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Effect of recycling on the rheological properties of LDPE

A Thesis

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By:

Saba Hamed Shaker Kreem

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Supervised by

Prof.Dr. Nizar Jawad Hadi

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Abstract

This work focuses on reducing the damage of the plastic west by recycle of low density polyethylene LDPE. A 30g of LDPE was extruded for three cycles at 190 °C and range shear rates (1-1000) s^{-1} to obtain three samples using capillary rheometer. Rheological and physical tests of three samples of the LDPE were tested. The relation between viscosity and shear stress with shear rate were analyzed. Density, hardness and Differential scanning calorimeter (DSC) were also tested for all samples.

The results show that viscosity decreases and shear stress increases with shear rate increasing for all sample. In general, viscosity curve consist of two stages. The first stage in the range (1-200) s^{-1} the viscosity decreases rapidly, while the viscosity in the second stage decreasing gradually and the LDPE melt tends to be Newtonian.

The density and the hardness tests showed a little change with the increases of cycle's number. The color and transparency of three samples were approximately the same. DSC test results show the melting temperature for three samples is 113°C. The results of numerical simulation were obtained the same velocity and pressure counters for the three samples.

بــسم الله الـرحمن الـرحيم

سورة الزمر /آية {9}

صدق الله العلي العظيم

Supervisors Certification

We certify that this thesis entitled (**Effect of Recycling on The Rheological properties of Polymer**) was prepared by (Saba Hamed Shaker) under our supervision at University Babylon / College of Materials Engineering/Department of polymer and petrochemical industries, in Partial Fulfillment of the Requirements for the Award High diploma Degree of Science in Materials Engineering / Polymers Engineering.

Name: Prof. Dr. Nizar Jawad Hadi

(Supervisor)

Date: / / 2022

Signature:

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Praise, thank, and gratitude to the benefactor and Single-handed **ALLAH** who enabled and helped me to achieve this work.

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Dedication

To
My Father and Mother
My Husband Ms. Muhanned Lewaa
My Daughter Zahraa
My dear supervisors (Prof. Dr. Nizar Jawad Hadi with all respect

My friends

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Symbols

Sample	Meaning	Unite
n	Power law index	
K	consistency index	Pa.sn
μ	Viscosity	(pa*s)
T	Temperature	C°
V	Velocity	(m/s)
γ	Shear rate	(s^{-1})
T_m	Melting temperature	C°
Tg	Glass transition temperature	C°

1. 1 Introduction

In recent years industries are getting more interested in the field of plastic manufacturing; so many commodities are being manufactured with plastics. Plastics have become a crucial part of lifestyle, and the global plastic production has increased immensely during the past 50 years. Plastic pollution is becoming the second biggest global environmental concern after climate change [1]. Statistics show that from 1950 to 2017, the global cumulative production of plastic is about 9.2 billion tons; till 2050, the global cumulative production of plastic will reach 34 billion tons, and the annual production of plastic waste is about 300 million tons. Now many countries in the world put a lot of manpower and material resource to develop various waste plastic recycling key technology and devote to reduce plastic recycling cost and suitable application field. At present, waste plastic recycling mainly includes energy recycling, physical recycling and chemical recycling [2].

1.2 Plastic solid waste (PSW)

Plastic solid waste (PSW) of polymers (like: high density polyethylene (HDPE), low density polyethylene (LDPE), Nylon etc.) is creating new challenges, which in today's scenario are major research concerns. A sharp rise has been observed in production of different products based on different plastic material. This huge increase in plastic commodities also increases the waste generation thus creating new challenges. Since they are non-biodegradable waste and the disintegration of the plastic in nature takes more than 400 years. Besides, according to Ocean Conservancy, 60% of seabirds and almost all sea turtles consume plastic waste [3] as shown in figure (1.1).
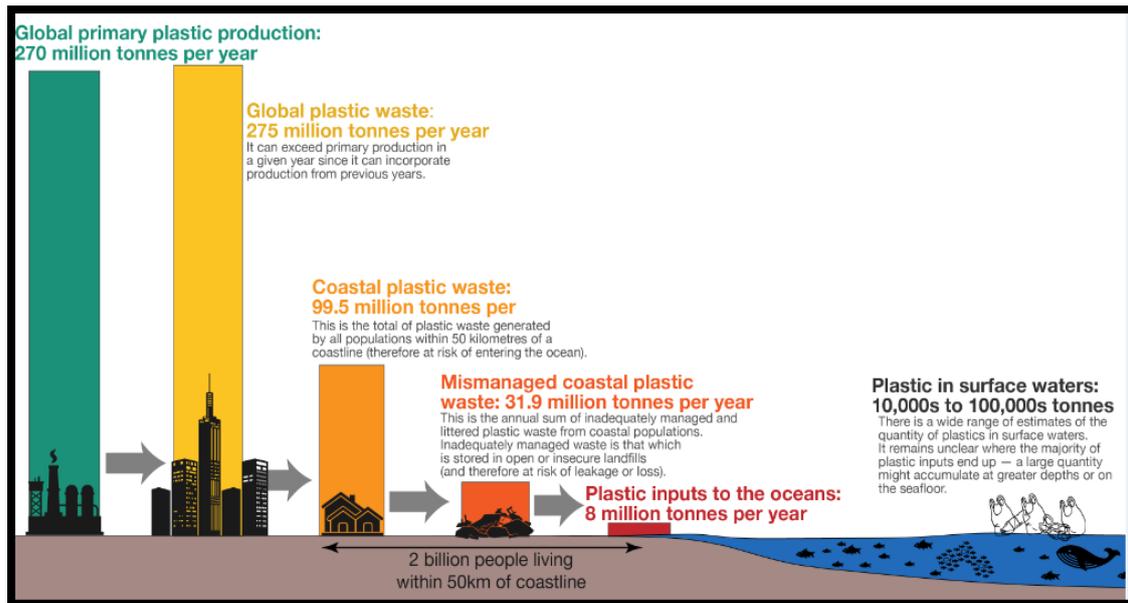
Figure (1.1): The Pathway by which plastic enters the world's oceans [3]

Sustainable development SD, according to its general concept, which is to preserve the environment from pollution, requires reusing materials correctly and safely. Therefore, recycling LDPE contributes to achieving the goal of SD [4]. In Europe, it is stated that low-density polyethylene (LDPE) constitutes 23% of the total plastic waste. Polypropylene (PP), high-density polyethylene (HDPE), polystyrene (PS), polyvinyl chloride (PVC), polyethylene terephthalate (PET) wastes are the remaining part of the contaminant materials. If it is not possible to prohibit the manufacturing of plastic, minimizing or eliminating plastic waste is necessary [5].

Recycled Polymer/plastic is highly used in manufacturing industries for the preparation of products. Industries are more interested in cost reduction, hence using recycled material is better choice for cost reduction and also helpful in reducing waste. Polymers are an excellent and a very useful material to replace ceramic, wood and metals because they are very functional, hygienic, light and economical [6].

Apart from cleaning up our oceans or landfills, which is a very significant step, the best way to address this huge plastic pollution is by recycling the plastic wastes. Plastic recycling is the process of collecting plastic wastes and reprocessing the material into useful products. Before reprocessing them, most plastics are sorted according to codes. There are represented by Unicode icons and numbered with (1) Polyethylene Terephthalate (PET), (2) High-Density Polyethylene, (3) Polyvinyl Chloride (PVC), (4) Low-Density Polyethylene (LDPE), (5) Polypropylene (PP), (6) Polystyrene (PS), and (7) other plastic types [7]. These resin codes indicate the type of material of each plastic and each of them has a unique material characteristic. Some of them are reusable, some are hazardous after several uses, and some need a more sophisticated recycling process. By understanding these seven codes, it will be easier to choose plastics and to know which plastics to be recycled. Due to their differences in material properties and characteristics, it is recommended to use a singular type of plastic in a production of a new product using recycled materials [8].

1.3 The reason for using polyethylene for recycling

In this study LDPE thermoplastic was used as this is inexpensive, lightweight and durable. LDPE is normally used as plastic bags, is considered less toxic than other plastics. This plastic is the easiest plastics to recycle. LDPE has the second highest share of installed recycling capacity as shown in figure (1.2). These unique properties make it suitable to be moulded into a variety of products for many applications, even after they have been recycled into a new form [9].

Low density polyethylene (LDPE), a thermoplastic, is a material that can be repeatedly molded and shaped when heated. This material is particularly flexible because of the many side chains that increase the

distance between the core C-C chains. Numerous containers, tubes, plastic bags, and agricultural covers are often made from LDPE.

Low-density polyethylene may also have a number of detrimental effects on the environment and human health if it is discovered in rubbish. Therefore, the best way to avoid negative effects is to recycle it for sustainable development [10].
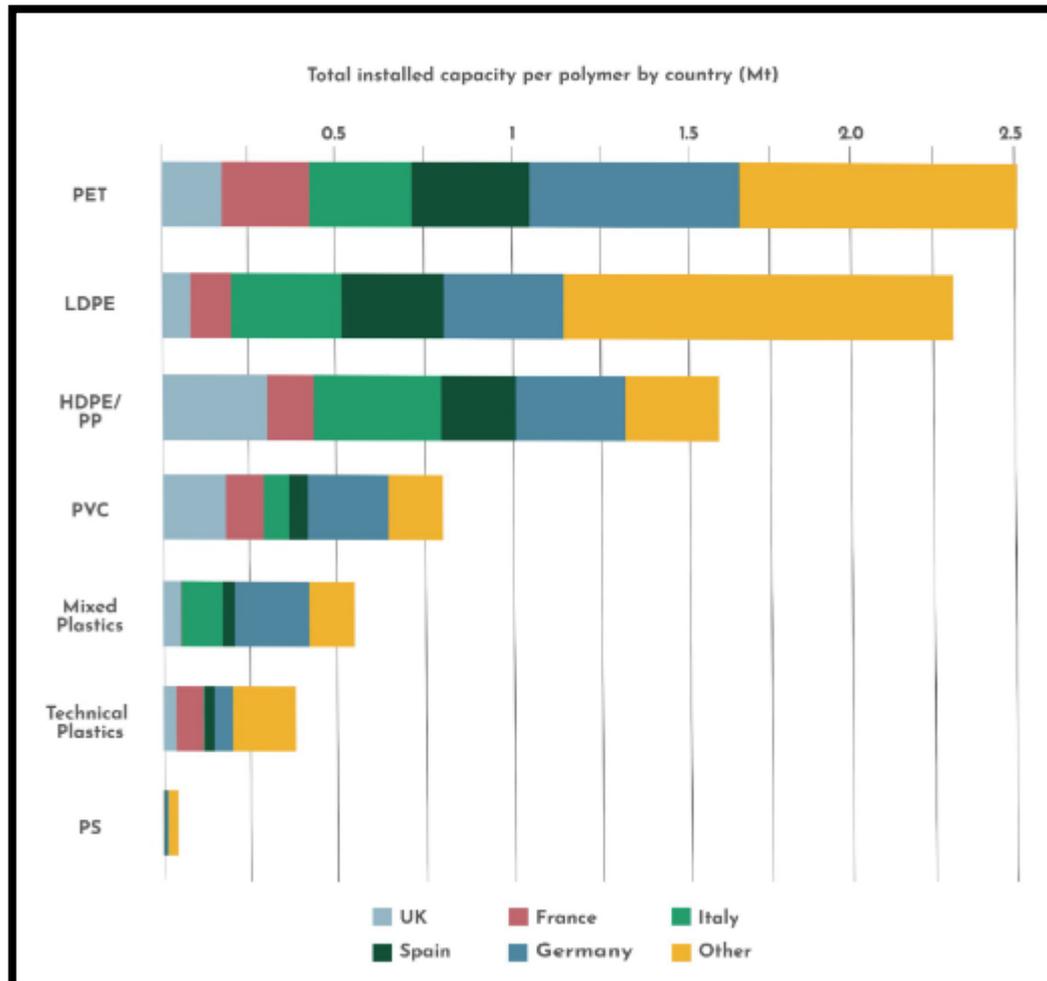
Figure (1.2): LDPE has the second highest share of installed recycling capacity Europe wide [9].

2.1The aims

The sustainability of LDPE is the subject of this research using the following techniques:

Study the effect of recycle number on the rheological and physical properties of recycled LDPE.

2.2 The objectives

Simulate the flow behavior of LDPE at each cycle using ansys program.

2.1.   Plastics Recycling

One way to lessen the effect on the environment and resource depletion is to recycle plastics. Recycling may consequently reduce the amount of energy and materials used to produce a given amount of output, improving eco-efficiency. Recycling is the only approach to reduce the environmental issues brought on by the buildup of polymeric waste from everyday uses of polymer materials, such as those used in building and packaging. The majority of polymer materials are generated from oil and gas, which helps to preserve natural resources [25].

