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Faculty of Materials Engineering
Polymer Department

Study the Irregular Crumbling of Recycled PET on the Mechanical and Energy by using Extrusion Process

A Thesis
Submitted to the Council of the College of the Materials Engineering / University of Babylon in Partial Fulfillment of the Requirements for the Master Degree in Materials Engineering / Polymer

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بسم الله الرحمن الرحيم

﴿قَالُوا سُبْحَانَكَ لَا عِلْمَ لَنَا إِلَّا مَا
عَلَّمْتَنَا إِنَّكَ أَنْتَ الْعَلِيمُ الْحَكِيمُ﴾

صدق الله العلي العظيم

سورة البقرة / الأية 32

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Firstly, all the thanks and praise be to **ALLAH** for enabling me to achieve this study.

*** *** ***

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*** *** ***

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Dedication

To Prof. Dr. Ali A.A. Al-Zubiedy**,**

To my Dad and Mom;

The reason for what I have become today. Thank you for your great support during my academic career.

To my wife

Rafal Adnan

To my Son;

Ali Hussain

***To my* colleague;**

Mustafa Helal thank you for everything.

With Respect

Hussain Ali

The Supervisor Certification

I certify that this thesis entitled (**Study the Irregular Crumbling of Recycled PET on The Mechanical and Energy by using Extrusion Process**) was prepared by (**Hussain Ali Adnan Umran**) under my supervision at Babylon University / Faculty of Materials Engineering/ Department of Polymer and Petrochemical Industries, in Partial Fulfillment of the Requirements for the Award Master Degree in Materials Engineering / Polymer.

Supervisor Signature:

Name: Prof. Ali A.A. Al-Zubiedy

Date: / / 2021

Abstract:

This work deals with studying the effect of the irregular crumbling for recycled polyethylene terephthalate (PET) bottles on the extrusion process using a twin-screw extruder and as a result of its effect on the mechanical properties of the product and the amount of electrical energy consumption.

The extrusion process was performed for different pieces of recycled polyethylene terephthalate (PET) bottles (4.75, 6.7, 7.15, and 10 mm), at temperature ranges between ($200\text{-}205^{0}$ C) at speed of 50 rpm. The results showed that the cutting length has a direct effect on the crystallinity which affects mechanical and thermal properties, such as elongation and tensile strength, where tensile strength and elongation decrease with increasing of the cutting length but the elastic modulus increases with the increase of cutting length.

The results of impact and hardness tests that the impact strength decrease by (33.156%) with the increase of the cutting length also hardness decrease by (23.88%) with increasing of the cutting length. It was observed during the extrusion process that the small pieces consumed less electrical energy by (52.5%), compared to the big pieces of recycled (PET) bottles, except the raw material from (PET). The results of digital microscope inspection that the amount of bubbles, as well as the bad surface finish, increases with increasing of the cutting length. The results of differential scanning calorimetry (DSC) tests that the melting temperature, degree of crystallinity, and glass transition temperature decrease with increasing of the cutting length. The results of melt flow rate (MFR) test that the melt flow rate increase by (19.696%) with increasing of the cutting length. The results of density tests that the density decreases with increasing the cutting length.

Contents

Chapter Three: Experimental Part

Abbreviations

Character	Item	Units
APET	Amorphous polyethylene terephthalate	_
ASTM	American Society for Testing Materials	_
CPET	Semi-crystalline polyethylene terephthalate	_
DSC	Differential Scanning Calorimetry	_
FTIR	Infrared Fourier Transform Spectrometer	_
HDPE	High-density polyethylene	_
I.S	Impact strength of the material	kJ/m^2
KBR	Potassium bromide	_
LDPE	Low-density polyethylene	_
MFR OR MFI	Melt flow rate or melt flow index	g/10min
MW	Molecular weight	g/mol
PET	Polyethylene terephthalate	_
PP	Polypropylene	_
PE	Polyethylene	_
PEEK	Polyether ether ketone	_
ISO	International Organization for Standardization	_
NaOH	Sodium hydroxide	_

RWS	Recycled Woven Plastic Sack Waste	_
DEM	Discrete Element Modeling	_
CF	Carbon fiber	_
RWS	Recycled woven plastic sack waste	_
RAC	Recycled aggregate concrete	_
RPET	Recycled polyethylene terephthalate	_
rpm	Revolutions per minute	_
TP	Total extruder power	_
MT	Mass throughput	_
SEC	Specific energy consumption	_

Symbols

A	cross-sectional area of sample	m^2
D	Diameter	mm
L_f	Final length	mm
L_o	Initial length	mm
S	Step of the screw	_
Tm	Melting temperature	°C
Tg	Glass transition temperature	°C
T	Temperature	°C
Tc	Crystallization temperature	°C
U_C	Impact energy	Joule
W	The average weight of cutting time of the sample	g
γ	Shear rate	s^{-1}
ρ	Density	g/cm^3
E	Elastic modulus	MPa

Ɛ	Strain	Dimensionless
σ	Tensile strength	MPa
ΔL	Length change	mm
T	Transmittance	_
A	Absorbance	_
F	Tension force	_
µm	Micrometre	_
S-P	Small pieces	_
Me-P	Medium pieces	_
Mi-P	Mixed pieces	_
B-P	Big pieces	_
R-M	Raw material	_
A	Ampere	_

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Chapter One

Introduction

1.1. Introduction:

Recycling is the process of reusing industrial, agricultural, medical and household wastes to decrease the influence of these wastes on the environment [1]. This process is done by classifying and sorting these wastes based on the raw materials and then recovering each material alone [2-4]. Whereas, the recycling process of the material has a fundamental role in restoring the original form of the material that was in the beginning [5].

The recycling process is considered a very necessary process to get rid of waste, save materials, and help reduce global warming gas emissions. The topic of recycling is not new, glass was recycled and metal materials such as aluminum and some metal materials were recycled to benefit from them once, twice, and several times before burning this waste, which leads to air pollution. The interest of developed countries in the recycling process is to support the national economy and protect the environment and the most important materials that are recycled: paper of all kinds, thermoplastic polymers, rubber, glass, and metal materials [6].

It is also known that the process of recycling prevents waste of materials, reduces the consumption of raw materials, thus energy consumption will decrease, reduce air pollution resulting from burning waste, and reduce water pollution. Polymer recycling is a concept and a modern technology not only from an economic or research perspective but also from an environmental and economic perspective. [7-9].

Polymer (plastic) demand has grown worldwide, as it affects almost every part of the economy. The use of polymers is of greater benefit to society with its thriving applications, which include making processed food last longer, eliminating waste, and encouraging the use of polymer in pipes to provide safe drinking water, whereas polymer helps medical devices life-saving such as surgical equipment and others [10].

Polyethylene terephthalate (PET) is a thermoplastic material made from a polyester of ethylene glycol and terephthalic acid, owing to its inert chemical and physical properties; it is applied in many areas. In some developing countries, polymer recycling is at a minimum rate, resulting in increased sales of polymers at higher prices, manual labor was used to shred waste for local recycling of polyethylene terephthalate waste, which restricts the amount of shredding of PET waste since the cost and maintenance of shredding machines is very costly and therefore, a need for a suitable and efficient shredder machine. The polymer is one of the world's most useful and essential materials. A global rise in polymer production has been observed over the last 50 years and is expected to double in the next 20 years [11] as shown in Figure(1.1).

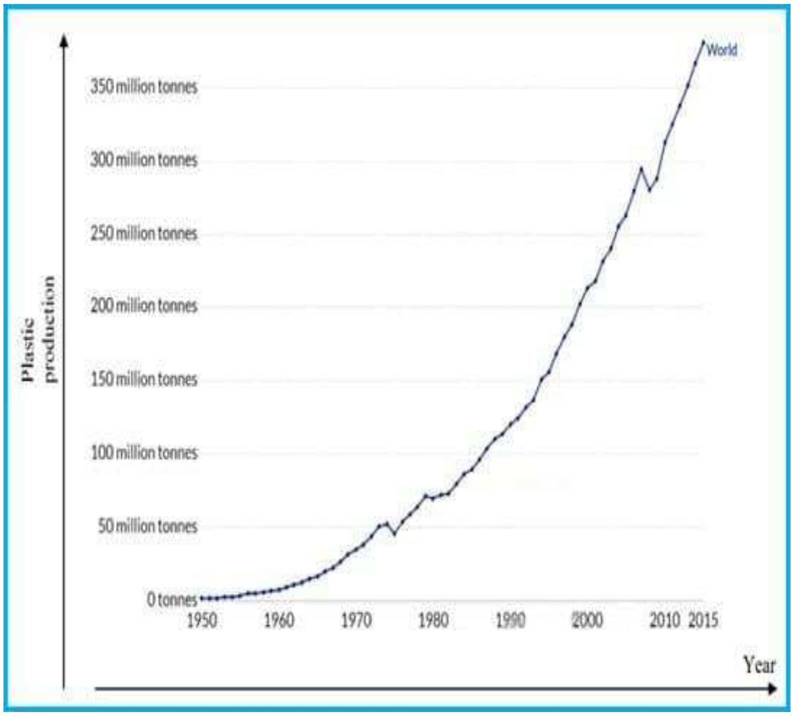


Figure (1.1): The polymer production rate from 1950 – 2015 [12].

The European Union (EU) is concerned with protecting the environment and its cleanliness from these wastes because the disposal or decomposition of these polymeric wastes takes a lot of time. The developed world has succeeded in recycling materials and benefiting from them, so the interest of these countries has become the recycling process because of its great importance in disposing of waste and reducing the waste of materials, as the consumption of polymers has become common everywhere from various aspects of life, for the importance of these materials and their properties, that is, they have physical properties that give these materials a property that is used instead of metal, wood, and other materials, due to their importance [13].

Due to its widespread use, has emerged a problem in the recycling and final disposal of these materials [14]. The developed world has taken upon itself an interest in the issue of recycling [15].

After scientific experiments and research, it appeared that polymeric materials have properties that enable them to be reused multiple times without losing their characteristics. As the material can be recycled and used for the same purpose, that it was in the beginning, or it is possible to produce another substance and use it other than the primary uses that were previously.

They thought through scientific research how to get rid of these wastes and benefit from them at the same time, so the research happened in the previous and they found that the polymeric material can be reused six times without losing its characteristics, then the research took place and they found it could be reused ten times without losing its characteristics [16, 17] Figure (1.2). Showing the growth in the recycling of (PP, PE).

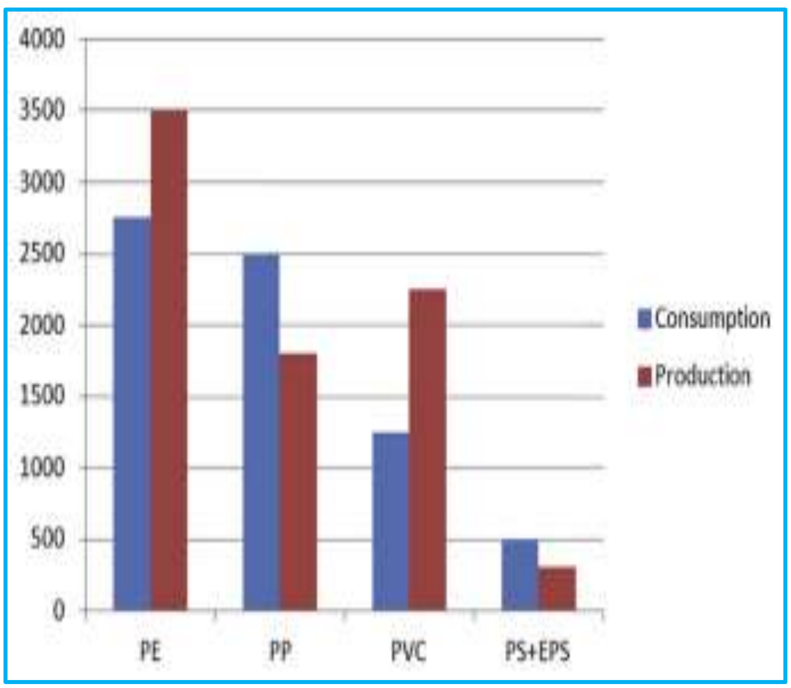


Figure (1.2): The production and consumption of different polymer materials [18].

