt flow index for LDPE at 170 ^{0}C

Measured Results								
Sample	*Load (Kg)*	*Force (N)*	*Length (mm)*	*Time (Sec)*	*Mass average (g)*	MFI $(g/10min)$	Density (Kg/m^3)	MVR $(cm^3/10min)$
1	2.16	21.19	5.14	60	0.097	0.97	798	1.22
2	2.40	23.54	6.40	60	.0.12	1.20	791	1.52
3	2.84	27.86	7.33	60	0.153	1.53	885	1.73
4	3.36	32.96	10.94	60	0.20	2.00	771	2.59
5	3.80	37.28	12.57	60	0.25	2.50	839	2.98
6	5.00	49.05	20.57	60	0.40	4.00	821	4.87

Table 4.2Measured melt flow index for LDPE at 190 ^{0}C

Measured Results								
Sample	*Load (Kg)*	*Force (N)*	*Length (mm)*	*Time (Sec)*	*Mass average (g)*	MFI $(g/10min)$	Density (Kg/m^3)	MVR $(cm^3/10min)$
1	2.16	21.19	9.74	60	0.18	1.8	779	2.31
2	2.40	23.54	12.75	60	0.22	2.2	730	3.02
3	2.84	27.86	16.66	60	0.28	2.8	710	3.95
4	3.36	32.96	21.57	60	0.37	3.7	724	5.11
5	3.80	37.28	26.55	60	0.45	4.5	714	6.29
6	5.00	49.05	40.57	60	0.73	7.3	761	9.62

Table 4.3Measured melt flow index for LDPE at 210 ^{0}C

Measured Results								
Sample	*Load (Kg)*	*Force (N)*	*Length (mm)*	*Time (Sec)*	*Mass average (g)*	MFI $(g/10min)$	Density (Kg/m^3)	MVR $(cm^3/10min)$
1	2.16	21.19	17.08	60	0.32	3.2	780	4.10
2	2.40	23.54	25.37	60	0.39	3.9	639	6.10
3	2.84	27.86	30.00	60	0.53	5.3	746	7.10
4	3.36	32.96	41.46	60	0.65	6.5	665	9.76
5	3.80	37.28	52.97	60	0.95	9.5	759	12.50
6	5.00	49.05	51.81	60	1.35	13.5	732	18.44

Table 4.4 Calculated Results of LDPE at 170 ^{0}C

Calculated Results							
Sample	Force (N)	$\dot{V}$ $10^{-9}(\frac{m^3}{s})$	$\dot{G}$ $10^{-6}(\frac{kg}{s})$	Δp (Mp_a)	τ_f (Mp_a)	η (Mp_a)	$\dot{\gamma}$ (S^{-1})
1	21.19	2.03	1.62	0.300	0.01965	0.00873	2.251
2	23.54	2.53	2.00	0.333	0.02181	0.00777	2.807
3	27.86	2.88	2.55	0.395	0.02587	0.00810	3.194
4	32.96	4.32	3.33	0.467	0.03059	0.00639	4.787
5	37.28	4.97	4.17	0.529	0.03465	0.00629	5.509
6	49.05	8.12	6.67	0.696	0.04559	0.00506	9.009

Table 4.5 Calculated Results of LDPE at 190 ^{0}C

Calculated Results							
Sample	Force (N)	$\dot{V}$ $10^{-9}(\frac{m^3}{s})$	$\dot{G}$ $10^{-6}(\frac{kg}{s})$	Δp (Mp_a)	τ_f (Mp_a)	η (Mp_a)	$\dot{\gamma}$ (S^{-1})
1	21.19	3.85	3.00	0.300	0.01965	0.00460	4.27
2	23.54	5.03	3.67	0.333	0.02181	0.00391	5.58
3	27.86	6.58	4.67	0.395	0.02587	0.00354	7.25
4	32.96	8.52	6.17	0.467	0.03059	0.00323	9.47
5	37.28	10.50	7.50	0.529	0.03465	0.00292	11.67
6	49.05	16.00	12.17	0.696	0.04559	0.00256	17.81

Table 4.6 Calculated Results of LDPE at 210 ^{0}C

Calculated Results							
Sample	Force (N)	$\dot{V}$ $10^{-9}(\frac{m^3}{s})$	$\dot{G}$ $10^{-6}(\frac{kg}{s})$	Δp (Mp_a)	τ_f (Mp_a)	η (Mp_a)	$\dot{\gamma}$ (S^{-1})
1	21.19	6.83	5.33	0.300	0.01965	0.00259	7.59
2	23.54	10.17	6.50	0.333	0.02181	0.00193	11.30
3	27.86	11.83	8.83	0.395	0.02587	0.00197	13.13
4	32.96	16.27	10.83	0.467	0.03059	0.00169	18.10
5	37.28	20.83	15.83	0.529	0.03465	0.00149	23.26
6	49.05	30.73	22.50	0.696	0.04559	0.00133	43.28