Recycling is the last stage in the collection, type-specific separation, and polymer processing. It lessens the amount of rubbish that is carelessly tossed away and dumped in landfills. It thus contributes for variety of issues, like the spread of illnesses and the deterioration of resources for the earth, air, and water [26]. Now one of the most crucial possibilities accessible for reducing these one of these effects is one of the most and one of the most active markets for plastics. It provides opportunities to decrease oil consumption, carbon dioxide emissions, and the quantity of trash that has to be disposed of.

2.2.   Solid Municipal Waste

Since the capacity MSW landfills decreases and the burning of plastics might produce harmful flies and bottom ash including lead & cadmium, the increase in plastic garbage has a substantial influence on MSW management strategies such as landfilling and incineration [27].

Recycling plastic waste is one method of reducing the net amount of MSW discarded. Even though the advantages haven't been measured, recycling Plastics might be used to result in measurable reductions in the consumption of fossil fuels because they can in contrast to "virgin" resins

made developed fossil fuels because of the energy needed to produce them perhaps lower than that required to produce corresponding virgin resins feedstock. Recycling plastic waste is a very simple method for greatly reducing the overall amount of MSW because it uses less material and energy [29].

The primary plastics recycled include high-density polyethylene (HDPE), low-density polyethylene (LDPE), polypropylene (PP), polyethylene terephthalate (PET), poly vinyl chloride (PVC), polystyrene (PS), and polycarbonate (PC).

Table (2.1) List the recycling codes for recyclable polymers [30].

Symbol	Acronym	Full name and uses
1	PET	Polyethylene terephthalate - Fizzy drink bottles and frozen ready meal packages.
2	HDPE	High-density polyethylene - Milk and washing-up liquid bottles
3	PVC	Polyvinyl chloride - Food trays, cling film, bottles for squash, mineral water and shampoo.
4	LDPE	Low density polyethylene - Carrier bags and bin liners.
5	PP	Polypropylene - Margarine tubs, microwave-able meal trays.
6	PS	Polystyrene - Yoghurt pots, foam meat or fish trays, hamburger boxes and egg cartons, vending cups, plastic cutlery, protective packaging for electronic goods and toys.
7	Other	Any other plastics that do not fall into any of the above categories. For example melamine, often used in plastic plates and cups.

2.3. Stages of Plastic Recycling

Recycling occurs at different phases, including:

1. Collection: Plastic trash is gathered from various places. Special containers may be kept at homes, public spaces, farms, and other locations to do this. Professional garbage haulers then gather these wastes and deliver them to the recycling facilities.

2. Cleaning: The plastic products are washed and dried at this procedure. Since clean waste resources sell for more money and raise the caliber of finished goods, cleaning is crucial. Several times throughout the

recycling method, including before, after and even while sorting, plastics may be cleaned.

3. Sorting: Both the polymers themselves and recoverable foreign items must be separated throughout this procedure.

4. Reducing the size of trash is the goal of size reduction facilitates the division of various polymers and the recovering the finely ground powder that serves as the feedstock for processing equipment. Plastic bits with erratic shapes that may be sold to businesses and workshops that specialize in reprocessing can be the end result of shredding. Blow molding, extrusion, injection molding, and film molding are just a few of the procedures these materials go through after being treated. After that, the raw materials are turned into a range of commodities, such as tubes, sacks, sheets, pipelines, and other things [28].

2.4.   Recycling Techniques

Some recycling methods include primary recycling, secondary recycling by mechanical means, tertiary recycling by chemical means, and quaternary recycling by energy recovery as shown in figure (2.1).

- Primary recycling entails using the same product in a new use cycle without making any significant modifications (Refillable containers, for instance, after cleaning).

- Mechanical recycling is the use of waste materials for new purposes without changing their chemical structure.

- Chemical recycling denotes a change in the substance's chemical composition, which allows the produced chemicals to be utilized to create the original material once more [30].
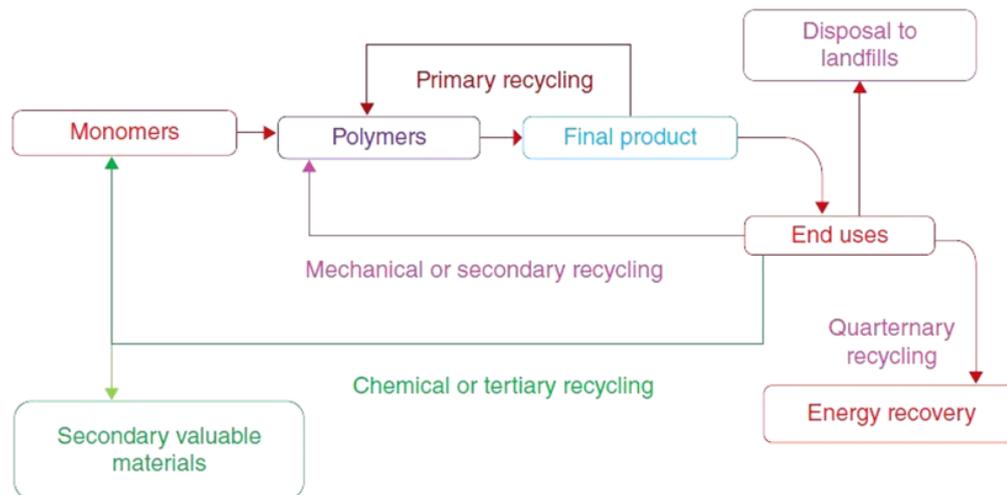
Figure (0.1): Four recycling techniques [30]

• Energy recovery is the process of recovering the energy included in plastic. The best method for reducing the amount of organic materials right now is incinerating materials with the intention of recovering energy. Due to the health danger posed by airborne harmful compounds like dioxins, this technology has been extensively criticized as being environmentally undesirable even if polymers are high-yielding energy sources (in the case of polymers that include chlorine) [28].

2.5. Benefits of Reusing and Recycling Polymers

Reuse and recycling are methods for handling plastic product waste at the end of their useful lives. These are becoming more and more environmentally and economically sensible. Recent trends show a significant rise in the rate of plastic trash recovery and recycling. The following are the main benefits:

1. Recycling aids in the most balanced form of environmental protection.

2. Recycling lessens the quantity of garbage that must be disposed of in landfills or burned.

3. Recycling resources like glass, metals, and other materials helps to reduce the pollution that would otherwise be created during the manufacture of new goods.

4. Reusing polymers also reduces energy consumption since it requires less energy to create a product from recycled materials than it does from raw ones.

5. Because it requires less energy and natural resources, reusing plastic is better than recycling. Some plastic products are just used once (have a single life or cycle), or are used only once and then end up in the trash stream (e.g., food packaging).

6. Since the creation of plastic consumes 4-8% of the world's oil output (4% for the raw material and 4% for the conversion), recycling plastic helps the environment.

7. Recycling lowers energy consumption and MSW production.

8. Recycling reduces emissions of sulfur dioxide (SO2), nitrogen dioxide (NO_2), and carbon dioxide (CO_2) [33].

2.6. Low-Density Polyethylene (LDPE)

The highly branched type of low-density polyethylene (LDPE) was first synthesized [34]. Regarding the quantity of energy or power required creating items and, in many applications, such as film blowing, LDPE is simpler to process [35, 36]. Along with a few long-chain branches, LDPE has short-chain propyl and butyl branches. However, LDPE has a considerable market share because of its excellent processing capacity. Some of the characteristics of LDPE are shown in Table (2.2). The commercial polymer low-density polyethylene (LDPE) is widely used in extrusion processes such coating, blown film, blow molding, and foaming. Polyethylene extrusion coating has been utilized extensively in the commercial sector on substrates including paper, various plastic films,

fabric, and glass fiber. The advantages of coating with polyethylene include increased tear and crease resistance; a good barrier against moisture, grease, and oil, flexibility, non-toxicity, and low coating cost.

Table (0.2): List of low-density polyethylene (LDPE) Properties [38].

Properties	Value	Unit
Density	0.91 – 0.93	g/cm^3
Melt temperature (T_m)	115	°C
Glass transition temperature (T_g)	about - 130	°C
Thermal conductivity	0.35	$Wm^{-1}K^{-1}$
Water absorption at saturation	<0.01	%
Elongation	50 – 800	%
Elastic modulus	0.1 – 0.3	GPa
Critical strain energy rate (G_c)	34	kJ/m^2
Thermal linear expansivity	5 – 25	$10^{-5}K^{-1}$

LDPE is a significant weight proportion of the poly olefins prevalent in waste streams. The typical mechanical characteristics of this polymer, which is further affected by the product's aging, make it uninteresting for certain applications. Its use is restricted because LDPE wastes are typically generated from bags and pallet covers and recycled as trash bags. If the poor mechanical qualities of certain types of LDPE wastes can be enhanced by the addition of other materials, new markets for them may be created [38].

2.7. Rheological properties

The scientific discipline of rheology investigates the behavior of fluids under flow-induced deformation. Rheologists have researched a wide range of materials, but they have found that polymers are the most fascinating and challenging. Shear thinning polymer melts are viscoelastic and their flow properties depend on temperature. When determining the behavior of polymers during processing, viscosity is the most commonly used material parameter. Shear rate dominates most polymer operations; hence the viscosity of the melt is often evaluated using shear deformation measurement equipment. Thermoforming, blow molding, and fiber spinning are examples of polymer processes where elongational deformation or a mix of shear and elongational deformation predominates. Additionally, certain polymer melts exhibit significant elastic effects during deformation [39].

2.7.1. Viscosity of Shear

Viscosity represents the shearing resistance, i.e. the forced flow of imaginary fluid slices like a card deck. It is the most significant property for polymer processing. Referring to figure (2.2), can define viscosity as the ratio of the imposed shear stress (τ) (force F applied tangentially, divided by the area A) and the shear-rate (γ) (velocity U divided by the gap h) [40]:

$$\eta = \frac{shear\ stress}{shear\ rate} = \frac{F/A}{U/h} = \frac{\tau}{\gamma} \qquad\qquad (2.1)$$

This correlation is known as Newton's law of viscosity. One Pa.s is equivalent to ten poises when expressing the viscosity (η) in Pa.s.
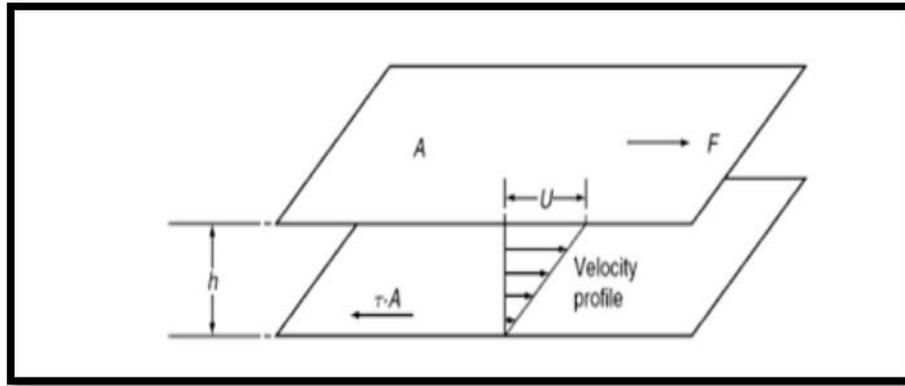
Figure (0.2): Shear flow between two plates [41].

A fluid is called Newtonian when the viscosity is independent of the shear rate. Molten polymers have shear-related viscosities, display several other uncommon flow properties, and are called non-Newtonian. Their shear-thinning conduct (also known as pseudo plastic conduct) is a notable property of polymeric liquids (melts and solutions). As the shear-rate rises, as shown in figure (2.3) the viscosity reduces. This viscosity decrease is due to the lengthy polymer chains molecular alignments and disentanglements. The greater the shear rate, the simpler it is to flow through dies and processing facilities for polymers [40].
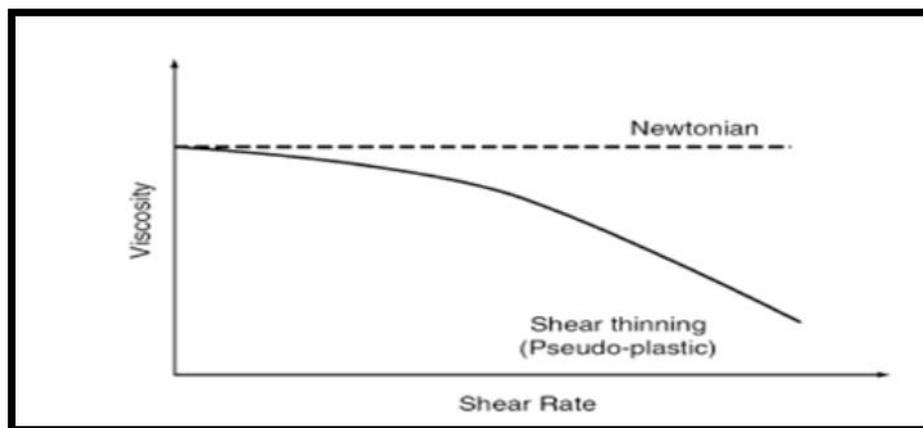
Figure (0.3): Shear thinning viscosity behavior and Newtonian behavior [40].