The crumbling machine reduces from polyethylene terephthalate (PET) bottles size to smaller sizes to increase their portability and ease preparation for use in a new product. The crumbling process is very necessary and one of the most important stages of the recycling process because the consumption of electrical energy depends on the crumbling process for thermoplastic polymers recycled, and this process is expensive with many problems occurring the crumbling process. It requires less labor work and there is no requirement for skilled labor in industry. In recycling, the process of plastic waste required low energy due to reducing the costs of recycling polymeric waste. In recycled plastic, the waste undergoes five different stages: sorting of plastic waste, washing of plastics to remove impurities, shredding of washed plastics, and extruding by melting the shredded pellets into sizes used for different plastic products. Out of these five stages, it is only the shredding and extruding stages that must involve a form of machinery. To this end, the aim of this study is the effect of the irregular crumbling for recycled polyethylene terephthalate (PET) bottles on the extrusion process and mechanical properties of the product[19,20].

1. 2. Scope and Objectives:

This work aims to study the effect of the irregular crumbling for recycled polyethylene terephthalate (PET) bottles on the extrusion process using a twin-screw extruder and as a result of its effect on the mechanical properties of the product and the amount of electrical energy consumption without the granulation process during the recycling to reduce workload, reduce electrical energy consumption on the machine this, in turn, will reduce from recycling costs.

Chapter Two

Theoretical Part
&
Literatures Review

2. 1. Introduction:

The present chapter consists of two main sections. The first section focuses on the recycling of polymeric wastes such as polyethylene terephthalate (PET), especially the process of stabilizing the shredding of plastic products made of (PET), and the effect of this process on the processing of the material by using the extrusion process, especially the calculating the consumption of electrical energy during the recycling process.

The second section focuses on the literature review on the shredding stability process for plastic wastes.

2. 2. Polymer Recycling:

Polymer recycling is the method of recovering different kinds of polymer material in arrange to reprocess them into diverse items or the same type. Recycling polymer is one way to reduce environmental effects, resource depletion, and energy consumption. The only method of reducing the environmental pollution caused by polymeric waste accumulation produced such as those used in construction and packaging is by recycling. This helps in the preservation of natural resources because most polymer substances are made from gas and oil [21].

2. 3. Stages of Recycling of Polymeric Materials:

When recycling polymer waste, polymer waste must pass through many stages to be reused again for the same purpose that it was at the beginning or for another use:

2. 3. 1. The stage of **collecting** polymeric waste in different ways, direct and indirect (from the consumer or from the producer). The polymer wastes can be collected for recycling from the roadside and the consumer by putting plastic waste bins in different places for easy collection later. The growth in the collection of polymer bottles from 1999-2008 is shown in Figure (2.1) [22, 23].

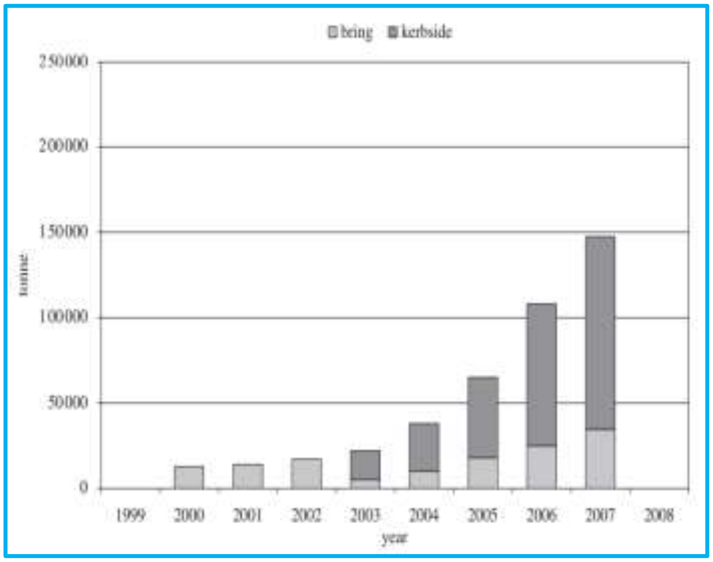


Figure (2.1): The growth in the collection of polymer (plastic) bottles from 1999-2008 [23].

2. 3. 2. Sorting Stage: One of the important stages in the recycling of polymeric materials. This stage requires good waste sorting because polymers lose some of their properties if contaminations are present with them. The sorting process is carried out in several ways, including manual or mechanical...etc. In mechanical sorting, the sorting is carried out by automatic separation through using the magnet to attract metals, while manual sorting, includes the use of hands to separate the wood, metals, etc., or separate the plastics waste into different polymers from mixed waste for recycling [24,25].

2. 3. 3. Washing Stage: Polymers wastes are washed by entering them in large basins that contain hot water and caustic soda or detergents are added to them. The recycling of polymers requires that they be free from dirty such as dust, oil, grease, paper, and foreign bodies... etc. The washing of the polymer wastes can be done either mechanically or manually using the washing tank, where the unclean water can be drained out easily [25].

2. 3. 4. Drying Stage: After washing the polymer waste, it is dried to minimize the moisture and remove water, where it is transferred to the drying basins by exposing it to direct sunlight or using the mechanical method of drying such as the oven as shown in Figure (2.2). The polymer flakes are left in the oven at the temperature of 50^0 C for five to six hours [25, 26].

Figure (2.2): The oven used for purpose drying [26].

2. 3. 5. Shredding Stage: In this stage, the polymer waste is broken or cut in shredding machines as shown in Figure (2.3) into smaller parts or volumes according to the type of polymeric material to be recycled, and the size of crushing or cutting is controlled according to the required specifications. In the case of large plastic bottles, it is necessary to cut the bottles into small dimensions before feeding them into the shredder [26, 27].

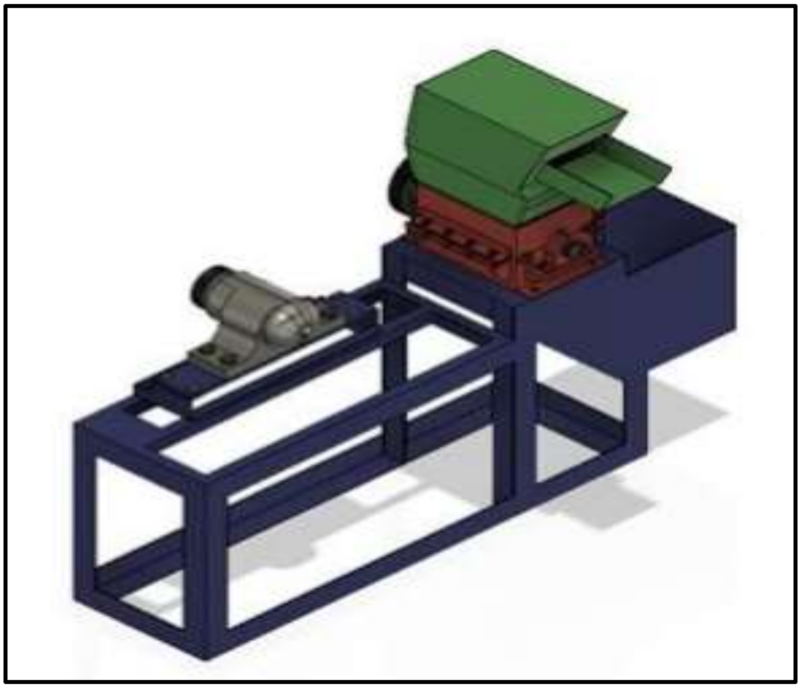


Figure (2.3): Illustrates the polymer crumbling machine [27].

2. 3. 6. Granulation Stage: then these cut materials are transported (in the form of pieces or powder) to the granulation machine. Where it is granulated or manufactured in the form of polymeric granules into a raw material that can be used to make new polymeric materials. Each type of cut polymer must be separated from the other to manufacture new products from only one type of polymer [25].

2. 3. 7. Formation Stage: In this stage, polymers are formed into products that can be used again. This stage is done in different ways according to the quality of the product, among these methods are extrusion, injection, blow, injection blow molding process, and others [27].

2. 4. Recycling Methods for Polymer Waste:

2.4.1. Primary Recycling:

In this type of recycling, the conversion of waste plastic into products has a performance level comparable to that of original products made from virgin plastics. The recycled scrap or waste is either mixed with virgin material to assure product quality or used as second-grade material. Primary recycling is very simple without any precautions except the proper and clean collection of waste in the plant [28].

2.4.2. Mechanical Recycling (or secondary recycling):

Is the reprocessing of materials of waste plastics by physical means, like cutting, shredding, washing, and so on, into plastic products. In this approach, the polymer is separated from its associated contaminants, and it can be readily reprocessed into granules by conventional melt filtration extrusion. The size of the waste plastic is reduced after it is sorted, cleaned, and dried, and then directly processed into end products or flakes of consistent quality, which can be further used for manufacturing other goods. The succeeding steps for recycling can vary from operation to operation and the end-use. In mechanical recycling, only thermoplastics can be used because they can be re-melted and reprocessed into end products [29].

The basic polymer is not altered during the process. Among the main issues of secondary recycling are the heterogeneity of the solid waste and the deterioration of the product’s properties in each cycle. This occurs because the molecular weight of the recycled resin is reduced because of chain-scission reactions caused by the presence of water and trace acidic impurities. Strategies for maintaining the polymer’s average molecular weight during reprocessing include intensive drying, reprocessing with vacuum degassing, the use of chain extender compounds, and so on [30].

2.4.3. Energy Recovery (Quaternary Recycling)

Energy recovery (quaternary recycling) refers to the recovery of plastics' energy content. This is an effective way to reduce the volume of organic materials. Incineration aiming at the recovery of energy is currently the most effective way to reduce the volume of organic materials. Though this method yields considerable energy from polymers, it is ecologically unacceptable owing to the health risk from airborne toxic substances, for example, dioxins (in the case of chlorine-containing polymers) [30].

2. 4. 4. Chemical Recycling: One of the most important processes in the recycling of polymeric wastes because polymer (plastics) are converted to their monomers, which can then be recycled, as the polymers are broken down by many methods such as hydrogenation, thermal treatment, the presence of a suitable catalyst such as benzene, styrene, propylene, ethylene, and vinyl to obtain the monomer and chemical depolymerization, and thus the monomer is chemically polymerized to produce different classes of polymers [31,32].

2. 5. Benefits of Recycling Polymer Waste:

There are many advantages of the recycling process that can be recognized after understanding the recycling processes and stages of polymeric waste:

2. 5. 1. Social Benefits: There is a lot of huge polymer waste that the percentage of this accumulated waste reaches about (60-70%). It is necessary to recycle it to reduce huge amounts of polymer waste and reuse it again and benefit from it.

This recycling process not only contributes to increasing the production of polymer materials but also contributes to protecting the environment from this accumulated waste and prevents diseases related to plastic waste [33].

2. 5. 2. Economic Benefits: Through the polymer waste recycling process reduce dependence on raw materials that require complex preparation methods and more energy consumption. Which has an important role in preserving natural resources and energy. Raw materials are obtained from this recycled polymer waste to produce a virgin polymer.

The purpose of preserving natural resources, water, and petroleum is to achieve a balance in nature [34, 35].

2. 5. 3. Environmental Benefits: In the absence of the recycling process. An accumulation of polymer waste occurs. Which must be reused again and utilized by the recycling process for these polymer wastes because the recycling process of polymer waste helps to get rid of the accumulation of these wastes and thus the environment is preserved from this polymer waste and we avoid burning waste that causes many problems. Studies and research have proven that when throwing waste of another type such as polymer waste on the same ground, decomposition of these wastes will occur after a period of throwing the waste and as a result of this decomposition toxic fumes are emitted from this decomposing waste and this fume is harmful to the environment and causes pollution in the area in which these wastes have been decomposed and this type of pollution causes different types of diseases such as lung and skin diseases [36-38].

The recycling process, therefore, plays a very important role in protecting the ecosystem from these environmentally damaging wastes. To keep them clean of this waste. Thus, this approach is not limited to the correct use of polymer waste. Instead, it leads to the safety and preservation of the environment [39].

2. 6. Materials Used in this Study:

2. 6. 1. PET Material:

It is one of the thermoplastic polymers made from esterification reaction between terephthalic acid and ethylene glycol [40]. The polyethylene terephthalate material is two types according to the arrangement of the chains during the solidification process, when rapid cooling the arrangement of the chains is random and not regularly arranged so it is called amorphous (APET) and when cooling slowly there is sufficient time to arrange the chains in a regular and in the form of a crystal structure which it is called semi-crystalline (CPET) because there is no 100% crystalline polymer, as shown in Figure (2.4) [40].