Table 4.7 to calculate the activation energy of LDPE at different temperature

	A	η_0 Pa.S	K^{-1}	R^2
LDPE at 1700C $\eta_0 = 9986\ e^{-0.07\dot{\gamma}}$	9986	7328.17	2.257	0.946
LDPE at 190^{0}C $\eta_0 = 4954\ e^{-0.04\dot{\gamma}}$	4954	3462.17	2.160	0.912
LDPE at 210^{0}C $\eta_0 = 2687\ e^{-0.02\dot{\gamma}}$	2687	1905.33	2.070	0.868

Table 4.8 to calculate the viscosity of LDPE by using power law at different temperature.

Power Law : $\eta = \bar{K}\dot{\gamma}^{n-1}$

Material	η	$\bar{K}$	n
LDPE at 170^{0}C	$12092\dot{\gamma}^{-0.39}$	12092	0.61
LDPE at 190^{0}C	$7937\dot{\gamma}^{-0.39}$	7937	0.61
LDPE at 210^{0}C	$5868\dot{\gamma}^{-0.42}$	5868	0.58

Table 4.9 to calculate the viscosity of LDPE by using carreau model at different temperature.

Carreau model: $\eta_a = \eta_0 * a_T\,[\,1 + a_T(\,A_t * \dot{\gamma}_a\,)^2\,]^{\frac{n-1}{n}}$

$$\log aT = \frac{C_1(T_1 - T_0)}{C_2 + (T_1 - T_0)} - \frac{C_1(T_2 - T_0)}{C_2 + (T_2 - T_0)}$$

Material	η_0 Pa.S	a_T	A_t	n
LDPE at 170^{0}C	5060	0.716	0.111	0.61
LDPE at 190^{0}C	2560	0.741	0.056	0.61
LDPE at 210^{0}C	1330	0.764	0.029	0.58

APPENDICES

B

(Polypropylene)

Table 4.10Measured melt flow index for PP at 190 ^{0}C

Measured Results								
Sample	*Load (Kg)*	*Force (N)*	*Length (mm)*	*Time (Sec)*	*Mass average (g)*	MFI (g $/10min$)	Density (Kg $/m^3$)	MVR (cm^3 / $10min$)
1	1.2	11.77	3.72	10	0.063	3.80	722	5.26
2	1.285	12.60	4.20	10	0.073	4.38	739	5.93
3	1.525	14.96	5.65	10	0.090	5.40	673	8.02
4	1.965	19.27	7.98	10	0.133	7.98	706	11.30
5	2.16	21.19	9.53	10	0.153	9.18	680	13.50
6	2.40	23.54	10.77	10	0.180	10.80	697	15.30
7	2.84	27.86	13.10	10	0.230	13.80	753	18.33
8	3.36	32.96	18.80	10	0.317	19.02	712	26.71

Table 4.11Measured melt flow index for PP at 210 ^{0}C

Measured Results								
Sample	*Load (Kg)*	*Force (N)*	*Length (mm)*	*Time (Sec)*	*Mass average (g)*	MFI (g $/10min$)	Density (Kg $/m^3$)	MVR (cm^3 / $10min$)
1	1.2	11.77	2.88	5	0.053	6.36	781	8.10
2	1.285	12.60	3.70	5	0.060	7.20	684	10.50
3	1.525	14.96	4.60	5	0.083	9.96	764	13.09
4	1.965	19.27	7.28	5	0.107	12.84	718	20.70
5	2.16	21.19	8.23	5	0.120	14.40	615	23.40
6	2.40	23.54	10.16	5	0.147	17.64	609	28.90
7	2.84	27.86	11.80	5	0.197	23.64	703	33.59
8	3.36	32.96	15.22	5	0.250	30.00	693	43.30

Table 4.12Measured melt flow index for PP at 230 ^{0}C

Measured Results								
Sample	*Load* *(Kg)*	*Force* *(N)*	*Length* *(mm)*	*Time* *(Sec)*	*Mass average* *(g)*	MFI $(g/10min)$	Density (Kg/m^3)	MVR $(cm^3/10min)$
1	1.2	11.77	4.75	5	0.077	9.20	680	13.52
2	1.285	12.60	4.83	5	0.080	9.60	698	13.75
3	1.525	14.96	5.73	5	0.100	12.00	735	16.31
4	1.965	19.27	10.11	5	0.177	21.20	738	28.78
5	2.16	21.19	12.14	5	0.200	24.00	695	34.55
6	2.40	23.54	13.70	5	0.220	26.40	676	38.99