There are several straightforward empirical formulae to investigate the shear rate based on viscosity, including:

1. Power low (Ostwald and de Waele).

2. Law of Carreau-Yasuda.

3. Law of Bird-Carreau.

4. Law of Cross.

5. Law of Bingham.

6. Law of Log-Log.

7. The law of modified Bingham [42].

Study shear-thinning phenomena using the power law model in this thesis.

2.8.   Phenomena of Flow

Polymeric liquids have three key characteristics that set them apart from basic fluids:

1. Normal shear flow stresses.

2. Non-Newtonian viscosity.

3. Elastic effects.

Due to their complicated molecular structure, polymer macromolecules cause all of these effects [39].

2.9.   Behavior of Non-Newtonian Fluids

Non-Newtonian substances that behave nonlinear along a flow curve (shear rate vs. shear stress). The apparent viscosity and shear stress at temperature and pressure are two flow conditions that are influenced by shear rate and die shape. These resources are grouped into three broad categories: Time-independent fluids are those whose shear rate at any given position is solely dictated by the shear stress value at that precise moment in time. These fluids include: -

1. Standardized fluids of Newtonians.

2. Fluids that change over time have a more complex connection between shear stress and shear rate.

3. Viscoelastic fluids; materials exhibiting properties of both ideal fluids and elastic solids and partial elastic recovery after deformation [39].

2.9.1. The Time-Independent of Fluid Behavior

It is possible to describe the flow behavior of this class of materials under a simple shear using a constitutive relationship of the form [45]:

$$\tau = f\,(\dot{\gamma}) \tag{2.2}$$

Where $\dot{\gamma}$ is the shear rate and τ is the shear stress (pa) (s^{-1}).

This equation states that the present shear stress at any location inside the sheared fluid is the only value that can be calculated there, or vice versa. These fluids may be further divided into three groups based on the kind of function in equation (2.2):

(a) Shear - thinning, pseudo plastic.

(b) Viscoelastic.

(c) Shear - thickening, dilatant.

For these three different forms of fluid behavior on linear scales, Figure (2.4) illustrates the linear connection characteristic of Newtonian fluids [47].
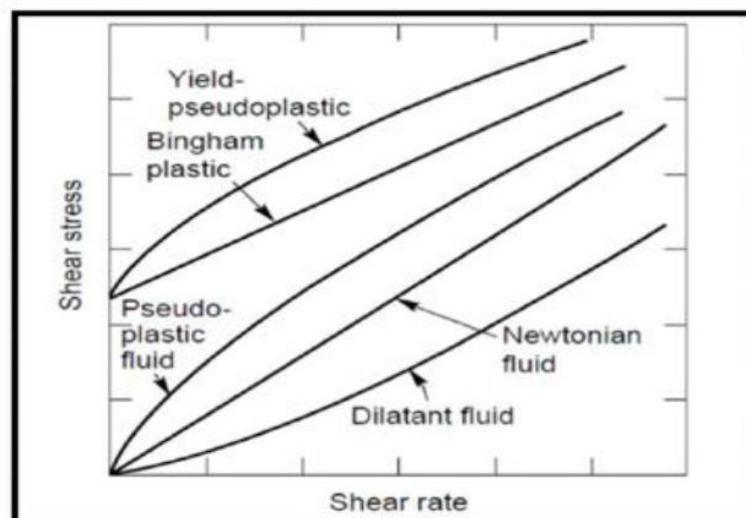
Figure (0.4): Types of time-independent flow behavior [45].

2.9.1.1. Shear-Thinning or Pseudo Plastic Fluids

Pseudo plasticity or shear thinning, defined by an apparent viscosity that reduces with rising shear rate, is the most common form of time-independent non-Newtonian fluid behavior observed. Most shear thinning at very small and very high shear rates.
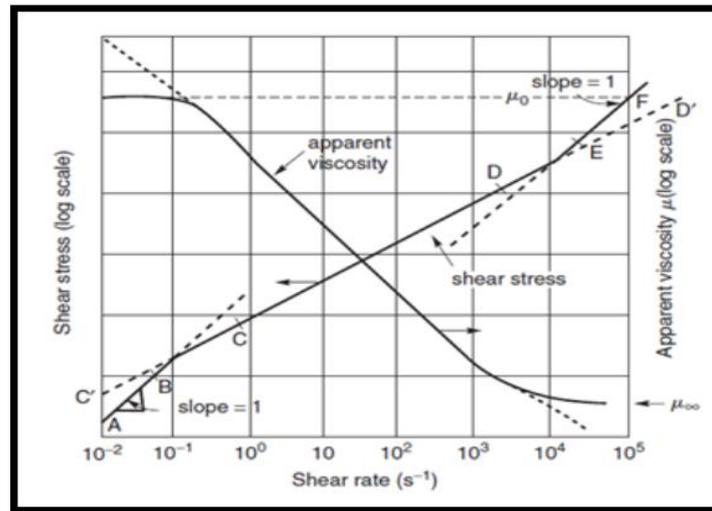
Figure (0.5): Shear thinning behavior is shown schematically [46].

As shown schematically in figure (2.5), Because of the non-Newtonian behavior of polymer solutions and melts, shear stress shear rate graphs turn into straight lines and, on a linear scale, cross the origin. The zero-shear viscosity η_0, and an infinite shear viscosity η_∞, are the resultant values of the apparent viscosity for extremely low and large shear rates, respectively η_0 to η_∞. Finding data that indicate the complete spectrum of pseudoplastic behavior over a large enough range of shear rates is difficult [45].

2.10. Power Law (Ostwald and De Waele)

Is an easy-to-understand and practical model, but it misses the Newtonian plateau at low strain rates on the strain rate curve, which appropriately portrays the shear thinning area in the viscosity vs. This is one way to represent the power law model[39]:

$$\eta = K \, (\gamma^{\cdot})^{\, n-1} \tag{2.3}$$

The fluid has shear-thinning characteristics for n< 1.

The fluid exhibits Newtonian behavior when n = 1.

The fluid exhibits shear-thickening behavior when n > 1.

Where n stands for the power law index, K for the consistency index, and for the viscosity η. The power law index depicts the shear-thinning behavior of the polymer melt, and the consistency index may incorporate the viscosity's temperature dependency [39].

The true shear rate for the power law model is:

$$\gamma^{\cdot}_{\text{true}} = \left(\frac{3n+1}{4n}\right) \gamma^{\cdot}_{\text{app.}} \tag{2.4}$$

Where:

$\gamma^{\cdot}_{\text{true}}$: Authentic shear rate.

n: Index of a power law.

$\gamma^{\cdot}_{\text{app.}}$: Predictable shear rate.

2.11. Measurement of Rheological Properties by Capillary Rheometer

A capillary rheometer is a device intended to assess the shear viscosity and other characteristics of rheology. Capillary rheometers as shown in figure (2.6) are piston die systems that are intended to assess the viscosity of polymer melts as a result of temperature and deformation rate. They can test fundamental polymers, compounds, multiple composites with small strengthening particles or fibers, metal injection molding feedstock and similar materials [47].

The capillary is the small cylinder channel through which during the experiment the materials flow. It is defined by a length and a diameter

and the ratio of the two named L/ D. It is machined by the die, and sometimes the L/ D is immediately referred to the die [48].

The temperature, sample, shape and preheating parameters refer to the indication given for the basic rheological test. All other parameters affecting the strength of the melt are analyzed as follows:-

1. Melting temperature: The temperature has a significant impact on both the stretching filament's breaking strength and its melting characteristics.

The melting strength of the molten polymers increases when the experiment's temperature is lowered.

2. Die dimensions, which are the same as for basic rheological testing, have an impact on the force of drawing, which decreases with die diameter.

3. Initial velocity: This velocity is that of the extruder just as it leaves the die after swelling; the extruder hasn't yet undergone a significant drawing. Alternative settings for this initial velocity include pulley start rate (mm/s), output mass flow rate (g/min), and piston velocity (mm/s).

4. Stretching velocity and acceleration: The velocity that the drawing unit imposes on the shorter end of the extrudate is known as the stretching velocity, which is a function of acceleration and correlates to the velocity increase.

5. The length of the drawing is determined by the distance between the die's exit and the spot where the first extruded material comes into contact with the revolving drawing units.

6. Load cell: A force transducer attached to the pulley of the draw force detector measures the melting force of the polymer.

7. Speed pulleys: Common pulleys used in melt stretching tests are composed of rubber and don't have any issues with sticking or winding up [48].
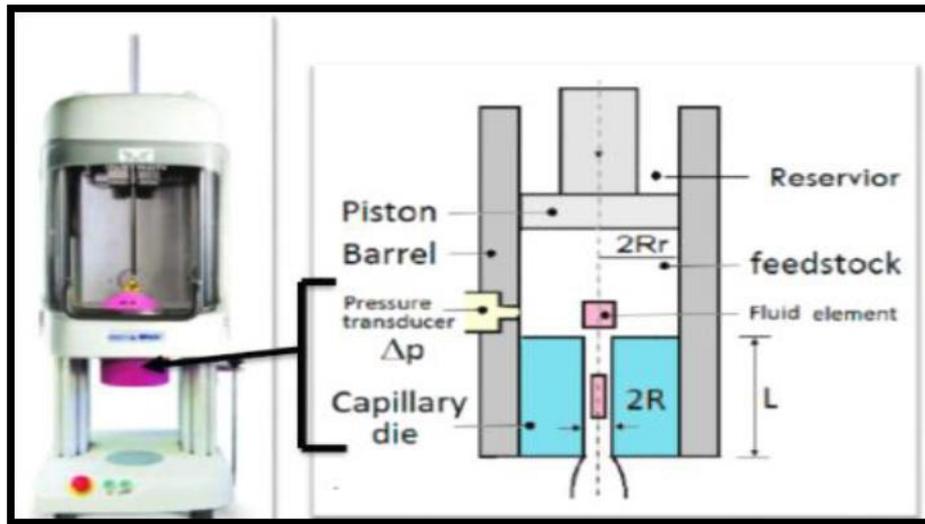
Figure (0.6): Capillary rheometer SR20 reservoir and die area [49].

The relationship between flow rate and pressure drop through the die needs to be measured because pressure drops may be influenced by the flow through the extruder barrel and, more specifically, by frictional losses between the piston and barrel wall. It is much preferable to measure the pressure drop in the melt immediately above the die rather of relying on the pressure drop brought on by the force required to move the piston [49].

2.11.1. Apparent Capillary flow-induced shear viscosity

In the vast majority of typical experiments, viscosity and shear can be used to derive the Hagen-Poiseuille law for capillary flow [50]:

$$\tau_{app.} = \frac{R\,P}{2\,L} \tag{2.5}$$

$$\dot{\gamma}_{app.} = \frac{4Q}{\pi R^3} \tag{2.6}$$

$$\eta_{app} = \frac{\tau_{app}}{\gamma_{app}} \tag{2.7}$$

Where R is the capillary radius (m), P stands for pressure drop, Q for volume flow rate, and L for capillary length (m). By utilizing the appropriate tools, it is easy to measure shear rates in the range of 0.01 to

$5*10^5$ s^{-1}. Controls the piston's speed or control the shear rate are the two operational modes that have been employed (requires the volumetric flow rate to be measured). The Poiseuille equation yields an apparent rather than a true estimate of viscosity at the operating temperature when used to analyze capillary rheometry data [50].