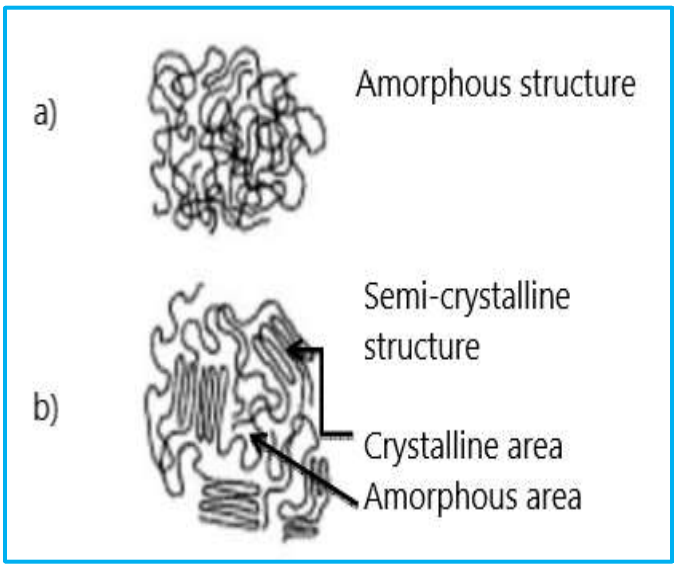
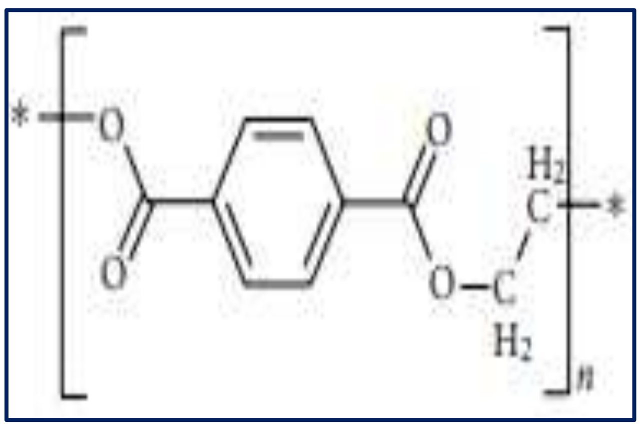


Figure (2.4): The arrangement of the chains during the solidification process a) Amorphous structure b) Semi-crystalline structure [40].

Polyethylene terephthalate (PET) is considered a recyclable material because it is a thermoplastic material that is softened by heat and hardened upon cooling. It is utilized in carpets, jackets, fillers, bottles, containers, films, sheets, strappings and making soft drinks bottles and water bottles because this material is a barrier to water and gas. The general characteristics of polyethylene terephthalate (PET) are that this material is characterized by resistance to chemicals, the resistance to corrosion, strength, toughness, transparency i.e. clear amorphous state on cooling, prevents water and gas leakage through it, temperature tolerance, wear resistance, and also chemically stable. The crystallinity of PET is an important property to measure, as it influences the mechanical properties [41]. Polyethylene terephthalate (PET) is one of the most common polymers which is recycled in abundance and has a special symbol to distinguish it from the rest of the polymers during the recycling process, which is the number “1” [41].

And the chemical structure of this substance is shown in Figure(2.5):



Figure(2.5): The chemical structure for (PET) material [42].

Polyethylene terephthalate (PET) is the most utilized material in the packaging of certain sorts of items. Packages made for PET are lightweight, clear, highly impact-resistant, do not chemically interfere with the contents, and are not toxic. Both of these distinctive features helped PET achieve a large position in the worldwide manufacturing market [43]. A typical semi-crystalline polyethylene terephthalate (PET) may have a transition temperature (Tg) of about 80˚C, a melting around 250

˚C, and crystallization temperature of 160 ˚C [44]. Polymer bottles are made of petroleum product (PET) and give large carbon emissions during manufacturing as well as they can be used in the manufacture of diesel fuel [45]. Usually, hot polyethylene terephthalate (PET) bottles crystallize faster than cold bottles. The hot polyethylene terephthalate (PET) bottles need to have higher thermal stability to withstand applications at higher temperatures [46]. Figure (2.6) shows the behavior of a semi-crystalline polymer upon stretching.

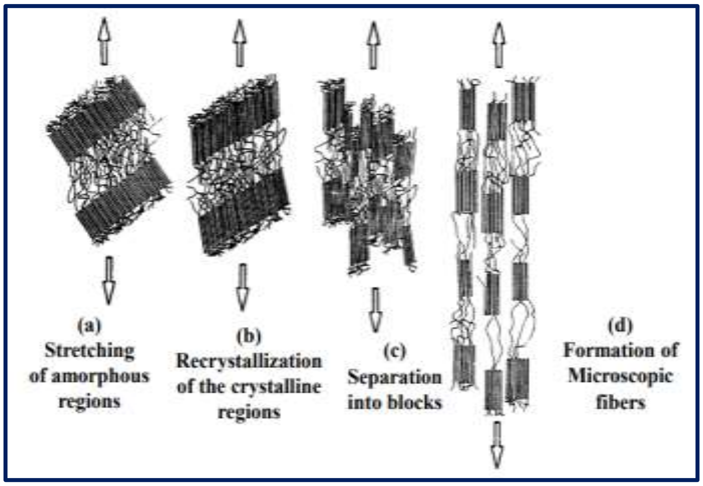


Figure (2.6): The polymer deformation stages of a semi-crystalline polymer [47].

2. 7. Low-temperature Grinding:

It is a special technique for grinding polymers and the result particles have equal dimensions smaller than (5 μm). This process is carried in an agitated media mill and special solvents such as [hexane and ethanol] at a temperature as low as [-80 ° C], as shown in Figure (2.7).

The fine polymeric particles that can be used in the first rapid stage are usually produced by polymerization or cryogenic grinding of thermoplastic materials and some other materials in impact mills in close contact with liquid nitrogen at a temperature [-196^0 C] or by dry grinding using solid carbon dioxide at [-78^0 C]. Polymer particles can be produced in another way [48].

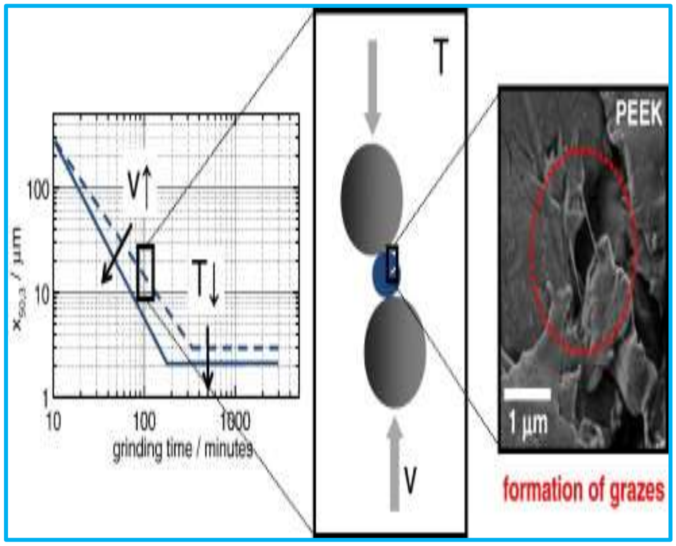


Figure (2.7): The wet grinding process for materials [48].

2. 8. Shredding Process:

Shredding is one of the most important and energy-intensive processes in the industry as in equation (2.1). The shredding of polyethylene terephthalate (PET) bottles is another processing method used to reduce waste PET bottles to flakes. This is achieved using a plastic shredding machine that is designed to reduce large plastic PET bottles into smaller pieces, shapes, or flakes. The disintegrated polymers, which look like little size particles, are collected in a holder constructed at the bottom end of the device (machine). The particles are taken for more processing into various items. Depending on the quality anticipated of the product to be constructed, the particles may be mixed at various proportions with virgin polymer (plastic) resins. This adds value to the shredded waste polymer bottles, making them raw materials for more processing. The polymer crushing devices are normally designed to have different production capacities and performance efficiencies [49-52].

$$L = L_1 + L_2 \quad \text{……………………} \quad (2.\ 1)$$

Where:

L: The energy exerted by external forces. (KJ)

L_1: The deformation energy. (KJ)

L_2: The energy to create a new space (a new shape of matter). (KJ)

2. 9. Cutting Mechanism:

Below is an explanation of the cutting mechanism [53]:

2. 9. 1. Cutting caused by the effect of the vertical forces:

It resulted by normal vertical forces (N), without any side effects of cutting knife (lateral force), the angle of friction ($\tau = 0$)as shown in Figure (2.8a).

2.9.2. Cutting without the slip, under the effect of the lateral component:

Caused by the effect of the normal vertical force (N) with some lateral force (T) and without slipping. In this type, the angle of the effect of the cutting tool is smaller than the angle of friction ($\tau < \varphi$), as a consequence. The cutting process is carried out under the effect of the normal force (shear -and- beater type), as shown in Figure (2.8b).

2.9.3. Cutting with the slip, under the effect of the lateral component:

Result from the perpendicular forces (N) with some lateral forces (T) and with slip. In this type, the angle of cutting is greater than the angle of friction ($\tau > \varphi$) and the cutting is sharp with the cutting tool, as shown in Figure (2.8c).

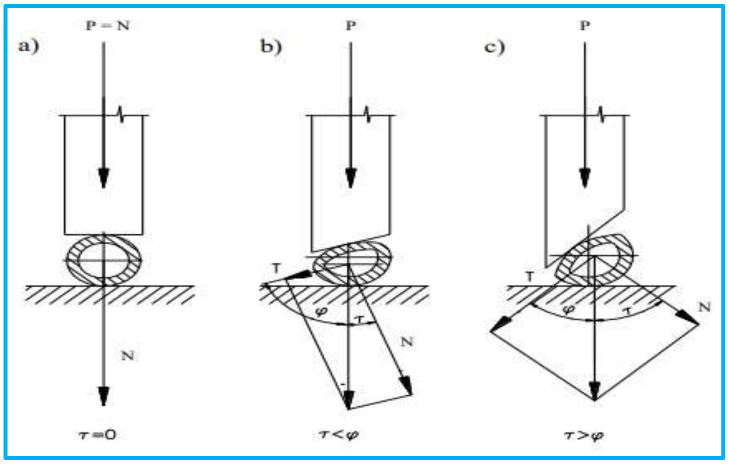


Figure: (2.8): The mechanism of cutting [53].

2. 10. Cutting Models:

Below is an explanation of the most important cutting models [50, 52 53]:

2. 10. 1. Hitter Shredder Model:

This type of cutting is a result of a knock that changes the polymeric pice, as shown in Figure (2.9).

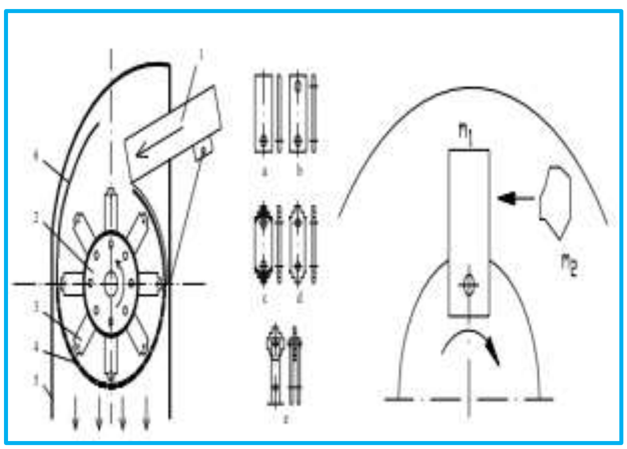


Figure (2.9): The hitter shredder model and the direction of movement of the racket.

The hitter shredder model can be represented by the following equation:

$$M_1 (V_1P - V_1K) = M_2 (V_2K - V_2P) \quad \ldots\ldots\ldots\ldots\ldots (2.\ 2)$$

Where:

M_1: The racket weight. (N)

M_2: The piece weight. (N)

V_1P: The initial velocity of the racket before the stroke.(m/sec)

V_1k: The final speed of the racket after the stroke.(m/sec)

V_2P: The speed of the piece before the smash.(m/sec)

V_2k: The speed of the piece after the smash.(m/sec)

2. 10. 2. Friction Cutting Model:

This type of cutting resulted from friction and crushing for brittle materials. The process of crushing takes place, because of friction between the materials and the discs. In this type of cutting model, the gap between the discs should be taken into account, as shown in Figure (2.10).