Table 4.13 Calculated Results of PP at 190 ^{0}C

Calculated Results							
Sample	Force (N)	$\dot{\mathrm{V}}$ $10^{-9}(\frac{\mathrm{m}^3}{\mathrm{s}})$	$\dot{\mathrm{G}}$ $10^{-6}(\frac{\mathrm{kg}}{\mathrm{s}})$	$\Delta\mathrm{p}$ $(\mathrm{Mp_a})$	τ_f $(\mathrm{Mp_a})$	η $(\mathrm{Mp_a})$	$\dot{\gamma}$ (S^{-1})
1	11.77	8.77	6.33	0.167	0.0109	0.001125	9.69
2	12.60	9.88	7.30	0.178	0.0117	0.001065	10.99
3	14.96	13.37	9.00	0.212	0.0139	0.000937	14.83
4	19.27	18.83	13.30	0.273	0.0179	0.000857	20.89
5	21.19	22.50	15.30	0.300	0.0196	0.000788	24.87
6	23.54	25.83	18.00	0.334	0.0219	0.000774	28.29
7	27.86	30.55	23.00	0.395	0.0259	0.000764	33.90
8	32.96	44.52	31.70	0.467	0.0306	0.000619	49.43

Table 4.14 Calculated Results of PP at 210 ^{0}C

Calculated Results							
Sample	Force (N)	$\dot{\mathrm{V}}$ $10^{-9}(\frac{\mathrm{m}^3}{\mathrm{s}})$	$\dot{\mathrm{G}}$ $10^{-6}(\frac{\mathrm{kg}}{\mathrm{s}})$	$\Delta\mathrm{p}$ $(\mathrm{Mp_a})$	τ_f $(\mathrm{Mp_a})$	η $(\mathrm{Mp_a})$	$\dot{\gamma}$ (S^{-1})
1	11.77	13.50	10.6	0.167	0.0109	0.000731	14.9
2	12.60	17.50	12.0	0.178	0.0117	0.000601	19.5
3	14.96	21.68	16.6	0.212	0.0139	0.000578	24.0
4	19.27	34.57	21.4	0.273	0.0179	0.000467	38.3

5	21.19	38.97	24.0	0.300	0.0196	0.000455	43.1
6	23.54	48.28	29.4	0.334	0.0219	0.000409	53.5
7	27.86	55.97	39.4	0.395	0.0259	0.000417	62.1
8	32.96	72.05	50.0	0.467	0.0306	0.000383	79.9

Table 4.15Calculated Results of PP at 230 ^{0}C

Calculated Results							
Sample	Force (N)	$\dot{V}$ $10^{-9}(\frac{m^3}{s})$	$\dot{G}$ $10^{-6}(\frac{kg}{s})$	Δp (Mp_a)	τ_f (Mp_a)	η (Mp_a)	$\dot{\gamma}$ (S^{-1})
1	21.19	22.62	15.4	0.300	0.01965	0.000436	25.00
2	23.54	22.88	16.0	0.333	0.02181	0.000459	25.49
3	27.86	27.17	20.0	0.395	0.02587	0.000461	30.15
4	32.96	48.03	35.4	0.467	0.03059	0.000336	53.27
5	37.28	57.47	40.0	0.529	0.03465	0.000309	63.43
6	49.05	64.98	44.0	0.696	0.04559	0.000304	72.04

Table 4.16 to calculate the viscosity of PP by using power law at different temperature

Power Law $: \eta = \bar{K}\dot{\gamma}^{n-1}$

Material	η	$\bar{K}$	n
PP at 190^{0}C	$2421\dot{\gamma}^{-0.34}$	2421	0.66
PP at 210^{0}C	$1903\dot{\gamma}^{-0.37}$	1903	0.63
PP at 230^{0}C	$1715\dot{\gamma}^{-0.42}$	1715	0.60

Table 4.17 to calculate the viscosity of PP by using carreau model at different temperature

Carreau model: $\eta_a = \eta_0 * a_T \left[1 + a_T(A_t * \dot{\gamma}_a)^2 \right]^{\frac{n-1}{n}}$

$$\log aT = \frac{C_1(T_1 - T_0)}{C_2 + (T_1 - T_0)} - \frac{C_1(T_2 - T_0)}{C_2 + (T_2 - T_0)}$$

Material	η_0 Pa.S	a_T	A_t	n
PP at 190^{0}C	619	0.546	0.020	0.66
PP at 210^{0}C	383	0.593	0.0125	0.63
PP at 230^{0}C	304	0.634	0.0139	0.60