2.11.2. Molten polymer flow instability

Extrusion instability may be seen while a molten polymer passes through a die. The extrude is uniform in cross-section and smooth at low flow rates, and the flow is quite steady. Figure illustrates the primary flaws that were discovered (2.7). Surface defects, also known as sharkskin, oscillation flaws, also known as stick-slip defects, and melt fracture defects, which may be more or less ordered, are the three categories that are often identified: A consistent helical form may be seen in certain extrudates. Others have a completely jumbled appearance (2.7 D). High extrusion velocities may cause waves in the extrudate in areas where the polymer is not allowed to relax. This phenomena, which is often referred to as shark skin, is seen in figure (2.7.a) for high density polyethylene. It is possible to extrude at such high rates that the internal die walls and melt sometimes separate, as shown in figure (2.7.b). This phenomenon, also known as the stick slip effect or the spurt flow, is caused by an increased shear stress between the polymer and the die wall. When the shear stress approaches the critical value of 0.1 MPa, this event occurs. If the velocity is increased even further, a helical shape is extruded for a polypropylene extrudate, as illustrated in figure (2.7.c). When the speeds reach a certain point, a chaotic pattern similar to the one in figure (2.7.d) begins to form. This well-known occurrence is known as melt fracture. Shark skin effects are often missing, and spurt flow seems to only occur with linear polymers [39].
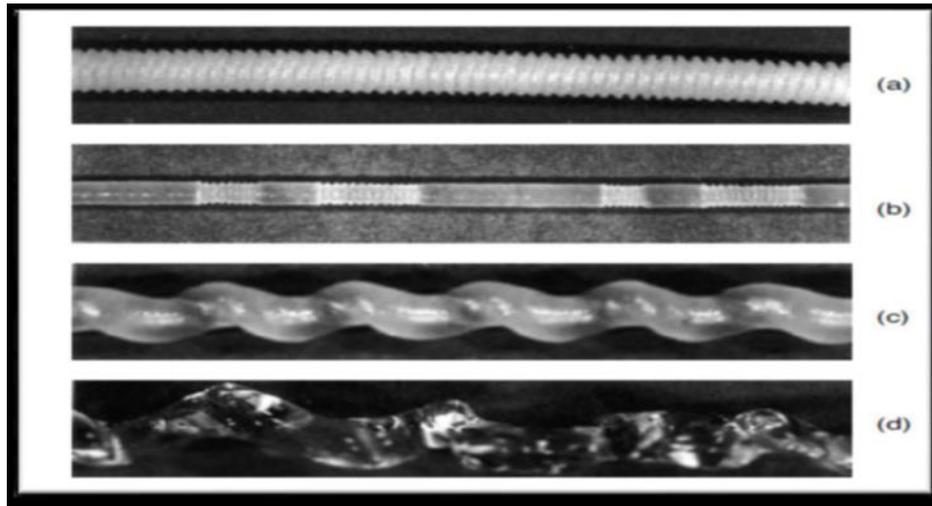
Figure (0.7): Example of extrusion defects: a) Shark skin, b) Oscillating defect,
and, c, d) Volume defects [39].

2.12. Polymer Mechanical Properties

The mechanical characteristics are often the most crucial considerations when choosing a product's specs. Tensile strength, modulus, elongation, and impact strength are just a few of these qualities that are important when choosing a material for various end-use applications [51]. Because they are viscoelastic, polymers stress their position as a middle ground between viscous liquid and elastic solid. Hooke's low is seen in an ideal linear elastic solid, where stress operates prior to strain. Newtonian's law, which states that stress operates prior to strain rate of change, is followed by an ideal viscous. The temperature and the duration of any deformation have a significant impact on the mechanical characteristics of polymers. A polymer may be glass-like at low temperatures, with a Young's modulus value in the range of 10^9-10^{10}Pa, and it will break or yield at stresses larger than a few percent. With a modulus in the range of 105-106 Pa and the ability to resist significant expansions of order 100% or more without permanently deforming, it may behave rubber-like at high temperatures or low

frequencies. The polymer may permanently distort under stress and act like an extremely viscous liquid at even greater temperatures. The polymer is neither glassy nor rubber-like in the so-called glass-transition region, where it possesses an intermediate modulus and viscoelastic characteristics [52]. All of the primary mechanical characteristics of polymers are influenced by structural elements in addition to chemical composition, including the following:

1. Molecular weight.

2. Branching and cross-linking.

3. Copolymerization (block, graft, and random).

4. Molecular orientation.

5. Fillers.

In addition to the structural and molecular factors listed above, the following environmental or external variables are important in determining mechanical behavior:

1. Temperature.

2. Frequency, time, rate of stressing or straining.

3. Pressure.

4. Type of deformation (shear, tensile, biaxial) [53].

2.12.1. Property of Hardness

Hardness is characterized as a solid material's resistance to cutting, scratching, wear, indentation, penetration, and workability. In other words, the ability of tools and machines to maintain a material's surface intact and holding together while being affected by external forces ensures that the material won't permanently deform [54]. This ability is demonstrated by the material's resistance to scratching by tools and machines that are harder than it. The sort of bonding force between

molecules or atoms, the type of material surface to which hardness should be evaluated, temperature, and other factors all determine how hard a material is. In the past, materials' hardness was determined by their capacity to cut through other materials; the hardest substance is the one that can do this. The most significant techniques for determining hardness are:

1. Brinell hardness

2. Vickers hardness

3. Rockwell hardness

4. Shore Hardness (A, D) [55].

2.13 Numerical analysis (CFD Simulation using Ansys 2021).

In order to model the flow of low-density polyethylene through the capillary tube, computational fluid dynamics (CFD) is used. The finite element or finite volume model, which accurately depicts all the underlying physical processes, would be the greatest substitute for an analytical solution after that. By doing numerical simulations, computational fluid dynamics (CFD) approaches have been utilized to examine and assess the impacts of repeatedly reheating polymers as well as other systems involving fluid dynamics and heat transport.

Computational Fluid Dynamics (CFD) consists from:

1. Pre-processor.

2. Solver.

3. Post-processor.

2.13.1. Pre-processor

Pre-processors consist of:

a) Creation an interest-defining grid.

b) Describe the chemical and physical qualities that need simulating.

c) An explanation of fluid characteristics

d) Application boundary conditions.

2.13.2. Solver

The numerical computations required to provide accurate simulations of the flow issue are carried out by Solver. The solver only used spectral, finite element, finite size, and finite difference approaches. The key distinctions between these strategies may be seen in how the estimating process and flow variables are approximated. These are based on the usage of FLUENT, a commercial CFD software program that is well-known for its extensive capabilities in the solution of various fluid flow issues using a finite volume strategy.

2.13.3. Post-processor

The post-processor contains the simulation results that were computed by the solver. The majority of the CFD systems now on the market have created graphical capabilities that enable the viewing of the derived data. The examples that follow: -

a) Vector plots of the velocity field.

b) Path of a particle through the domain.

c) Contour plots.

d) Fluid flow animations.

e) View manipulation (translation, rotation and scaling etc.).

2.14. Literatures Review

In 2013 Nizar, J, et.al, Studied the rheological attitude of waste the PP-Runner mixes for use in syringe production for medical applications. Rheological properties of polypropylene (PP) injected with different

volumes of waste (runner).To get the same qualities needed for the manufacture of medical syringes (cylinder part) [19].

In 2015 Nizar, Displayed the morphological and rheological properties of calcium carbonate nanoparticle-reinforced virgin and used polypropylene. The slower speed results in good nanoparticle dispersion and less agglomeration through the polymer matrix. Higher levels of crystallinity and lower MFR values result from less agglomeration and more nanoparticle dispersion [20].

In 2017 David Hui, et.al, Mentioned there are four main approaches for recycling of plastic solid waste (PSW); as primary, secondary, tertiary and quaternary recycling. Recycling technique of polymer does play an important role in the generation of new polymer. Each technique has its own advantages and disadvantages. When material undergoes a recycling process it starts losing some of properties in terms of tensile strength, wear properties and dimensional accuracy [56].

In 2018, Ravindra. R , et .al Used the incorporated CNTs in recycled PET act as heterogeneous nucleating agents, resulting in an increase in the degree of crystallinity and crystallization temperature. Incorporation of CNTs into recycled PET slightly improves the thermal stability and glass transition temperature [22].

In 2019 Pooja. T , et .al, Examined different technological processes have been reported for recycling of polymers such as chemical or feedstock recycling with energy recovery, and mechanical recycling. The recycling process involves new development and separation techniques for waste polymers with novel energy-recovery procedures for effective cost management from an economical standpoint [23].

In 2020 R.K , Dr.Nizar.J , et .al, Investigated the mechanical and rheological behavior of waste PET, LDPE, and HDPE that had been combined for use in bio-pipes. For all mixes, the shear stress rises and the shear viscosity decreases as shear rate increases. When RPET is added the viscosity at each shear rate in relation to the reference mix is decreased. Density increases as more RPET is incorporated into the benchmark mixture [24].

In 2021 Laongdaw Techawinyutham, et. al, Cleared the plastic processing plants can decrease and add value of their post-processed plastic wastes by producing plastic product from recycled LDPE/PETG and HDPE/PETG with high PETG loading [57].

In 2022 Nicholas G, et. al Discovered a PSO Experimenter was used which enables to find the optimal parameter setting for recycling LDPE materials. The results of the study show that PSO Experimenter is a free, easy-to-use and effective program for finding optimal dynamic memory parameters and can also used in a wide range of other fields and processes [58].

3. 1. Introduction

This chapter includes:

Listing the properties of LDPE used in this study. Describing the procedure used for recycling process of LDPE. Sample preparation. Testes: rheological test, mechanical tests, thermal tests and other tests.

3.2. The used material

3.2.1 Low Density Polyethylene (LDPE)

The pellets of low density polyethylene (LDPE), manufactured by Amir Kabir Petrochemical Copany (AKPC), are shown in the Figure (3.1). The following table (3.1) displays the LDPE's properties.
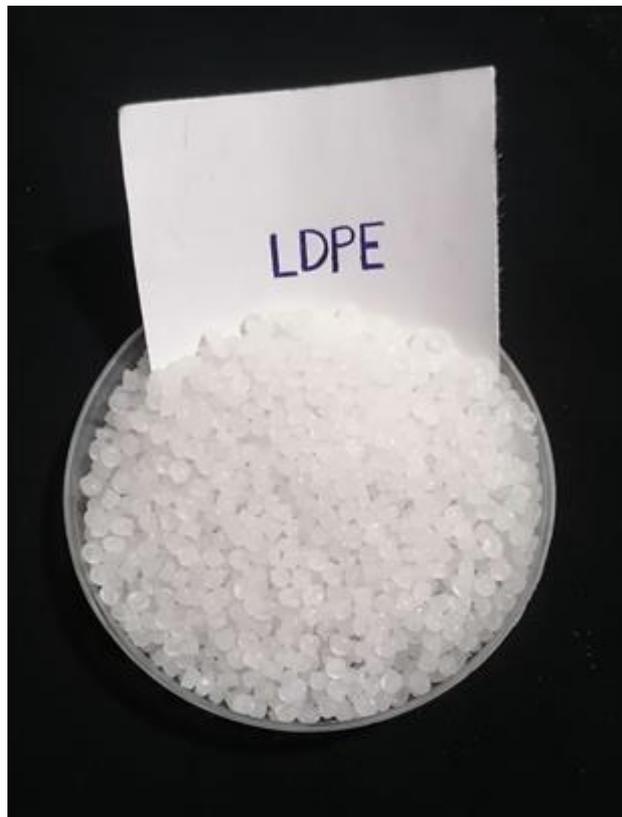

Figure (0.1): Low density polyethylene (LDPE).

Table (0.1): LDPE properties.

Property	Value
Density	0.923 g/cm^3
Melt flow rate (MFR)(190°C/2.16Kg)	0.25 g/10 min
Tensile Stress at Yield	10 MPa
Glass transition temperature (Tg)	-130°C
Melting temperature (Tm)	113°C

3.2.2 Procedure of the work:

The procedure of recycling process of LDPE illustrated in figure (3.2).
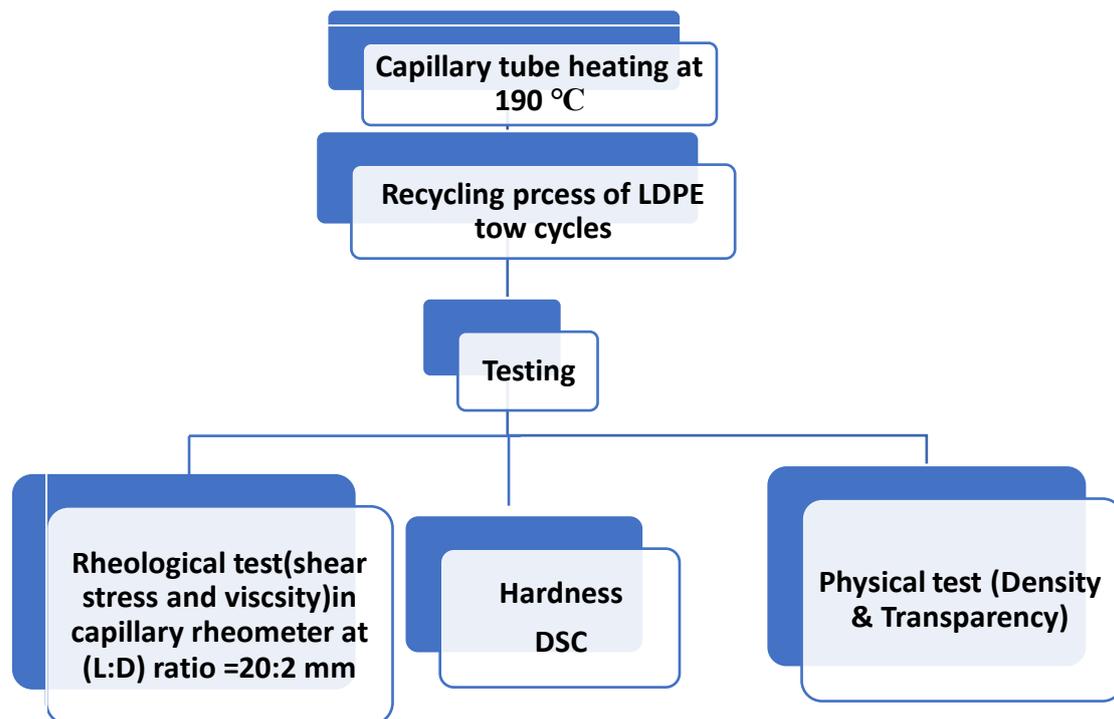

Figure (3.2): block diagram.