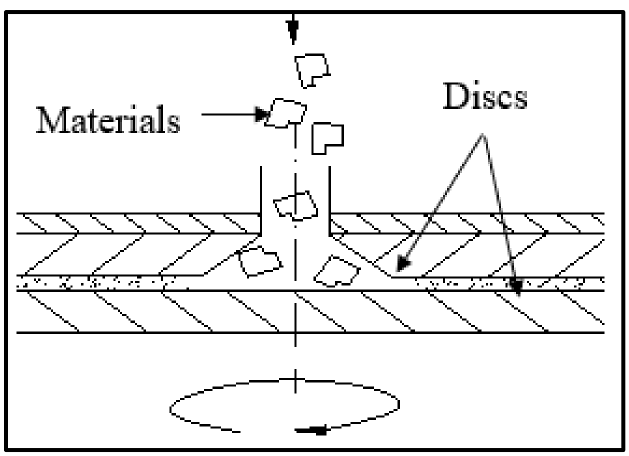


Figure (2.10): The friction cutting model.

2. 10. 3. Angle Cutting Model:

This type of shredding is the result of the striking of the cutting tool directed at a certain angle, as shown in Figure (2.11).

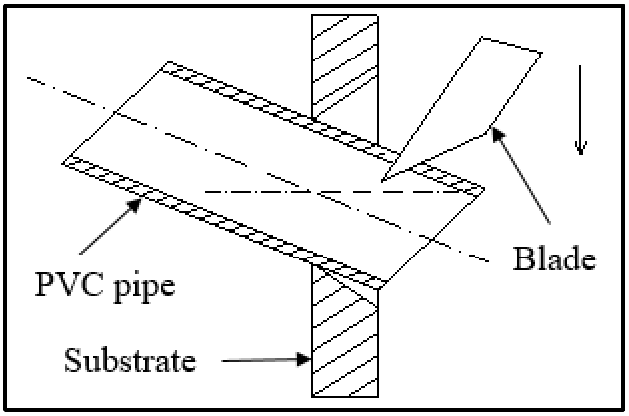


Figure (2.11): The cutting angle of the PVC pipe cutting in the multi-disc machine.

2. 11. Types of Cutting Machines:

2. 11. 1. Knife Cutting Machine:

It is one of the types of cutting machine used for polymeric materials that depend on the presence of knives in the machine, as shown in Figure (2.13), where there are rotating knives and others fixed on the wall of the machine, the cutting process takes place as a result of cooperation between the edges of fixed knives and the edges of moving knives. The cutting process depends on rotating knives, as shown in Figure (2.12) [54].

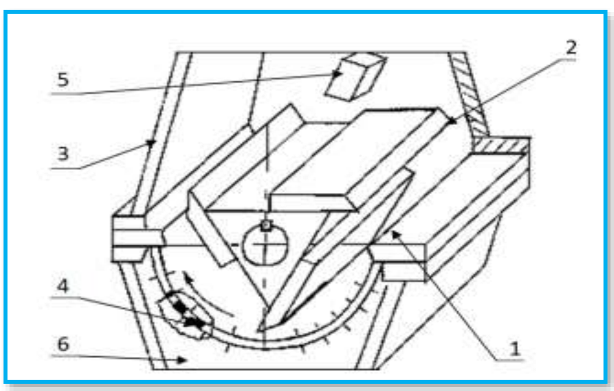


Figure (2.12): The scheme shows the cutting machine with knives and its main components: (1) constant knives, (2) rotary knives, (3) habitation, (4) sieving, (5) material Feed, (6) The primary grains(product).

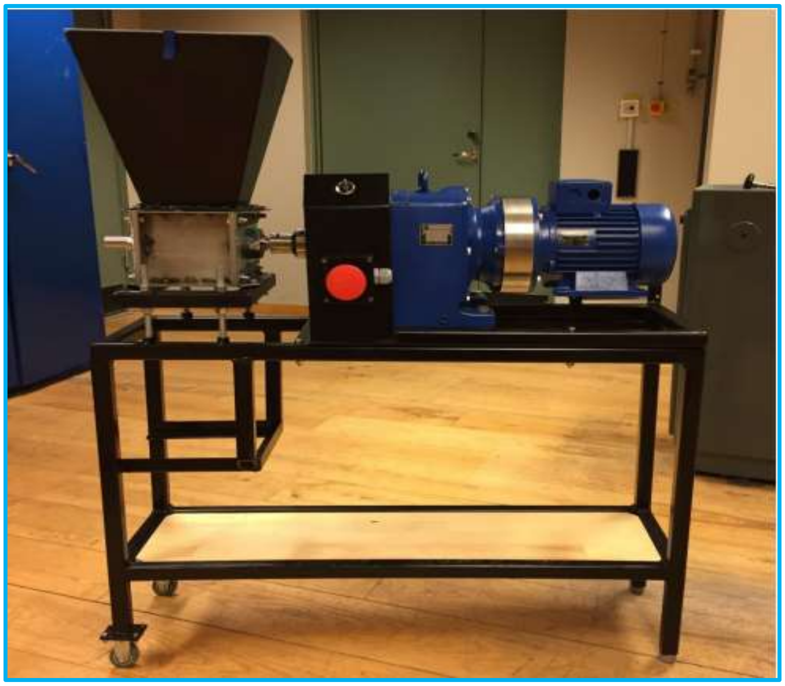


Figure (2.13). Shows a knife cutting machine (one of the types of polymer cutting machines) [55].

2. 11. 2. Multi-disc Cutting Machine:

It is a process that contains three or more discs and usually, the number of discs is odd (3, 5, 7) and each disc has slots, each slot is prepared at a certain angle, as shown in Figure (2.14) and the most important thing in this type of the machines it is the distance between the discs as this distance that is restricted by the type of material to be cut. Discs are usually made of steel for this type of machine and are intended for cutting thermoplastic materials. This type of machine is considered one of the best types of polymer cutting machines. It is easy to design, manufacture and operate, and its work efficiency is more than (80%) [54-57].

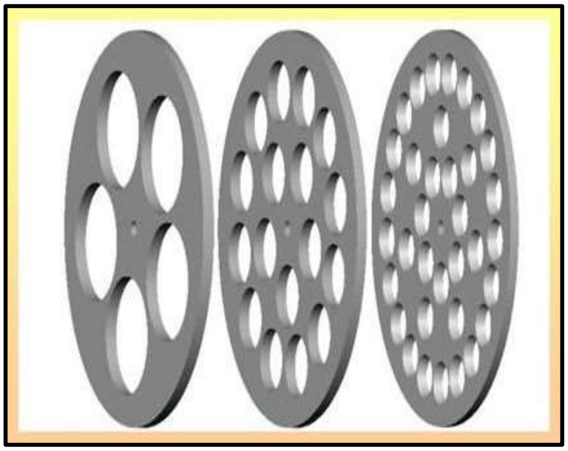


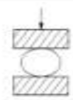
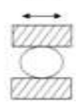
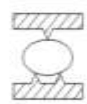
Figure (2.14): Shows the disks for the multi-disc cutting machine. Right: the shape of one of these discs. Left: the three disks with different slots [53, 54,].

After the cutting process, the materials are washed in some cases, and then these cut materials are transferred to granulation machines (granulation stage), where they are granulated or worked in the form of polymeric beads to become a raw material, that can be used to make new polymeric materials and products when they enter the formation stage, and it is done at this stage forming polymers into products for their use using forming techniques such as extrusion. Studies have shown that these machines are more efficient, productive, and have less energy consumption during the cutting, compared to other cutting machines. To analyze the regularity work of multi-disc crumbling machines, it is best to use the following parameters that have a direct relationship to the stability of the shredder to facilitate the formation of the shredded materials during recycling and to reduce consumption the electrical energy such as the crumbling resistance unit, The properties of the materials to be crumbled, The time taken for the system to work, irregular feeding of the material before cutting, irregularity of interlock and others, crumbling resistance and applied stresses when crumbling, etc… [56-62].

2. 12. Shredding Techniques:

The shredding process consists of reducing the geometrical dimensions of the material (reduced into smaller sizes) depending on the nature of the applied loads and the stresses arising in the material, as shown in Table2 [62].

Table (2.1): Shredding types (methods) according to the applied load.

Type	Load Model	Stresses
Crushing		Compressive stresses
Shear		Shear stresses
Abrasion		Surface pressures
Impact		Surface pressures
Breaking		Bending stresses

2. 13. Factors Affecting Energy Consumption during the Crumbling process:

Many factors affect energy consumption during the crumbling process:

- Total crumbling time.
- Crumbling resistance.
- Applied stresses when crumbling.
- The properties of the materials to be crumbled.
- Dimensions of the pieces before and after crumbling.
- Crumbling conditions.
- The angular speed of cutting edge.

The quality of the product depends on the intensity of the material contact with the cutting edges and the time of grinding. In the case when a smaller amount of material is located between the cutting edges causing less resistance of the elements) but when the amount of material increases the resistance of the elements increase. During the grinding process, some phenomena that occur in the machine affect the process of grinding such as grinding irregularity, energy losses by contact of cutting tools (elements) with the machine wall. It is very essential to define the relationship between grinding unit design and material flow during the milling process using a special program that fits the process of grinding because the design of the grinding unit can greatly affect the actual efficiency, the degree of grinding, and the consumption of electrical energy [56, 62, 63].

2. 14. Complete Shredding System:

The complete shredding system consists of:

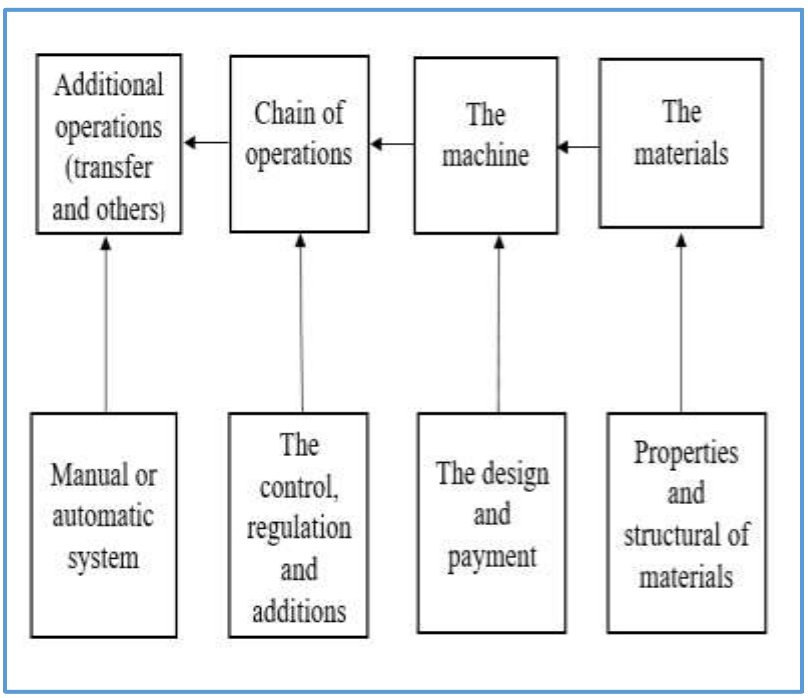


Figure (2.15): Diagram showing the complete shredding system [64].

2. 15. Extrusion Process:

It is a continuous process that is used to produce many things, including (sheets and pipes, etc.). It is also used in the production of a compound consisting of a mixture of material and reinforced material [65].

The thermoplastic granules are put in the hopper in this process and then these pellets are heated by the thermal heaters in the extrusion machine wall or by the mechanical movement of the screw to melt. After that easily fed the melt into the mold without any difficulty through the extrusion machine and a cooling cycle is added to the mold after the material stays within the mold [66], as shown in Figure (2.16).