3.2.3 The LDPE Recycling Process

In this study, low-density polyethylene recycling was carried out using a capillary rheometer, as seen in the Figure (3.3). After the LDPE specifications were established and the data was prepared in the device's software, 30g of virgin polymer granules were added to the apparatus. The data included polymer shown in table (3.2).

Table (3.2): LDPE data used in capillary software.

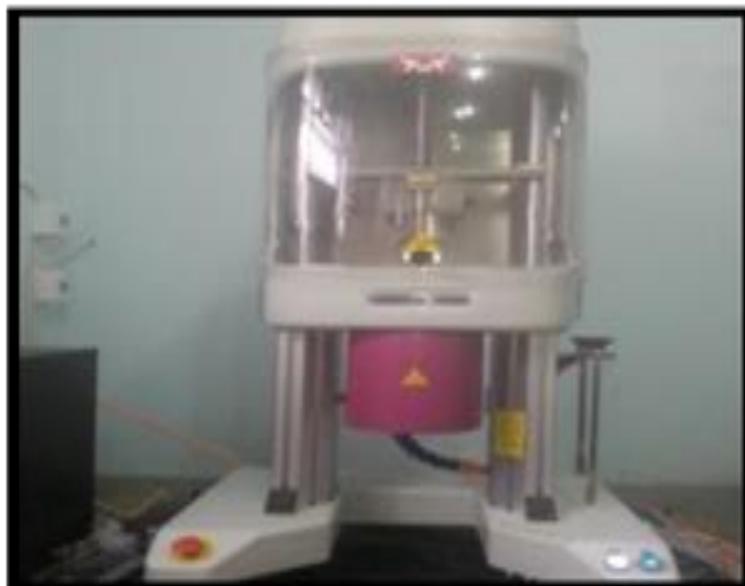| Data	Value
density (ρ)	0.925 g/cm^3
mold dimensions L/ d	20 / 2 mm
force F	300 N
preheat	500 sec
temperature T	190 °C
shear rate γ	1, 5, 10, 50, 100, 200 s^{-1}

Figure (0.3): Capillary tube device.

The polymer is chopped into granules of about 2 mm in size in the first sample of LDPE. The material then extruded once again via the capillary tube. Each time get a thread, it is sliced into granules and fed to the device's nozzle that is responsible for inter the material.

3.3 Tests

Physics tests

3.3.1. Density

As shown in figure (3.4), density is tested at room temperature in accordance with ASTM D-792 using a Matsu Haku High Accuracy DENSITY TESTER GP-12OS with a digital accuracy of 0.0001 g/cm^3. This tool calculates density according to Archimedes' rule. The sheet, rod, tube, molded items, and powders may all be employed using this technique. A sinker and wire were used to retain the sample totally immersed as needed while it was being weighed in distilled water and air at room temperature.

Figure (0.4): Density Tester GP-12OS.

3.3.2. Hardness

To examine the impact of cycles on the hardness of LDPE, a hardness test (Shore D instrument) as shown in figure (3.5), using the ASTM D-2240 standard was carried out on the sample at room temperature. For each sample, an average of three readings must be calculated in three areas, with the needle of the shore instrument positioned perpendicular to the sample. This process takes 0.5 minutes. Three samples were used, with comparable findings.

Figure (0.5): Show D hardness test.

3.3.3. Infrared spectroscopy IR.

The infrared spectrum of a polymer is a profile of its absorption characteristics plotted against infrared wavenumber (or frequency). It is generated when infrared radiation interacts with the molecular moieties that constitute the polymer material. The IR spectroscopy theory utilizes the concept that molecules tend to absorb specific frequencies of light that are characteristic of the corresponding structure of the molecules.

KBr (potassium bromide) is used as an optically transparent material in infrared spectroscopy, thus it is used as a carrier in the form of a tablet or pellet.

3.3.4. Differential Scanning Calorimeter DSC

DSC is often used to pinpoint the temperatures of transitions of polymeric materials. The glass transition temperature (T_g), crystallization temperature (T_c), and melting temperature are significant thermal transitions (T_m). The observed thermal transitions can be utilized to compare materials, although the transitions alone do not uniquely identify composition. Standard compilations provide the melting and glass transition points for the majority of polymers, and by lowering the projected melting temperature.

3.4 Numerical analysis(CFD Simulation using Ansys 2021).

3.4.1 Modeling processing

The modeling process using ANSYS workbench fluid flow (fluent). The main objective of the numerical analysis in this work is simulating the flow of LDPE in capillary tube and study the effect of cycle number of LDPE on the flow properties.

Assumption:

- Fluid flow (fluent).

- Transient laminar flow.

- Heat flow via a capillary tube using LDPE.

- Non-Newtonian power law model is necessary for continuous material flow.

$$\mu = \ k\gamma^{.n-1} \dots\dots\dots\dots\dots\dots\dots\dots (3.1)$$

μ: Viscosity (pa*s).

k: Consistency index.

γ: Shear rate is 1/s.

n: Flow index.

3.4.2 Design modular

The geometry was sketched in 2-D micro capillary. The dimension of micro capillary was similar to the original capillary used in this research figure (3.6). One inlet and one outlet were drawn.

Steps of Model sketching:

1. Open ANSYS fluent design modular and changing units of drawing to millimeters.
2. Notify the XY-Plane and create a rectangle. Next, draw a smaller rectangle at the bottom to show the material flow, including the inlet and outlet.
3. Modification takes connected corners using term and fillet commands.
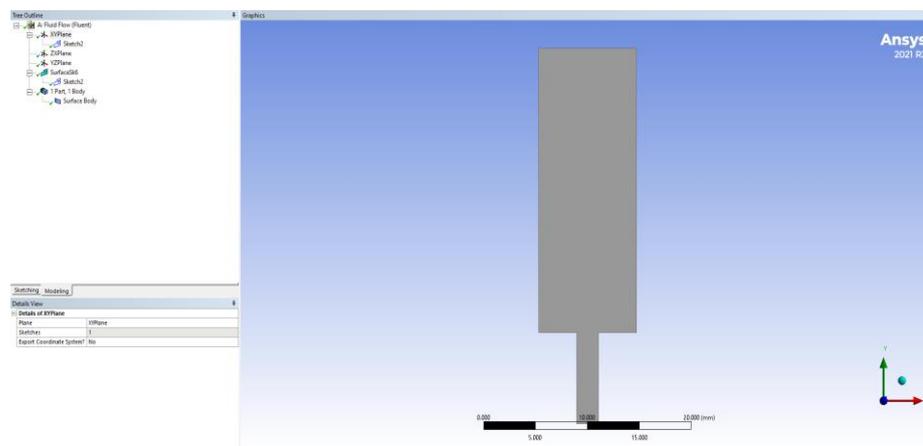**4.** Drawing surfaces were added to the sketch, figure (3.6).

Figure (0.6): Show capillary tube model drawing steps.

3.4.3 Meshing

Meshing step was very important to specify the nature of the micro-capillary.

Meshing steps can be identified as following:

1. Face sizing was used with size element of (500µm) for a uniform and dense meshing to the entire model as shown in figure (3.7).
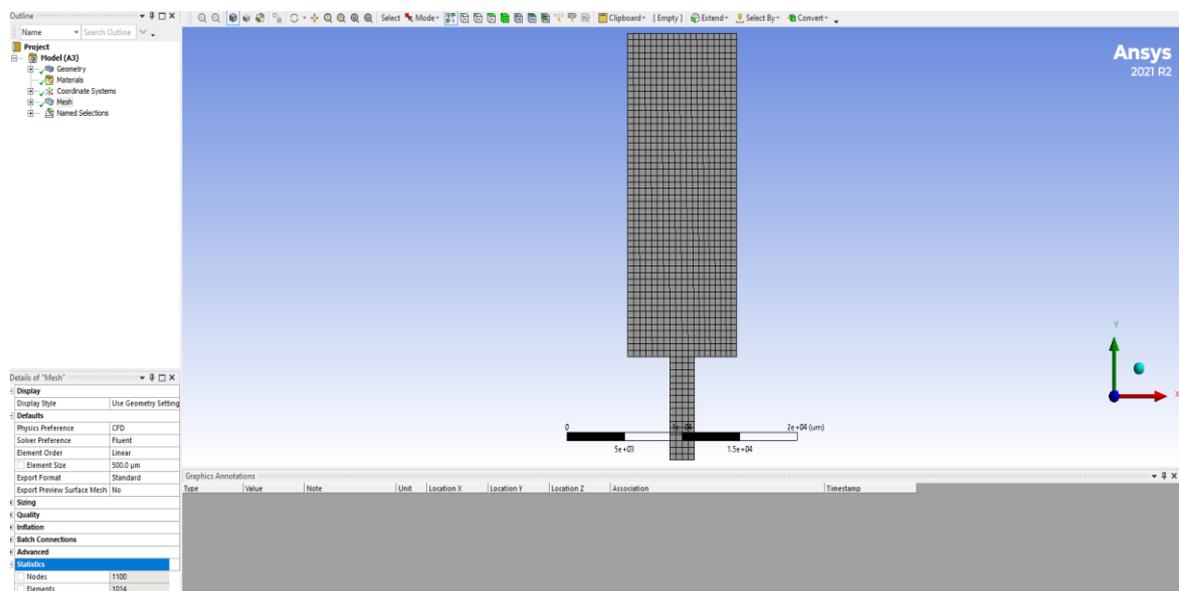

Figure (0.7): Meshing step.

2. Inflation was used to define the shear thought walls. All walls were selected and then unselect the inlet and outlet to delete inflation layers from them. Five inflation layers were used around the wall.

3. For each of the inlet and outlet, boundary criteria were defined using named selection.

4. After each step that is advanced, re-meshing is very important. The number of nodes was (1100), and the number of elements was (1014) as shown in figure (3.8).
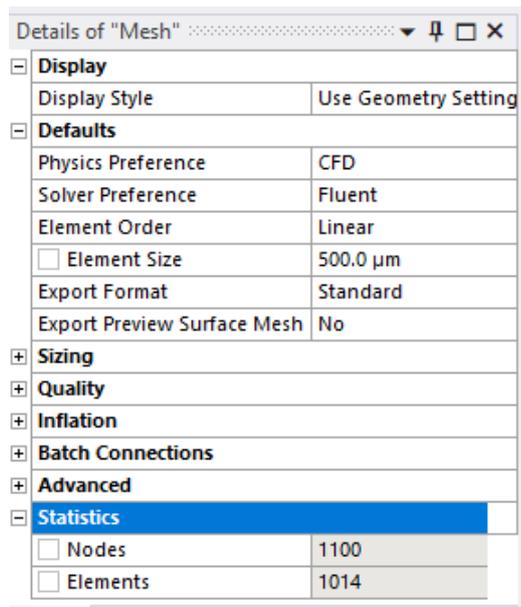
34

Figure (0.8): Re-meshing.

3.4.4 Setting up

To model the process of capillary tube flowing, several parameters were required. Each and every parameter used in the setup process is listed in (Table 3.3). To achieve the highest level of parallelism between computational and experimental work, these parameters were evaluated experimentally.

Table (3.3): List experimental properties of continuous and dispersed phases.

Material	Density g/cm^3	Hardness	Viscosity pa*s
cycle 1	0.8475	33.9	49900
cycle 2	0.8396	46	58450
cycle 3	0.9183	44.5	59975

After choosing number of processes a list of operation must take place orderly, as following:

1. Solver: The solver used was pressure based with transient time of 2D planar space, figure (3.9).
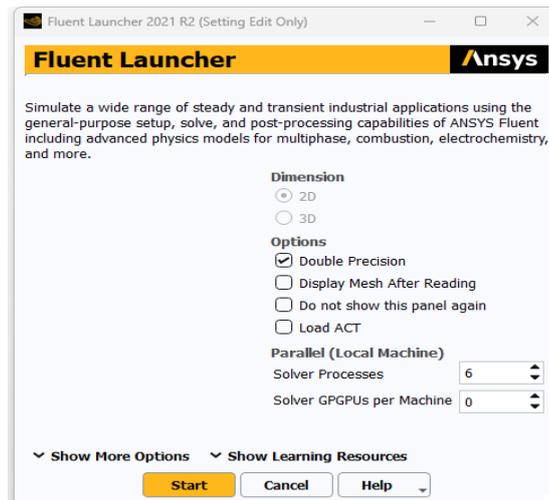
Figure (0.9): Show the solver.

2. Materials: This step will specify phase's materials. Density and viscosity were needed in this step, figure (3.10). The program was feed with the experimental data for each material in table (3.3).
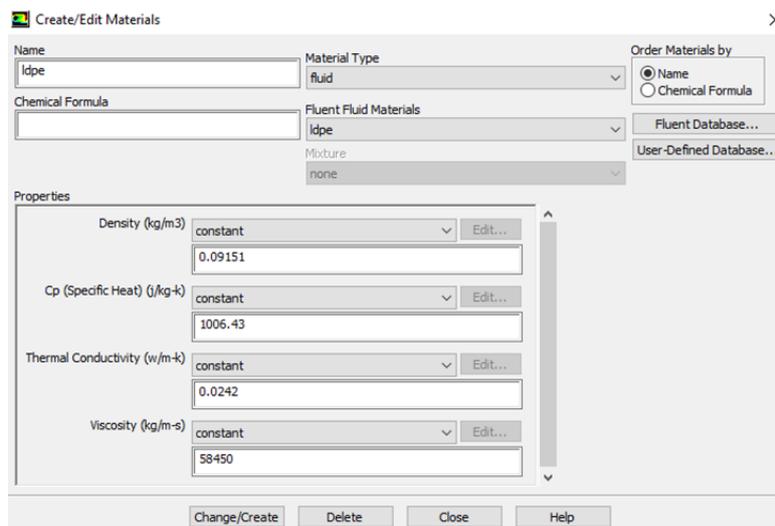

Figure (0.10): Specify phase's materials.

3. Boundary conditions: This stage involves the feeding velocity for each point. The pressure was within normal limits. For LDPE in a capillary tube, the interior-surface body.

4. Solution methods: The scheme was simple. The spatial discretization method employed was least squares cell based. The pressure was implicitly Bounded second order.

5. Solution initialization of the solution: Relative cell zone was employed with a standard initialization (3.11).
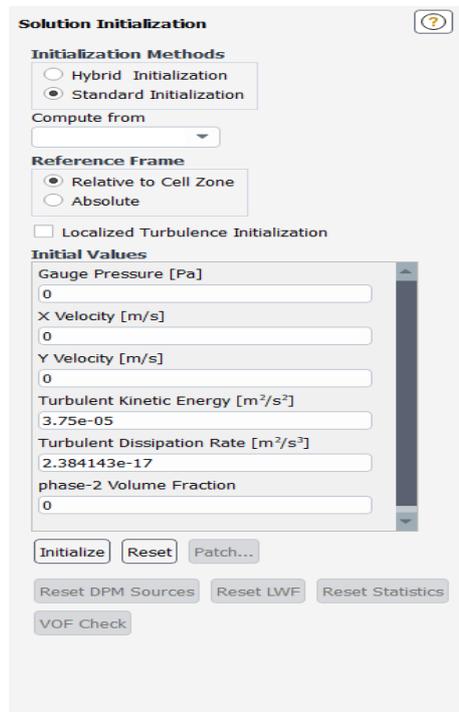

Figure (0.11): Initializing the solution.

6. Running calculation: There were 100 iterations. The reporting interval was 1, and the profile updating interval was 1. And then pressing calculates, figure (3.12).
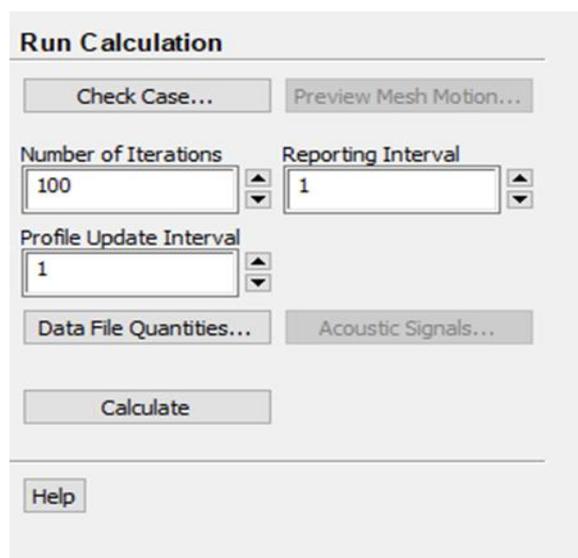

Figure (0.12): Show running computation.

4.1. Introduction

This chapter provides and discusses the results of experimental test; including the results that are obtained from rheological test, as well as the results that are obtained from the tests (density and hardness) and other tests. It includes the results of analysis by ANSYS program fluid flow (fluent) and comparing the results obtained with the experimental results. A comparison between experimental and numerical results is achieved to check the reliability of solution.

4.2. Rheological results

4.2.1. Shear viscosity

The ability of a polymer to resist flow is indicated by its melt viscosity. Increasing the free volume, decreasing the crosslinking density and poor intermolecular contact will reduce the flow resistance. These elements depend on molecular properties such as branching, molecular weight distribution, and molecular weight. Figure (4.1) shows how the shear viscosity behavior of several polymer cycles involves significant structural changes. The polymer used in this study is one of the majorities of polymeric materials on the viscosity curve that exhibit shear thinning behavior. Figure (4.1) show the shear rate increases with the shear viscosity of the polymer decreases. Viscosity decreases rapidly between (0 and 20) s^{-1} shear rates before gently gradient to $100s^{-1}$ shear rates. However, at shear rates up to $100s^{-1}$, the viscosity becomes more stable.
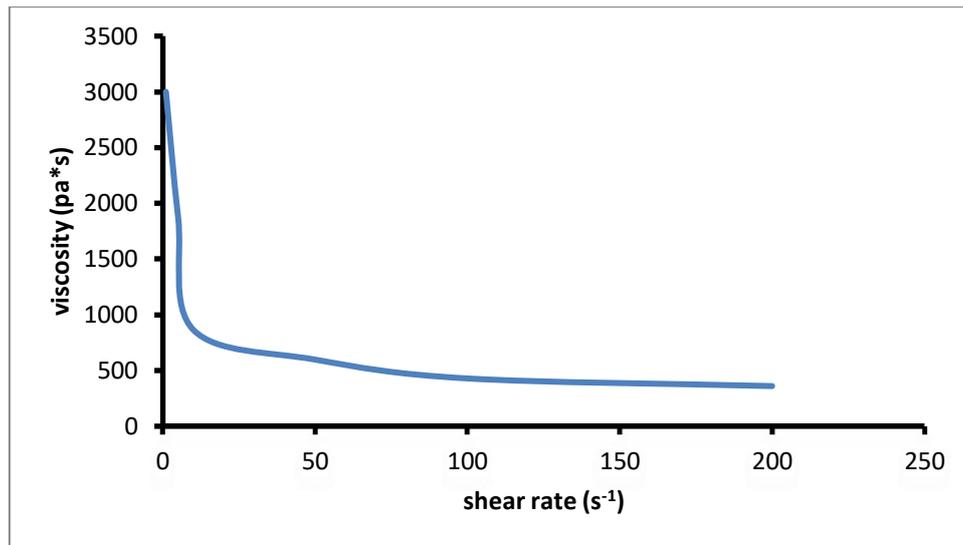
Figure (4.1): Show the LDPE viscosity behavior at cycle (1) with respect to shear rate.

The same kind of shear-thinning behavior is seen after the second cycle of LDPE, with a nearly equal reduction throughout the curve. When LDPE is recycled twice, as in figure (4.2), the resultant viscosity curve is close to the first recycle viscosity.
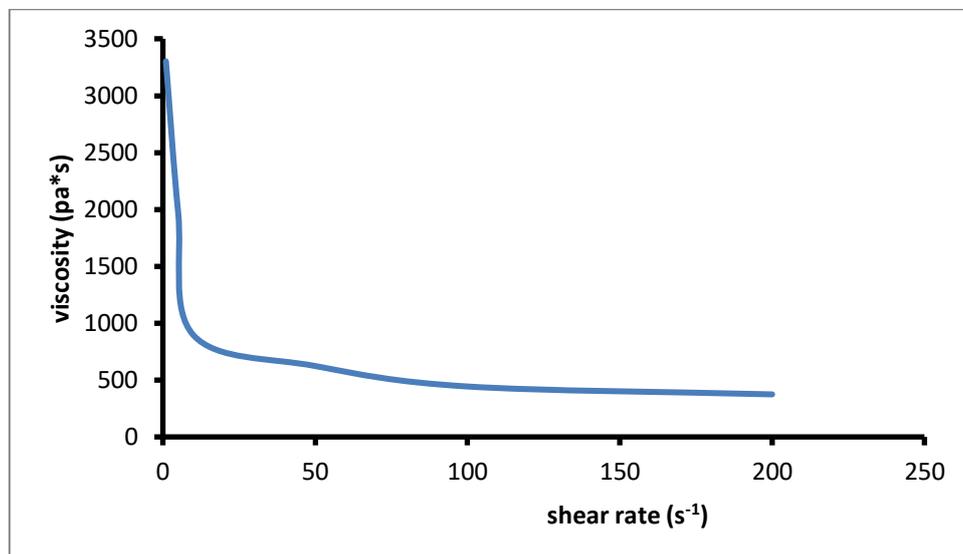

Figure (4.2): Show the LDPE viscosity behavior at cycle (2) with shear rate.

When the LDPE is recycled once again, the viscosity behavior also decreases with increasing shear rate, as shown in figure (4.3).
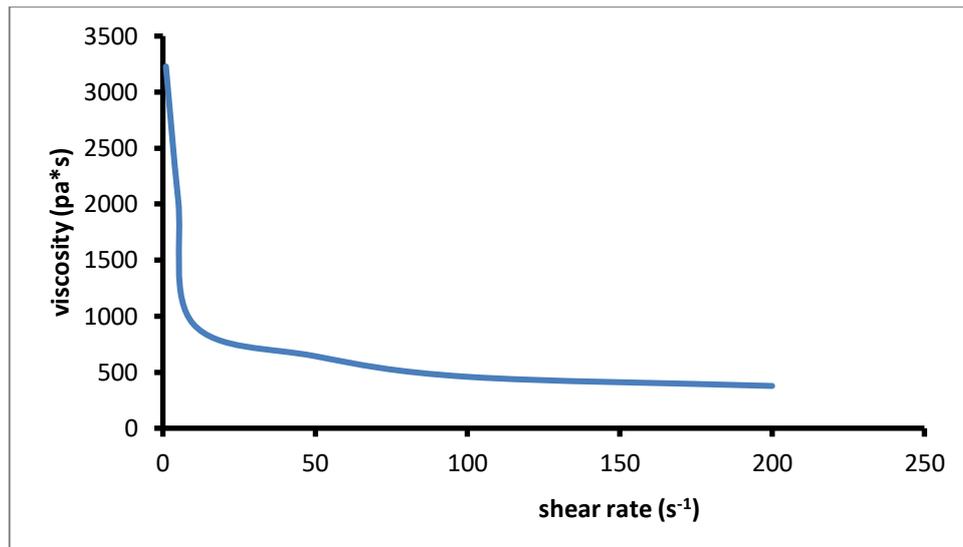
Figure (4.3): Show the LDPE viscosity behavior at cycle (3) with shear rate.