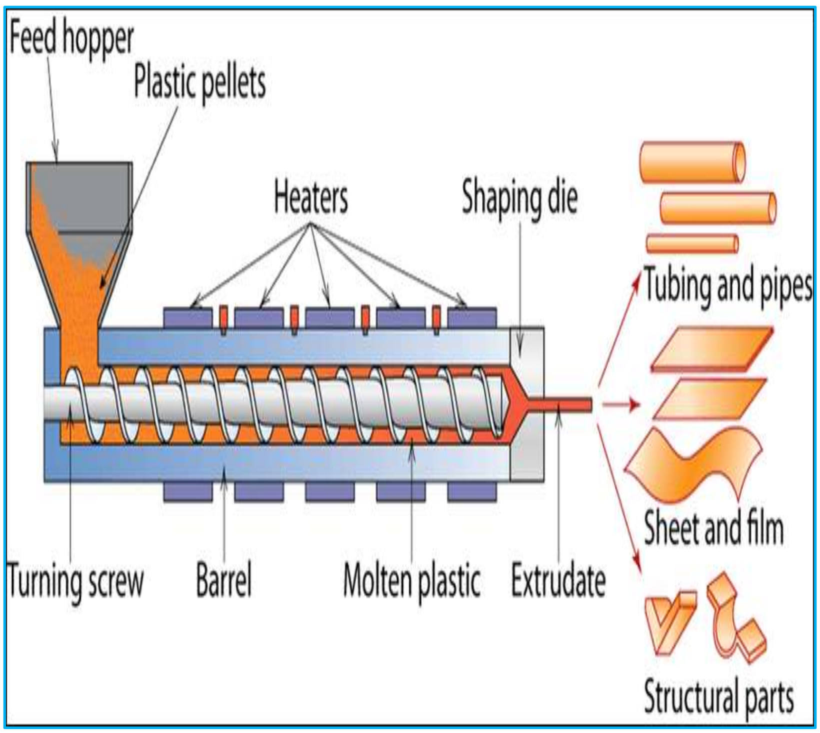


Figure (2.16): Shows the extrusion process for thermoplastic materials [67].

2. 16. The Polymer Types based on Recyclability:

It is known that polymers cannot be decomposed easily, so they need a long time to be decomposed, so they are recycled to benefit from them and preserve the environment from pollutants. On this basis, the polymers are divided into two types, which are thermoplastic materials and thermosetting materials where thermoplastic materials can be recycled, for example, polyethylene terephthalate (PET) and thermosetting materials, they cannot be recycled because they do not have properties similar to the properties of thermoplastic materials [68].

2.17. Comparison between thermoplastic materials and thermosetting materials:

2. 17. 1. In the arrangement of chains:

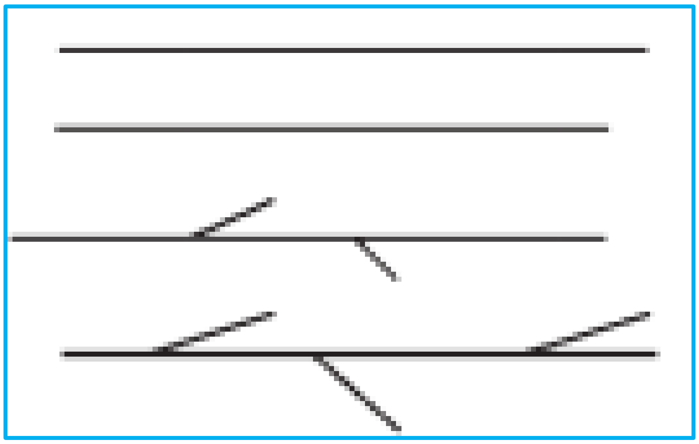


Figure (2.17): The arrangement of chains for thermoplastic materials [65].

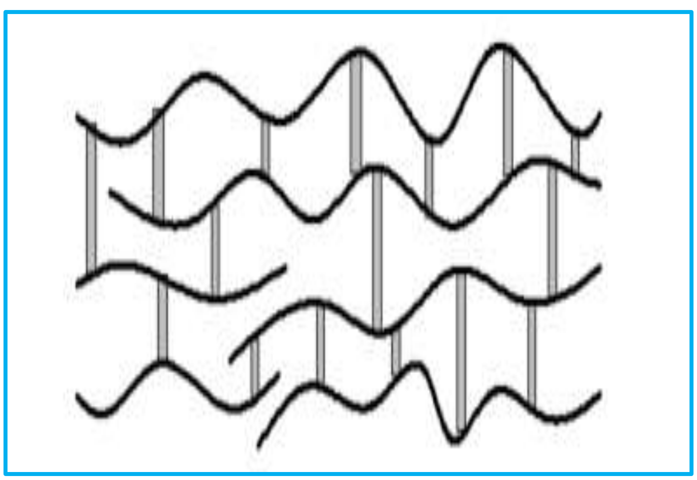


Figure (2.18): The arrangement of chains for the thermosetting materials [69].

2. 17. 2. In the Extrusion Process:

2. 17. 2. 1. Stages of Thermoplastic Material Forming Processes in the Extrusion Machine [70]:

A) Heating the polymer into the molten state.

B) Pumping the melt to the forming unit.

C) Forming the melt polymer into the required dimensions and shape.

D) Solidification and cooling.

2. 17. 2. 2. Stages of Thermosetting Material Forming Processes in the Extrusion Machine:

A) Feeding the material into the mold.

B) The pressure is applied to the mold.

C) Raise the temperature and stay in the mold for a period of the curing process.

D) Take out the product.

In the above process, we notice that the heating process takes place inside the mold only, because raising the temperature during forming breaks the crosslinking and thus leads to the failure of the material [71, 72].

2. 18. Types of the Extrusion Machines:

There are two kinds of machines which are single-screw machines and twin-screw machines. These two kinds of devices typically perform different functions, but the underlying concepts behind both are the same.

2. 18. 1. Single-Screw Extruders Machine:

The extruder with a single screw contains one screw called Archimedes screw which has a role in mixing, homogeneity of the polymeric material and can change the solid polymeric state through the hopper to prevent the overload of the solid polymeric material collected in the hopper. In this machine, thermoplastic pellets are placed through the hopper and these pellets are heated through the thermal heaters located in the wall of the single screw extruder so that they are easily pushed through this machine until it reaches the mold where the mold is the last zone of the flow stage of the molten polymeric material. The mold is the residential zone of the molten polymeric material [70, 73]. After that, a cooling cycle, which is cold water or any coolant liquid is applied to the mold for the polymeric material to be solidification inside the mold and produce the desired shape. Finally, the product is removed from the mold before it reaches room temperature [74], as shown in Figure (2.19).

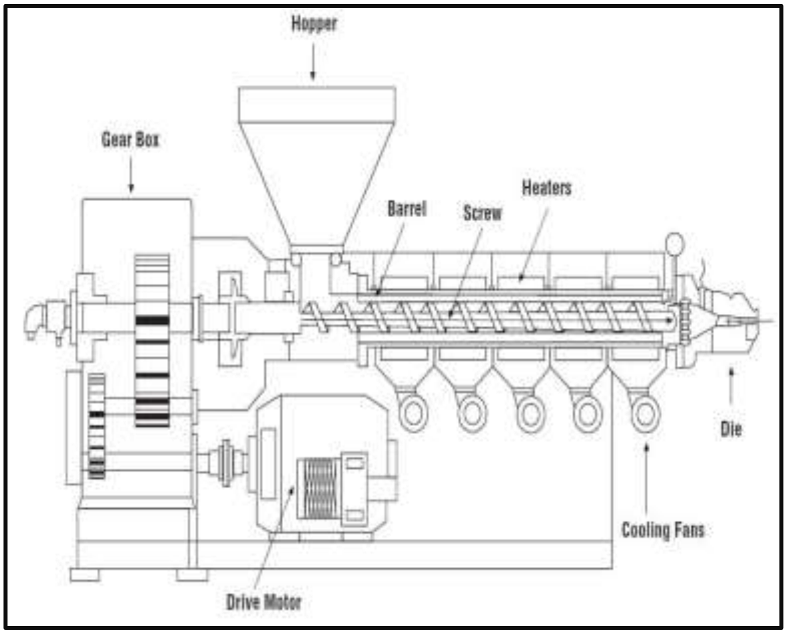


Figure (2.19): Shows single screw extruder device [75].

2. 18. 2. Twin-Screw Extruders Machine:

This machine has two screws the movement of one of the screws in an opposite direction to the direction of movement of the other and the twin screws are the basic structure of the twin-screw extruder machine. Where the double movement of the screws has an important role in controlling the temperature of materials. In the twin-screw extruder machine, the thermoplastic granules are placed in the hopper and then we apply high temperature until it reaches the melting stage through the heaters in the twin-screw extrusion machine for the material to be pushed into the mold easily after that, the pressure is suddenly applied to ensure the arrival of the material to the inside of the mold and then stay for some time for the solidification stage with a cooling cycle that was cold water or any coolant liquid, to gain stability in dimensions and then take out the product after the solidification stage before it reaches the room temperature for easy removal from the mold because at room temperature the product cannot be removed from the mold easily. After all, the thermoplastic will solidify in the mold [76-79], as shown in Figure (2.20).

The design difference between a single screw extruder machine and a twin-screw extrusion machine leads to controlling the effective mixing degree of the polymeric material, good homogeneity and heat distribution occur evenly for each part of the material. In addition that in the twin-screw extrusion machine can add auxiliary holes to remove volatile substances. Extrusion machines can be used to produce hollow sections, solid sections, sheets, strings, membranes, tubes, etc., while when the product is granules, it is considered a medium for its production by other additional processes such as injection molding or blowing [79,80].

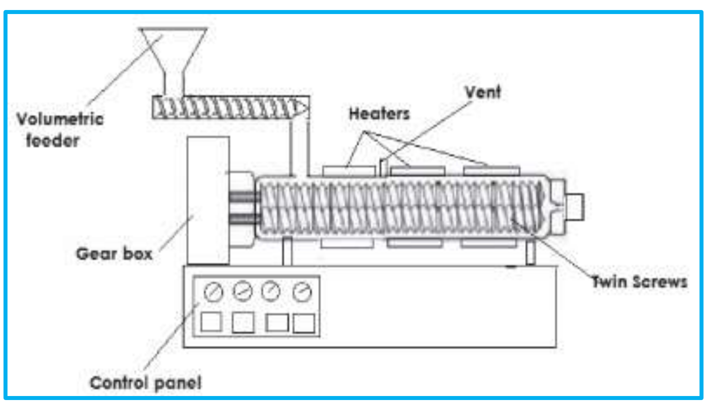


Figure (2.20): shows the twin-screw extruder device [81].

2. 19. Screw Design:

There are three zones for the screw in the thermoplastic material, and the screw design differs according to the type of polymer used, and the screw design does not include the three regions for some polymers:

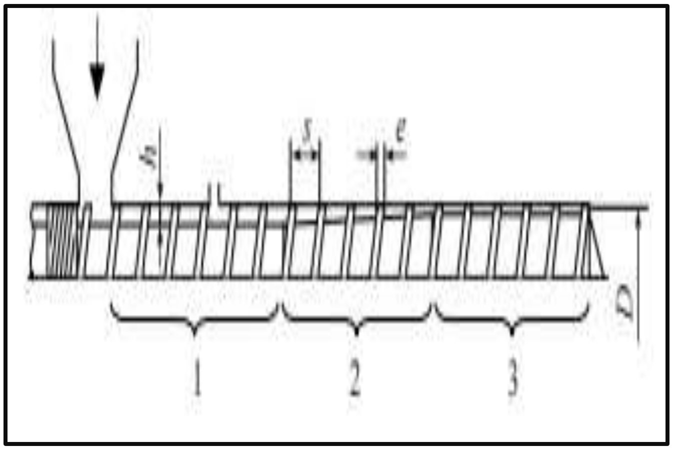


Figure (2.21): Shows the screw zones for thermoplastic material [82].

Most screws consist of the following zones:

2. 19. 1. Feed Region:

In this region, the resin is placed in the extruder and the depth of the channel is equal along this area, and it is called the transportation of solid materials [82, 83], as shown in Figure (2-21).

2. 19. 2. Melting Region:

In this zone, the polymer is partially melted, as the depth of the channel begins to decrease gradually and it has another name is the area of transition or pressure [82, 83], as shown in Figure (2-21).

2. 19. 3. Metering Region:

In this section, the depth of the channel is similar to the depth of the channel in the feeding zone, meaning that the depth of the channel is constant along this area with a small depth of the channel and the particles arriving in this area will dissolve, meaning that a uniform temperature is shed throughout the area for the particles to melt steadily [82, 83], as shown in Figure (2-21).