As stated in figure (4.4) illustrates viscosity very close for the same shear rate when LDPE is recycled three times. This behavior due to the Brownian movement. As a result, more heat is needed in the subsequent recycling cycle to break the long chains, increasing the viscosity of the polymer.
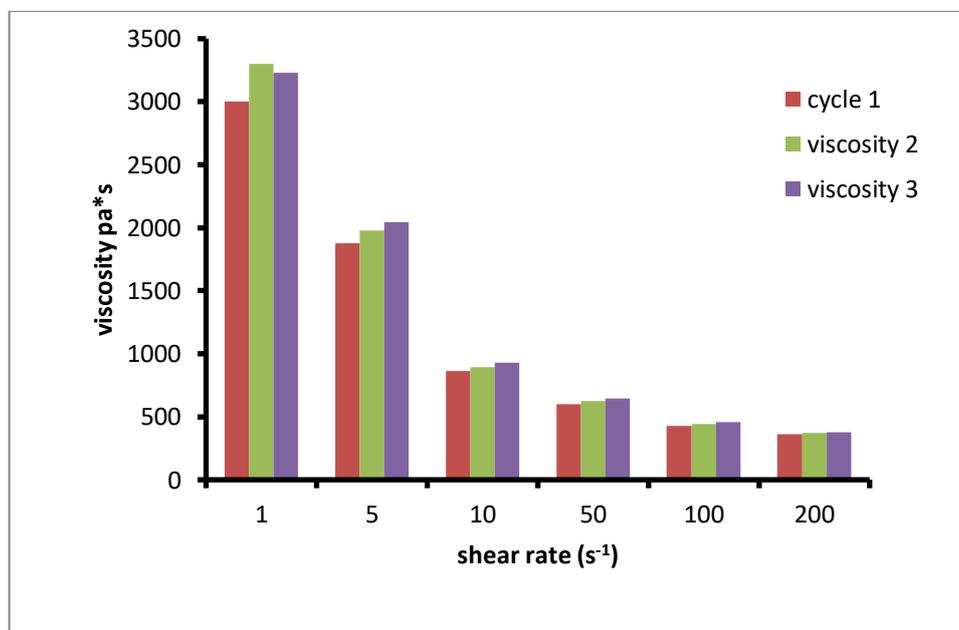

Figure (4.4): Show the LDPE curves viscosity with shear rate for three cycles.

Using the power low equation, the table (4.1) below lists the values for (k) and (n):

$$\mu=k\gamma^{\cdot n-1}$$

Table (Error! No text of specified style in document.**.1): Show values for (k), (n) using the power low equation.**

Cycle	k	n
1	2102.1	0.63
2	2006.9	0.64
3	1369.1	0.79

The values of (n) are fairly near, was less than 1 the recycling samples stayed in the non-Newtonian zone, and if more recycling occurs, the material will eventually become Newtonian. The near values of the (k) consistency index demonstrate that the material's viscosity characteristics are maintained when recycled in this manner. The values of (n and k) are consistent and complementing when compared.

4.2.2. Shear Stress

Figure (4.5) demonstrates how the shear stress steadily increases as the LDPE's shear rate increase. The shear stress of the polymer slightly increased after three recycling cycles of LDPE. Note that progressive increase in stress from (0 to 200) s^{-1} shear rates. Generally speaking, the shear stress increases linearly between (0 and 10) s^{-1} shear rates, then increases roughly exponentially between (0 and 200) s^{-1} shear rates.
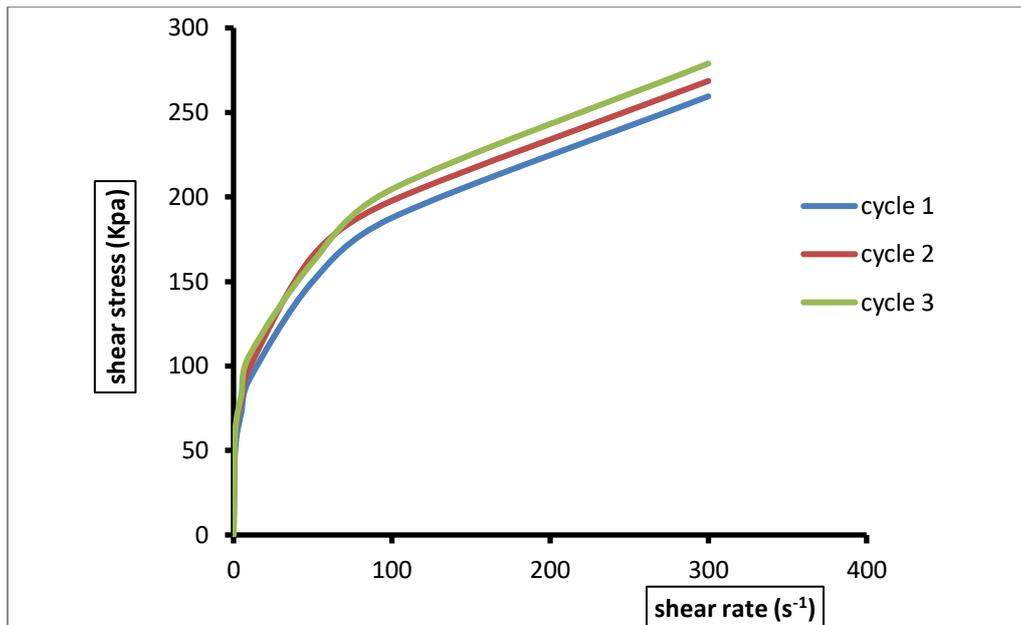
Figure (4.5): Shear stress behavior of LDPE cycles as a function of shear rate.

4.3. Density Test

Figure (4.6) shows the density test result. The melting at 190°C results in a modest drop in the density of the polymer. The findings of the density analysis of the LDPE samples are remarkably similar. The samples' density was slightly increase, which resulted decrease in the branches.

Figure (Error! No text of specified style in document..**6): Density test result for LDPE three cycles.**

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4.4. Hardness

The LDPE samples exhibit almost the convergent results in the hardness test, as illustrated in figure (4.7). Due to a decrease in viscosity, the hardness test reveals a slight increase from the first cycle to the third cycle. By lowering the density, one may easily increase the hardness of the samples by lowering the degree of crystallization.
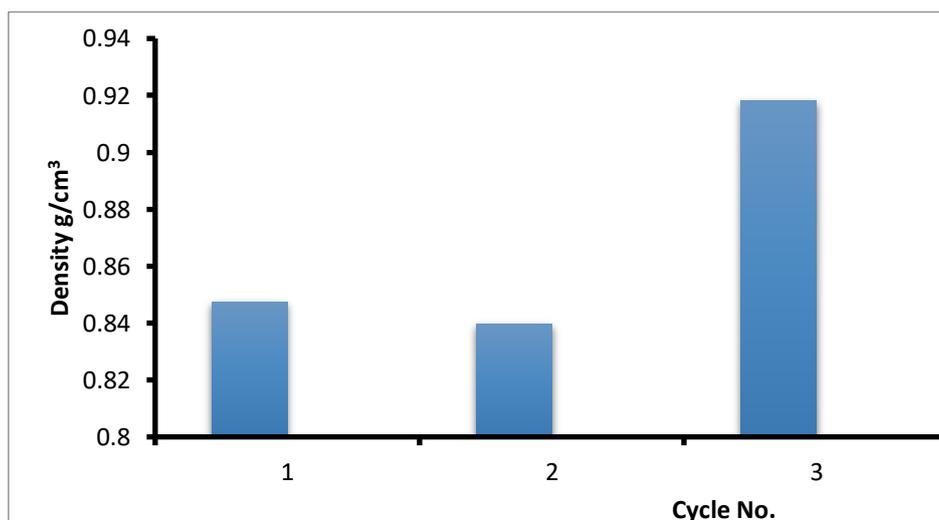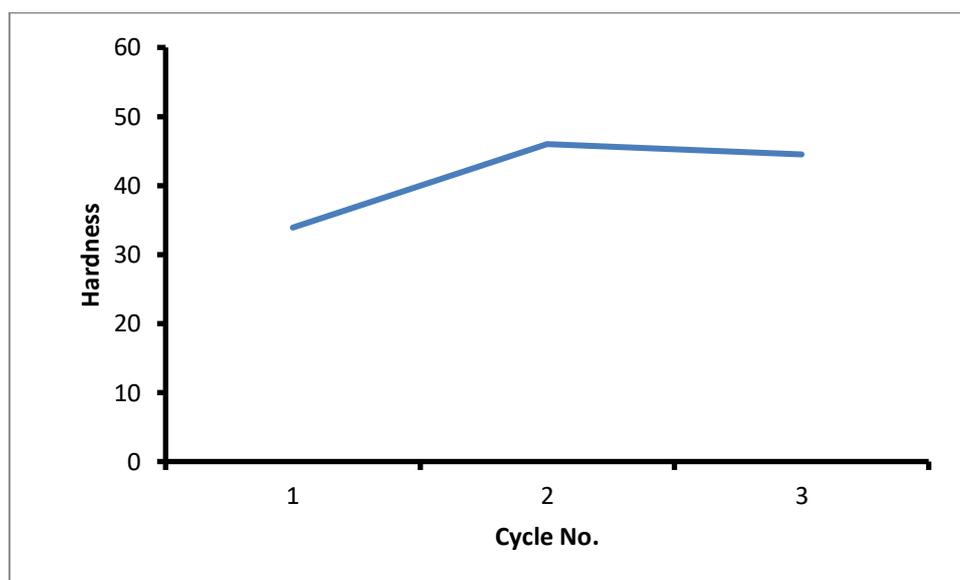

Figure (Error! No text of specified style in document.**.7): The hardness result for LDPE three cycles.**

4.5. Infrared spectroscopy IR

The infrared spectrum of LDPE irradiation shows the expected absorption bands for LDPE. The observed modifications in the spectrum are similar to those of pristine LDPE. The absorption bands that develop under irradiation are similar to those of the virgin LDPE which gives very close result for three cycles of LDPE in transparency range. It is known that the photo-oxidation of PE results in the formation of hydroxyl and carbonyl groups. The main oxidation nature of the products is well

established and the mechanisms by which oxidation occur can be considered to be fairly well understood.

Figure (4.8): Infrared spectroscopy IR result for LDPE three cycle (a, b and c) respectively.