2.20. Method to Determine the Crystallinity:

The various analytical methods used to determine the crystallinity of a polymer namely, wide-angle X-ray diffraction (WAXD), density, differential scanning calorimetry. The usual procedure in measuring the degree of crystallinity by DSC involves drawing a linear arbitrary baseline from the first onset of melting to the last trace of crystallinity and determining the enthalpy of fusion from the area under this endotherm. The degree of crystallinity is then defined as:

$$X_c = \Delta H_f(T_m)/\Delta H_f^o(T_m^o) \qquad \text{(2.1)}$$

Where X_C is the weight fraction extent of crystallinity, ΔH_f (Tm) is the enthalpy of fusion measured at the melting point, Tm, and ΔH^o_f (T^om) is the enthalpy of fusion of the totally crystalline polymer measured at the equilibrium melting point, T^om [84].

2. 21. Shredding Stability Solutions for the Thermoplastic Materials during the Crumbling Process.

The crumbling processes for thermoplastic material consist of special and specific processes and devices for grinding the materials regularly without the occurrence of deterioration or chemical reaction or explosion that occurs otherwise. It is known that at normal temperatures, thermoplastic materials and other solid materials are not liquid but become liquid at extreme temperatures. Where the thermoplastic materials can be crushed by grinding machine into a fine powder by adding a liquid hydrocarbon for example (solid carbon dioxide, which is known as dry ice). The studied invention provided an enhancement to what was mentioned above [85]. Whereas, the invention suggested adding other liquefied gases for example (sulfur dioxide and nitrogen … etc.) at a ratio of (5% - 20%) with solid carbon dioxide to crush materials that cannot be easily crushed by using only solid carbon dioxide, and we find it difficult in the crushing or grinding process due to mechanical problems or due to chemical reaction in the absence of the liquefied gases for some materials. Also, the discovery found that it is possible to grind two different materials and obtain a mixture by dividing the particles precisely. Whereas the gases mentioned above and used in this invention achieved a dual function due to their lower temperature leading to the materials being cooled and avoiding agglomeration and also making the substances friable and easy to crush. When the grinding process continues, the emitted gases absorb the heat from the milling process and cover the substances with the non-reactive material, which leads to preventing these materials from coming into contact with air, which may chemically combine with the substances or causing a reaction with the substances [85, 86].

2. 22. Literature review

2. 23. Summary of Literature Review:

In 2008, Yatish Patel, et al, investigates the conditions of contact between polymeric materials and cutting instruments when exposed to orthogonal cutting to improve the performance of the device and enhance orthogonal cutting. Where work was continuing to develop the performance of the device by changing the inclination of the cutting tool at the range between [20º- 30º]. Where the cutting was made at different rates [0.01 m /s] and [0.1m /s] on PMMA, nylon 4/6, and nylon 6/12. This work matches what Robin said and takes into account the following variables as temperature rise, contact conditions, and inelastic distortion [87].

In 2011, Obinna Ihesiulor, et al, studied the construction of a plastic recycling machine that minimizes the limitations of the already existing (imported) ones to a great extent and at the same time ensures effective waste management. The results of the experimental analysis show that for every used plastic fed into the hopper, about the temperature of 200 ^{0} C is required to melt it. The machine employs the principle of conveying and heating to effect shredding and melting of the materials fed through the hopper and requires only two persons to operate. The machine is designed using locally available raw materials which make it cheap and easy to maintain and repair. The performance test analysis carried out defines the characteristics of the machine and shows that at a speed of 268 rpm the machine functions effectively and efficiently in performing its task producing a high finishing recycling efficiency or recyclability of 97%, takes 2 minutes to recycle a batch of plastics and has a recycling capacity/throughput of about 265 kg/hr which translates to a significant time. It is cost and energy-saving since its specific mechanical energy consumption is low at about 30.23kJ/kg. Experiments also show that for a batch process, the power requirement of the machine is proportional to the time in the process. [88].

In 2014, Chamil Abeykoon, et al, focused on investigating the total energy demand of an extrusion plant under various processing conditions while identifying ways to optimize the energy efficiency. Initially, experimentally measured signals of both materials were studied to understand the process energy demand over the different processing conditions. The data collected over the last minute at each screw speed were used for the evaluation. The average values of the experimentally measured mean total extruder power (TP), the level of fluctuations of the total power (DTP), mass throughput (MT), and specific energy consumption (SEC) of the extruder for both materials with different screws and processing conditions. As expected, both the mass throughput and the total power increased with the screw speed regardless of the material being processed and the screw geometry used. Conversely, the specific energy consumption of the extruder is reduced with increasing screw speed regardless of the material and screw geometry. In this work, a virgin HDPE and a virgin PS were used with three screw geometries and three-set temperature conditions and SEC reduced with the screw speed (in the range of 2600–650 J/g) for all conditions tested [89].

In 2016, Tolulope Olukunle, et al, designed the components of the shredder machine because the shredder has a very necessary role in disposing of polymer (plastic) waste and reusing it again, so it must be taken care of, and noted that the optimization of the shredding process, occurs when the components of the shredder are as follows, for example, the electric motor used for this machine operates at a capacity of 10 kilowatts (kW) with the rotation speed of 500 revolutions per minute (rpm), and the size of the cutting reel in the machine is 400 mm, where the electric motor is 1000 mm from the pulley. The shredder rotational speed is (300 rpm). Consequently, as a result, the optimization of the shredding process was obtained to get rid of polymer (plastic) waste and reuse it again to save the materials and reduce the consumption of electrical energy [90].

In 2018, Okunola, et al, studied the recycled PET bottle grinding and washing machine. The goal of this research is to develop and evaluate polyethylene terephthalate (PET) bottles washing and shredding machine to grind PET bottles to the required sizes with an area of $0.001m^2$ (10 × 10)mm for pelletizing plastic. The machine advanced performs shredding, cleaning, and washing of (PET) bottles using rotating blades arranged appropriately for grinding similar to a drill. Taking into account safety, easy operation, cost, and high efficiency, the machine can grind (50-75) kg of PET bottles. The shredding efficiency, recycling efficiency, and percentage retention are the parameters used to evaluate the performance of the device using a 5 Hp three-phase electric motor as the prime mover at different operating speeds such as (187.5 rpm, 273.86 rpm, 350.2 rpm) and the feeding rates are (1.0 kg/hour, 1.8 kg/hour, 2.4 kg/hour). The results showed that a high percentage of grind plastic increased to the required volume at 1.8 kg/hour and 350.2 rpm and this represents 60.01% of the machine's grinding efficiency. The recycling efficiency of the machine is 93.73% at 1.8kg/hr feeding rate and 273.8rpm, with the highest retention of (17.9%) at 185.7rpm and 2.4kg/hr feeding rate [91].

In 2018, Ngoc Kien Bui, et al, investigate the mechanical properties of Recycled PET Bottles Waste (RPET) and Recycled Woven Plastic Sack Waste (RWS) fiber-reinforced Recycled Aggregate Concrete (RAC) to reduce the amount of solid waste as a solution for waste management and preserve the environment. The experimental results indicated that RPET and RWS fibers have high chemical resistance in alkaline environments. The RWS and RPET fiber enhanced the post-cracking behavior of RAC. The contribution of RPET in the improvement of the RAC properties was better than that of RWS fiber although the RWS fiber has higher tensile strength than that of recycled PET (RPET) fiber [92].

In 2019, Ali Al-Zubiedy, et al, studied the improvement of the grinding process for the polymeric materials and the reduction of energy consumption for the recycling process of the polymeric materials through the regularity of the grinding of the polymeric materials. As they found through this research that the irregularity of the operation of the feeder for the granular material depends on the diameter (D) and the step of the screw (S). They found that the regularity of grinding for polymeric materials increases when (S / D) is equal to 0.25 or 1.67. The results showed that the process of the regularity of grinding for polymeric materials is when S / D = 1.67 it is better than S / D = 0.25 [93].

In 2019, Ali Al-Zubiedy, study to identify the products of the grinding process for the recycling of elements, installations, and materials made from polymers was proposed. The ground material was analyzed, evaluated, and then described using the latest granulometric models. Based on the stability models, a mathematical description of the granulometric distribution of particle size of the recycled polymer products obtained in the grinding process was found. The elements were passed through sieves with diameters φ = 3, 5, and 10 mm. The performance characteristics, statistical analysis, and evaluation of the grain size and dimensions of the products obtained by grinding recycled polymeric materials (PP, PS) are shown. The results of sieve analysis enable us to determine the contribution of individual fractions Xi, according to the equation obtained from this research [94].

In 2019, Nasr. F.M et al, developed the blades specific by three edges to cut the polyethylene terephthalate (PET) bottle using Solid Works and analyze the static stresses and strains of the cutting blade by using the ANSYS program (Finite element type solid 185). These blades are made of low-carbon steel, as the three-edged blades are designed and engineered using a solid work program. The results show that the stresses apply in the process of chopping PET polymer material are well lower than the yield stress of blade material [95].

In 2020, Hillary Onyishi, et al, studied the behavior of Semi-crystalline polymers changing their mechanical properties upon processing due to the changes in the crystallinity of the polymer. This paper investigates the changes in the crystallinity and mechanical properties of biaxially stretched polyethylene terephthalate, PET, for different stretch ratios (or strains). Each stretched PET sample was heated to 100°C in a biaxial stretching machine before being stretched to the desired stretch ratio. The same strain rate of 2/s was used for the stretching and the mode of deformation was simultaneous on either side. Differential Scanning Calorimetry (DSC) was used to investigate the variation of induced crystallinity with stretch ratio. Tensile tests were used to determine the variation of the processing stretch ratios with mechanical properties. The results show that the higher the stretch ratio, the higher the amount of induced crystallinity in the PET sample. Also, the higher the stretch ratio, the better the mechanical properties [96].

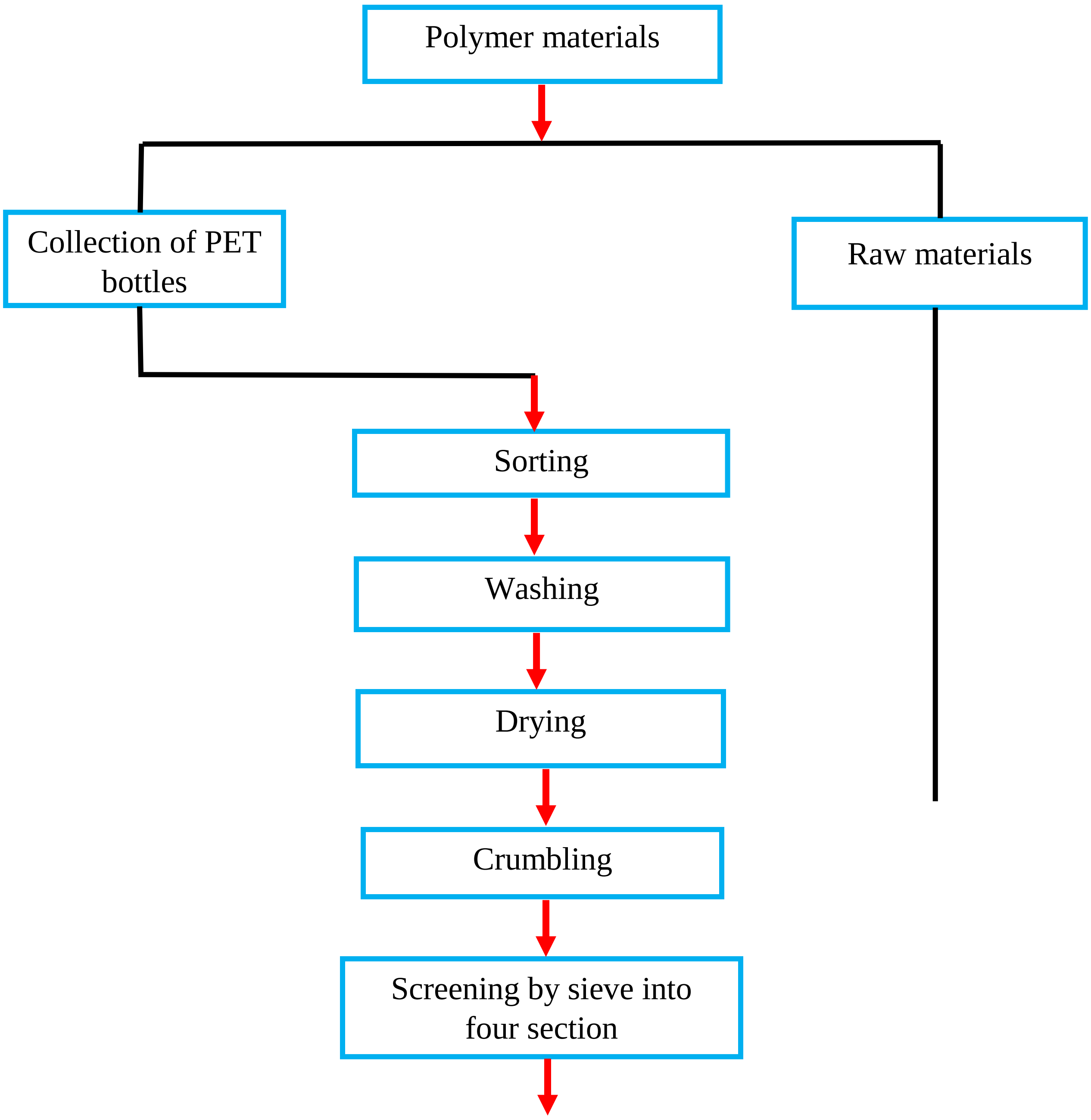
In 2021, Hui-Jin Um, et al, studied the crystallinity of polyethylene terephthalate (PET) variation according to the cooling rate to relate it to the mechanical properties of PET matrix-based carbon fiber (CF)/PET composites. The thermal characteristics and crystallinity were analyzed through differential scanning calorimetry (DSC) and X-ray diffraction (XRD) analysis. The mechanical behavior of CF/PET composites with different degrees of crystallinity was studied with varied temperature conditions. The failure modes of the fractured CF/PET composites were observed by using a digital microscope and scanning electron microscope (SEM). As a result, the crystallized CF/PET composites were improved 11.6 times higher in in-plane shear (±45° laminated) strength and 3.78 times higher in shear modulus than that of amorphous CF/PET composites at high temperature [97].

Chapter Three

Experimental Part

3.1. Introduction:

In this chapter, we will talk about the mechanism of our work that was conducted at the laboratories of the University of Babylon, as this chapter includes all the materials that were used in this research and it also includes the methods used for preparing the samples, as well as all the tests that were used in this research. The steps of the practical part can be illustrated, as shown in Figure. (3.1):



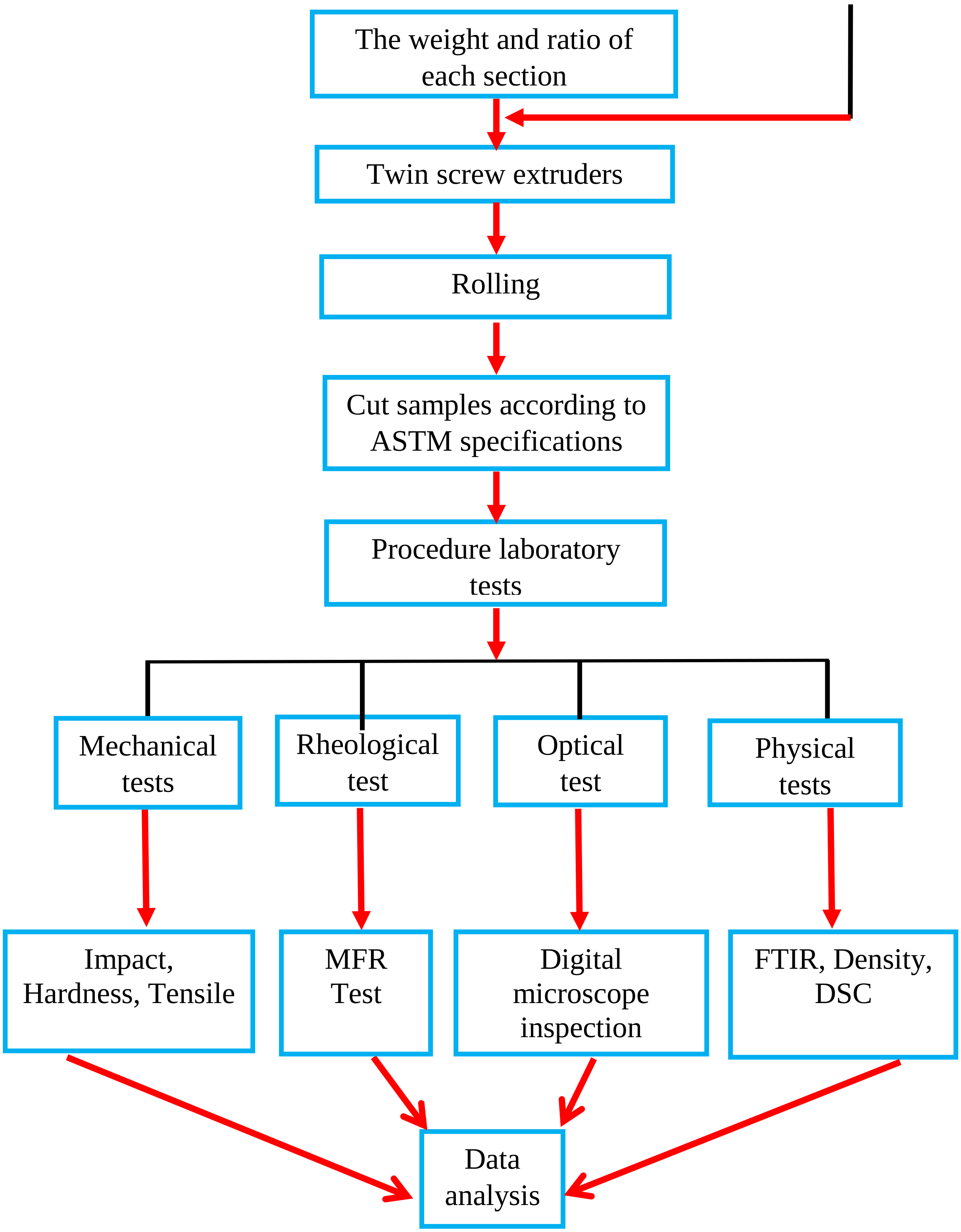


Figure (3.1): The steps of the experimental part.

3. 2. The Used Materials:

Recycled polyethylene terephthalate (PET) bottles were used from waste, also, used the raw material for polyethylene terephthalate (PET) with a purity of 99.8 percent, melting point (249±3 °C) obtained from Al-Muhannad Company for the production of the polymer as granular materials, Iran, product code U + 2673. The characteristics of polyethylene terephthalate (PET) are shown in Table (3.1).

Table (3.1): The characteristics of virgin polyethylene terephthalate (PET).

Characteristics	Units	Values
Elastic modulus	[MPa]	[2500-2850]
Density	[g/cm^3]	[1.37]
Tensile strength	[MPa]	[45-90]
Color	_	[Transparent]
Thermal stability	[°C]	[40-170]

3. 3. Sample Preparation Methods:

The 400 bottles from polyethylene terephthalate (PET) was collected and then cleaned in the laboratory with sodium hydroxide (NaOH) and warm water and dry through direct exposure to sunlight (≈50 °C), then they are cut by a knife cutting machine, and after the cutting stage, the product is sorted to different pieces by sieving, through this process we obtained three types of (PET) pieces as shown in Table (3.2). The first type is small pieces with a length (≈4.75mm) and ratio (47.924%), the second type is medium pieces with a length of (≈6.7mm) and ratio (23.467%), and the third type is big pieces with a length of (≈10mm) and ratio

(16.854%). Mixing part of these three types to obtain mixed pieces with a mean length of (≈7.15mm) and ratio (11.754%) to the comparison between it and the other three types. At the same time, the raw material for (PET) was prepared, after that, the different pieces of recycled PET and raw material were extruded by a twin-screw extruder machine (SIJ-30A), at a temperature ranging between (200-205 °C). The sheets are obtained by the two rollers in the twin-screw extruder device, and then the sheets resulting from the extrusion process are cut to perform the required tests according to the standard specifications.

Table (3.2): The sieve dimensions for different pieces of recycled PET bottles.

Type of crumbling for polymer materials	PET material
Sieve dimensions for small pieces (≈4.75mm)	
Sieve dimensions for medium pieces (≈6.7mm)	

Sieve dimensions for big pieces (≈10mm)	
Sieve dimensions for mixed pieces (≈7.15mm)	

3. 3. 1. Twin-screw Extruder:

In this process, there are different pieces (different shapes) for recycled polyethylene terephthalate (PET) bottles, were extruded using a twin-screw extruder as shown in Figure (3.2) with standard characteristics as shown in Table (3.3), and these pieces are the small pieces with a length of (≈4.75 mm), medium pieces with a length of (≈6.7 mm), big pieces with a length of (≈10 mm), and mixed pieces with a mean length of (≈7.15mm). Where each material was placed separately in the twin-screw extruder and then the materials were heated by the thermal heaters in the wall of the machine, for the materials to be transferred easily through the cylinder of the extrusion machine to the rolling stage by the screws in the extrusion machine so that each material is rolled separately and to obtain the final shape of each material.

It was observed during the extrusion process that the small pieces consumed less electrical energy, compared to the rest of the other different pieces, except the raw PET (new material) and also, the load applied on the machine decreases when the temperature rises, and consequently, the electrical energy consumption decreases.

The depth of the twin-screw channel is reduced along the length of the screw to compact the material.

The extrusion process was carried out for recycled polyethylene terephthalate (PET) with different cutting lengths and raw material at a temperature ranging between (200-205^0 C) and the screw speed used is 50 (rpm). It was noted that these used conditions are the ideal conditions for the extrusion process for all the above pieces because when the temperature rose to 220^0 C, a degradation occurred in the samples, as shown in Figure (3.3).

Figure (3.2): The twin-screw extruder device.

Figure (3.3): The degradation of the sample above 220 ^{0}C during the extrusion process.

Table (3.3): The properties of the twin-screw extrusion device.

Specifications of the twin-screw extruder	Data
Model	SLJ – 30 A
Diam of screw	30mm
Speed of screw	0 to 320 rpm
Output	20 Kg / h
Weight	450 Kg
Screw length	16D
Screw design	Double screw
Place of origin	Shandong - China
Certification	ISO9001

3. 3. 2. Rolling Process:

Is the process of polymer compression using the rollers (two circular cylinders), located in the end part of the twin-screw extruder device to obtain sheets with less thickness resulting from the compressive forces of the rollers on the polymer product. The thickness can be controlled by using levers in the machine. As a result of this compression, the sample will be extended and get in the final shape. The rolling process is the last stage of the twin-screw extrusion process. In this study, hot rolling was used for rolling polymeric products coming out from the twin-screw extruder device to obtain the sheets for each different pieces of recycled PET passed in the extrusion process, as shown in Figure. (3.4).

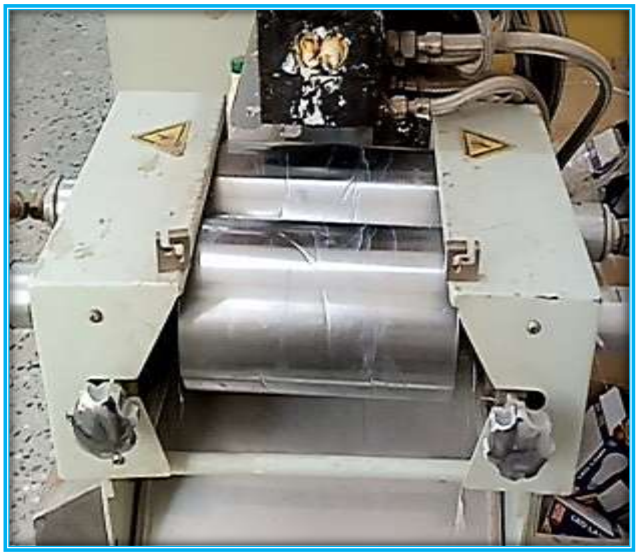


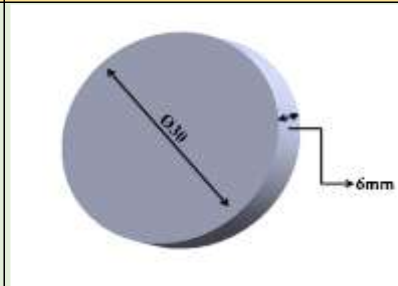
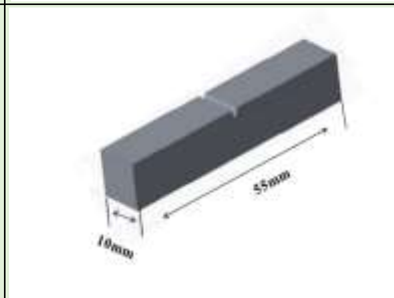
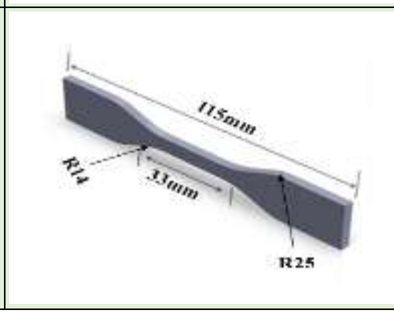
Figure (3.4): The rolling process for (PET) material.