4.6. Thermal results

4.6.1. Differential Scanning Calorimeter DSC

Using a differential scanning calorimeter, the thermal characteristics and crystallization behavior of LDPE were studied. Using the Netzsch 204-F1 apparatus, differential scanning calorimeter (DSC) study on
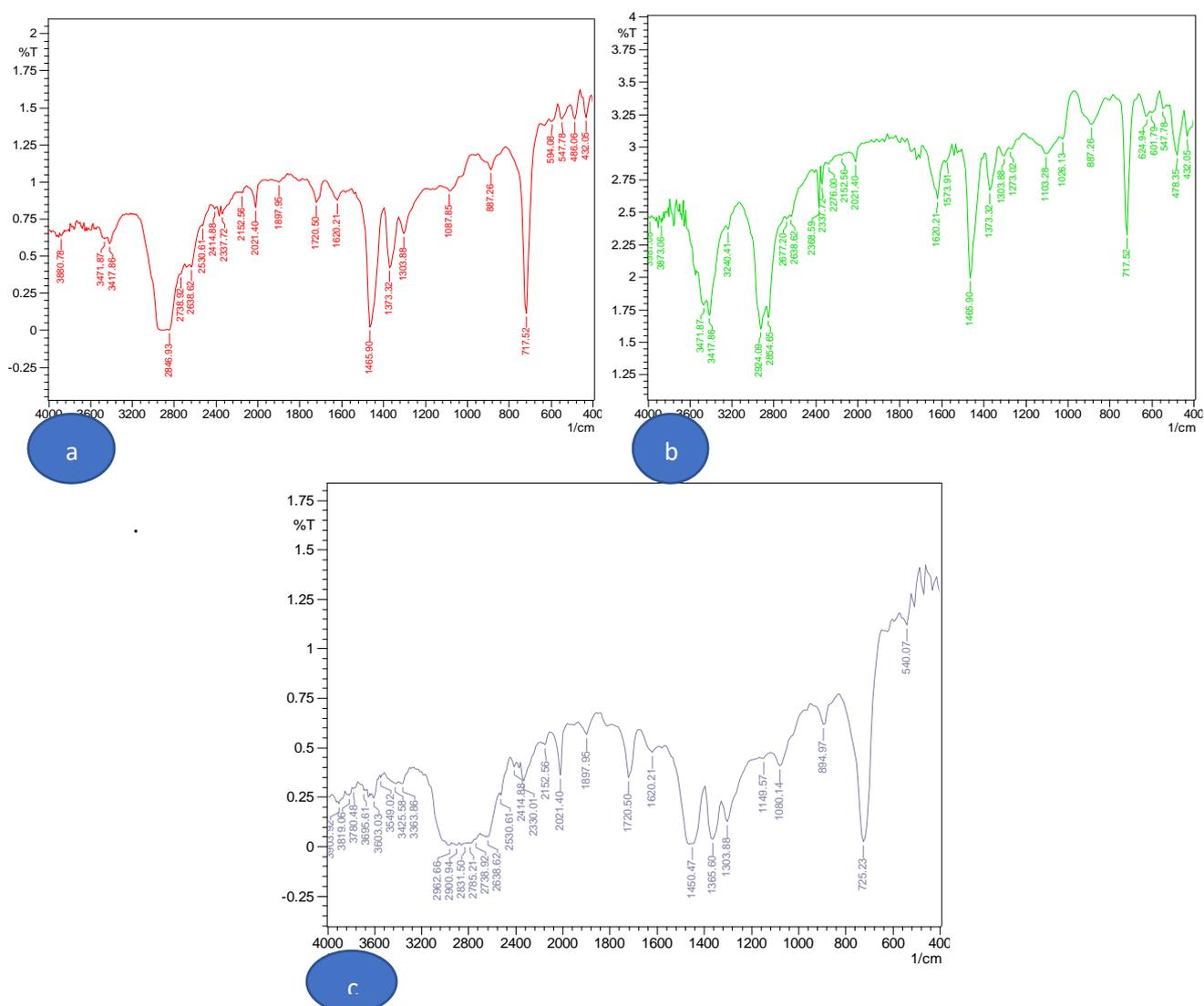
LDPE was carried out. According to the figure (4.9), the DSC analysis was used to find the melt and crystallization temperature of LDPE.
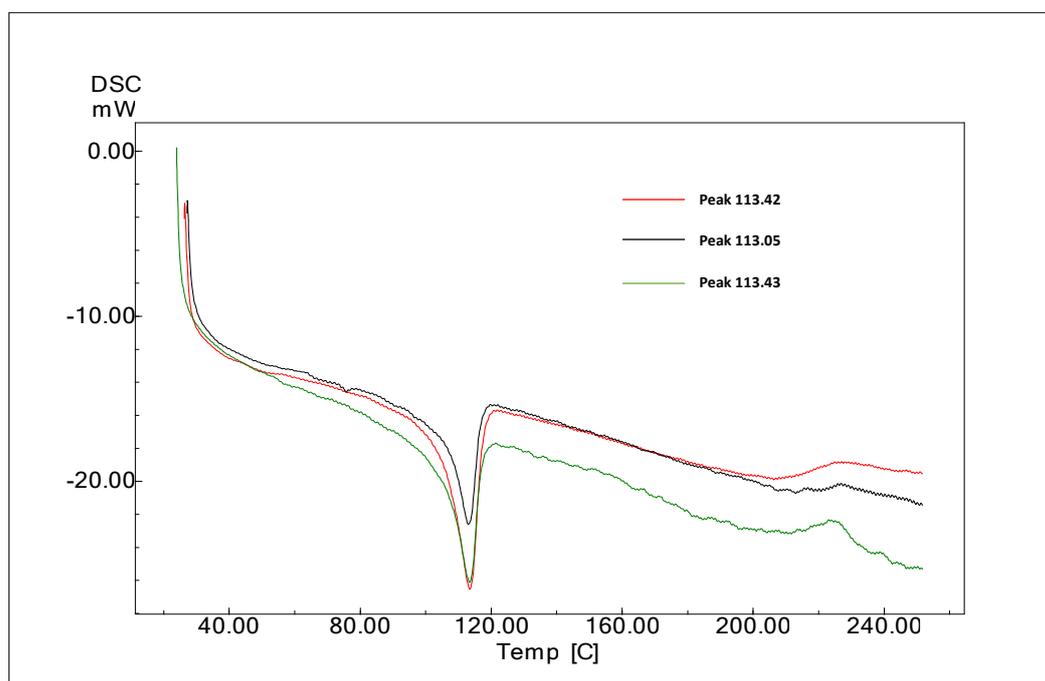
Figure (4.9): The DSC curves for LDPE.

The data clearly demonstrates a modest change in the material's temperature history during the capillary tube melting process. The melting point (T_m) of a material is the temperature at which it melts. The endothermic event's peak temperature is used to measure it. It is the highest temperature for materials, which also produced comparable outcomes. The results showed in the table (4.2).

Table (4.2): The T_m results for LDPE three cycles by using DSC.

Cycle No.	T_m
1	113.42°C
2	113.05°C

3	113.43°C

4.7. Numerical simulation results

It was discovered that the first sample's contour, stream line, and vector reveal that the material's greatest velocity and pressure are at the center, as well as for the second and third samples, after executing the simulation for the practical portion of the ansys program for the three samples of LDPE. The graphic illustrates how closely the outcomes were related figure (4.10). The validity of the practical portion is supported by numerical simulation for the three cycles of LDPE. The CFD-based Ansys software is based on the quantity, momentum, and Non-Newtonian viscosity models (power low).
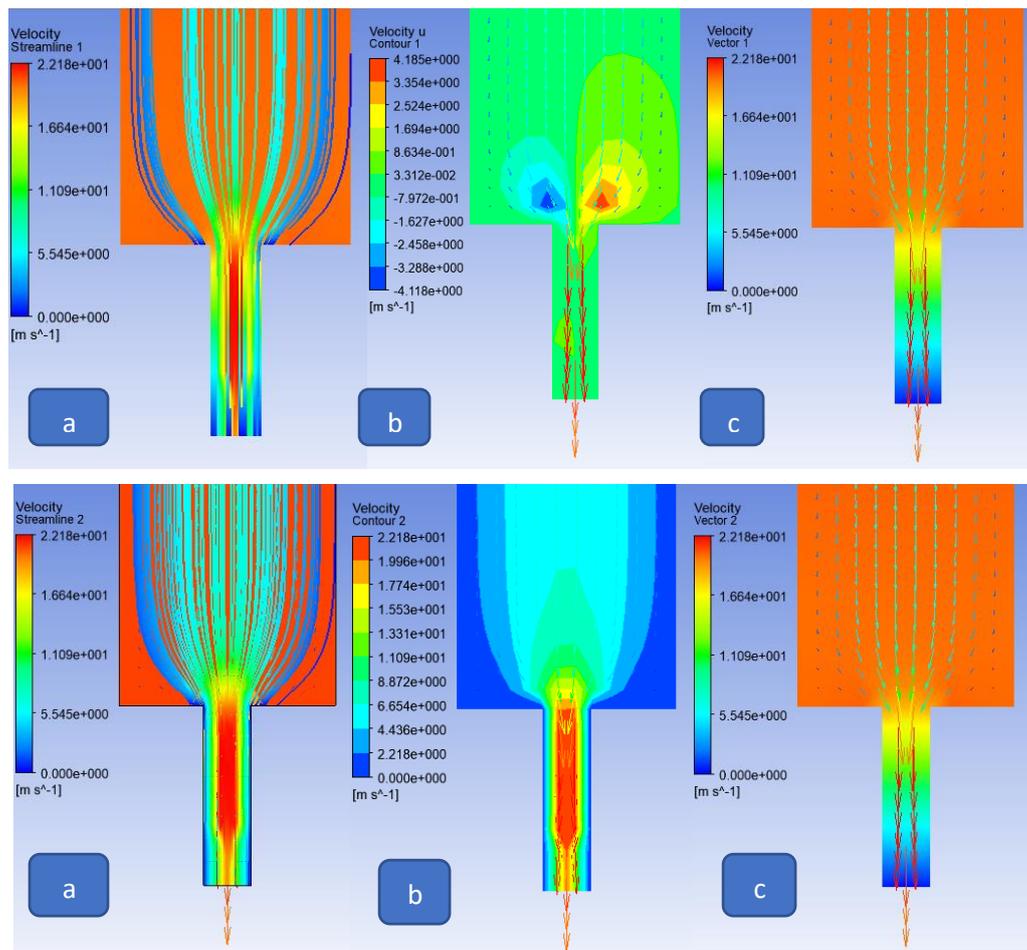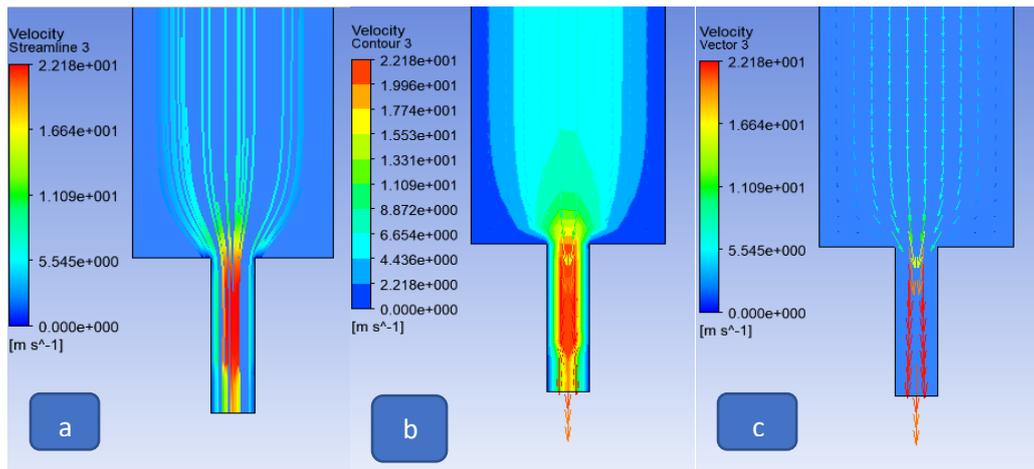
Figure (Error! No text of specified style in document.**.10): Shows numerical simulation by using ansys, (a) The vector picture for three cycles, (b) The contour image for three cycles, and (c) The stream line image for three cycles.**

5. Conclusion and Recommendation

5.1. Conclusions:

From the present work, the following can be concluded:

1. Shear thinning behavior and non-Newtonian flow dominate from viscosity curve of LDPE for three recycling.

2. Shear stress increasing with the shear rate increases.

3. Density, colors, T_m where changed in the range of the compatible with the rheological properties according to the same vision.

4. Numerical simulations for three cycles of LDPE indicate the same velocity, pressure, quality results. Where the same pressure and velocity distribution produced same structure and related rheological and physical properties.

5.2. Recommendation:

1. Using extrusion to obtain recycled polymer samples.

2. Using another type of polymer instead of polyethylene.

3. Using more than three cycles for more accuracy.

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Chapter One

Introduction

Chapter Two

Theoretical Part and Literature Review

Chapter Three

Experimental Part & Numerical Simulation

Chapter Four

Results & Discussion

Chapter Five

Conclusion and Recommendation

References

جمهورية العراق
وزارة التعليم العالي والبحث العلمي
جامعة بابل
كلية هندسة المواد
قسم هندسة البوليمرات والصناعات
والبتروكيماويات

تأثير إعادة التدوير على الخواص الريولوجية للبولي اثيلين واطئ الكثافة

رسالة مقدمة الى

عمادة كلية هندسة المواد / جامعة بابل

وهي جزء من متطلبات نيل درجة الدبلوم العالي/ هندسة المواد – هندسة البوليمر

من قبل:-

صبا حامد شاكر كريم

(بكالوريوس هندسة مواد ومواد مركبة/بوليمر .2016)

بأشراف

أ. د. نزار جواد هادي

2023م   1444هـ

الخلاصة

يركز هذا العمل على إعادة تدوير البولي إثيلين المنخفض الكثافة(LDPE) لتقليل الضرر الذي يلحقه البلاستيك بالبيئة. تم بثق 30 غم من LDPE لثلاث دورات عند 190 س° ومعدل القص (1000-1) ثا $^{-1}$ للحصول على ثلاث عينات باستخدام capillary tube. تم إجراء الاختبارات الريولوجية والفيزيائية لثلاث عينات من (LDPE). تم تحليل العلاقة بين اللزوجة وإجهاد القص مع معدل القص. كما تم اختبار مسعر المسح التفاضلي(DSC) والكثافة والصلادة لجميع العينات.

أظهرت النتائج أن اللزوجة تنخفض ويزداد إجهاد القص مع معدل القص لكل عينة. بشكل عام ، يتكون منحنى اللزوجة من مرحلتين. في المرحلة الأولى مع النطاق (300-1) ثا$^{-1}$ ، تنخفض اللزوجة بسرعة ، بينما تنخفض اللزوجة في المرحلة الثانية تدريجياً ويحاول منصهر ال LDPE أن يكون Newtonian.

يجدر الإشارة إلى أن الكثافة والصلادة تتغير قليلاً مع زيادة رقم الدورة. كذلك لون وشفافية ثلاث عينات متماثلة تقريبًا. تظهر نتائج اختبار DSC أن درجة حرارة الانصهار لثلاث عينات هي 113.42 درجة مئوية و 113.05 درجة مئوية و 113.43 درجة مئوية على التوالي. تم الحصول على المحاكاة العددية لنفس عدادات السرعة والضغط للعينات الثلاث. تم الحصول على اتفاق جيد.