3. 4. Samples Cutting:

Samples were prepared for mechanical tests. Mechanical tests include tensile tests, where the samples are cut according to the ASTM standards, as shown in Figure (3.5), as the dimensions of the tensile test, as shown in Table (3.4). Hardness test and impact test where samples cut according to the standard specifications and required dimensions as shown in Table (3.4).

Figure (3.5): The die for the tensile test of the samples according to ASTM (D1708-02a).

Table (3.4): The standard dimensions for testing samples.

NO	Type of test	Sample dimensions	ASTM
1	Hardness test (shore D)		(D 2240)
2	Impact test (Charpy method)		(D-22885)
3	Tensile test		(D1708-02a)

3. 5. Tests:

3. 5. 1. Hardness Test:

The (shore D) device was used to determine the hardness of extruded sheets resulting from PET bottles of different pieces for recycling. The testing samples were cut with standard dimensions according to ASTM (D2240), shown in Table (3.4). This test is carried using the device model (TH 210 FJ, Germany) as shown in Figure (3.6). The (shore D) device contains a needle-like indenter at the end of the measuring tool to determine the hardness of the samples. And the needle is directed in a direction perpendicular to the sample and the device is controlled by the lever and when performing the hardness test for samples at room temperature, the reading is taken after (0.5 minutes) from the start of the test and an average of four tests of each sample in different locations are calculated to obtain the required accuracy.

Figure (3.6): The hardness test device using (shore D).

3. 5. 2. Impact Test:

A Charpy method was used to determine the impact strength of extruded sheets resulting from PET bottles cut in different pieces for recycling. In this way, impact load is suddenly applied to samples. The samples were cut with standard dimensions according to ASTM (D-22885) as shown in Table (3.4). This test is carried using device equipes by (pendelschlagwerk, gunt, Hamburg, Germany) as shown in Figure (3.7). The impact test was performed at room temperature and the impact strength was calculated for each sample by the amount of absorbed energy of the material during fracture, through the energy indicator in the device, where the standard sample is fixed horizontally in the right place in the device so the effect of the impact is in the middle of the sample to obtain accurate results. After that, the pendulum was lifted and fixed at a height of (ho), i.e. the height of the pendulum before the impact test, and the energy indicator was reset, to start the impact test for each sample, after that the pendulum was released on the sample, with a specific impact force and the height became (hf), i.e. the height of the pendulum after the impact test and the sample begins to rapid failure as a result of the sudden impact force on it and calculates the amount of absorbed energy of the material during fracture through the energy indicator, as the impact strength is determined for each sample according to the equation below:

$$I.S = Uc/A \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \ (3.\ 1)$$

Where:-

I.S: Impact strength of the material (kJ/m^2).

Uc: Impact energy (kJ).

A: cross-sectional area of the sample (m2).

Figure (3.7): The impact test device using a Charpy method.

3. 5. 3. Tensile Test:

In the tensile test, samples were cut with standard dimensions according to ASTM (D1708-02a) as shown in Table (3-4). This test is done using the device model (WDW-5E, China) as shown in Figure (3.8). The two ends of the sample were connected to the jaws of the device as shown in Figure (3.9) to start a tensile test at room temperature by computer control, where a tensile test was performed with the speed of (10 mm/ min) and applied load (5 KN) until the failure occurred in the sample. The test was performed to determine the tensile properties such as elongation, tensile strength, elastic modulus, and a stress-strain diagram.

Figure (3.8): The tensile test device.

Figure (3.9): Shows the jaws of the tensile tester.

3. 5. 4. Infrared Fourier Transform Spectrometer Analysis:

Infrared Fourier transforms spectrometer analysis technique is used to know the type of materials and their chemical structure using (IR Affinity-1 Shimadzu –Japan) device, as shown in Figure (3.10).

For the samples to be examined, the device must be calibrated using (KBr), then the sample powder is mixed at room temperature with KBr (the mixing ratio is 99% KBr), as the mixing process must be with high accuracy, to obtain accurate results, then the mixture is pressed in the form of a semi-transparent disk to measure the possibility of penetration of radiation.

Figure (3.10): (FTIR) analysis device.

3. 5. 5. Differential Scanning Calorimetry Test:

A differential scanning calorimetry device (DSC) is used to determine the thermal transfers of extruded sheets resulting from PET bottles that are cut in different sizes for recycling. The test was performed using the device (SH1MADZ-4 DSC-60, Japan) as shown in Figure (3.11), according to the ASTM (D3418-03). A powder weighing 0.004 g was prepared from the samples to be tested, then it was pressed into the mold, after that it was placed in the device to perform the test. The samples were examined at the presence of nitrogen gas and the heating rate for each sample is 10° C/ min. The heating temperature used in the test is 280^0 C.

Figure (3.11): The differential scanning calorimetry (DSC) device.

3. 5. 6. Melt Flow Rate (MFR) Test:

The device (Shi Jia Zhuang Zhong shi testing machine) is used to calculate the melt flow rate (MFR) for all samples according to the international standard (ISO 1133: 2005), as shown in Figure (3.12), where the melt flow rate (MFR) of all samples were measured at a temperature of 255° C and a constant load of 2.16kg. During the test, the basic parameters must be specified such as temperature, applied load, cutting time, and cutting interval, after that, granules weighing 4g of each sample are placed into a heated barrel. After preheating, the test is started and the load is placed on the sample and after flow through the die, the polymer molten is cut into two pieces and the weight of these pieces is calculated. After that, the average weight of the two pieces is entered into the device to calculate the polymer flow rate (g / 10min) by the following equation:

Melt flow rate (MFR) = t ref *w / t ……………………. (3. 2)

Where:

t ref: 10min/ 600sec.

W: average weight of cutting time of the sample.

T: time.

To calculate the shear rate on the aperture walls at which the polymer flows at a constant temperature and constant load, the following equation is used:

$$\gamma = (1840/\rho)*MFR \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (3.\ 3)$$

Where:

γ: The shear rate at the wall (s^{-1}).

ρ: density of polyethylene terephthalate g/cm^3.

MFR: melt flow rate g/10min.

Figure (3.12): The melt flow rate device.

3. 5. 7. Density Test:

Density test is used to measure the density with digital accuracy = (0.0001 $g/\ cm^3$) at room temperature for all samples cut into different sizes (different shapes) for recycled polyethylene terephthalate (PET) bottles using the device High Precision [DENSITY TESTER (GP-12OS) Matsu Haku, china], as shown in the Figure (3.13), according to the ASTM (D-792) by placing each sample in a liquid. The same material may show different values in density tests this may be related to the change in crystallinity or absorption of solvent presences or removal of plasticizer or other reasons in our case the crystallinity is the main reason.

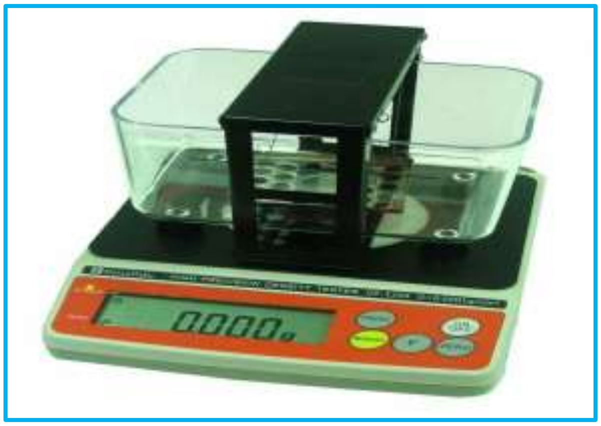


Figure (3.13): The density test device.

Two testing methods are depending on the liquid used for the test in our case we used solid plastics in water which is identical to method A, according to the ASTM (D-792).

3. 5. 8. Digital Microscope Inspection:

A digital microscope inspection was carried on PET sheet after extrusion process using AM4815T Dino-Lite Edge device with magnification rate (20x~220x) as shown in Figure (3.14) to find the relationship between the lengths of cutting with the density of bubbles as well the surface finish of the samples after the extrusion process.

Figure (3.14): The digital microscope device.

Chapter Four

Results

And

Discussion

4. 1. Introduction:

This chapter includes all the experimental results that are obtained from the physical, mechanical, optical, and rheology tests for recycled PET bottles with different cutting lengths under investigation.

The experimental tests and the results of extruded sheets system were used to investigate as follows:

a) Mechanical properties including hardness test, tensile test, and impact test.

b) Physical properties including density test and differential scanning calorimetry (DSC) test.

c) Chemical properties by using Fourier transform spectroscopy (FTIR) test.

d) Rheology properties by using the Melt flow rate (MFR) test.

e) Morphology properties by using digital microscope inspection.

In this chapter, the results of all samples that have been selected for extruded sheets system are presented:

- Small pieces
- Medium pieces
- Mixed pieces
- Big pieces

In the current chapter, the results that have been obtained from the tests above are discussed, in the beginning, discussed the DSC test to verify the thermal characteristics of the polymers with different cutting lengths and then correlate it with other tests that were performed in the laboratory.

4.2. DSC Results:

The differential scanning calorimetry device was implemented to inspect the effect of the mechanical cutting process in other words the piece length on the glass transition temperature (Tg) and melting temperature (Tm) as well as the percentage of crystallinity that may affect the final product mechanical and thermal properties. The results show the crystallinity percentage reduced with increasing the piece length as well as the melting and transition temperature, this behavior may be related to the orientation on the orientation of chains and the dispersion of temperature on the material to be extruded that may be related to the homogeneity of the extruded sheets, this behavior may give an indicator of selecting proper extrusion conditions.

After calculating the enthalpy of crystallization and the enthalpy of fusion, the melting temperature, degree of crystallinity, and glass transition temperature are obtained (appendix A), as shown in Table (4.1):

Table (4.1): The DSC results of all extruded sheets resulted from PET bottles cut in different pieces for recycling.

Cutting lengths (mm)	ΔHf (l/g) sample	Tm (^{0} C)	Tg (^{0} C)	% crystallinity
4.75	-6.126	251.304	100.5	22.776
7.15	-4.824	251.4968	88.025	17.934
6.7	-4.374	251.129	87.5	16.28
10	-3.4345	250.422	87	12.67

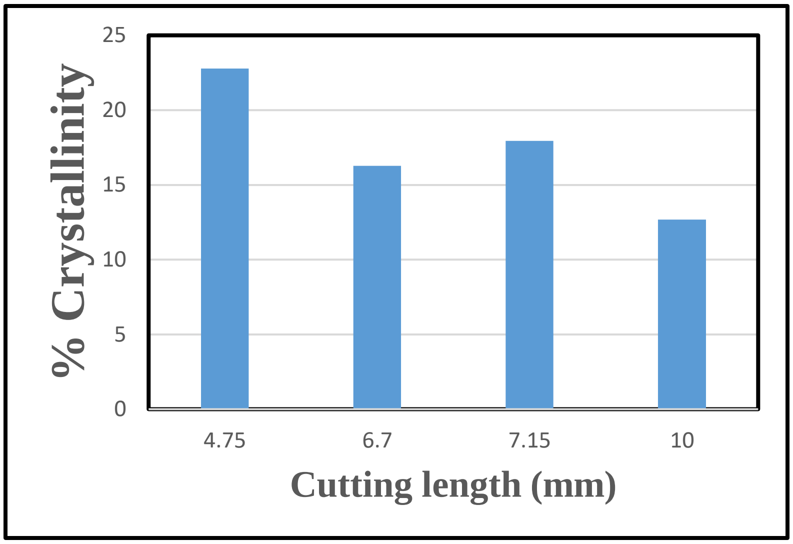


Figure (4.1): The relation between crystallinity and the cutting length of all extruded sheets resulted from PET bottles cut in different pieces for recycling.

By plotting the relationship between the crystallinity and the cutting lengths the result shows that the crystallinity increased with the decreasing the cutting lengths.

As expected, the glass transition temperatures, as well as the melting temperature, are affected sharply by the effect of cutting lengths, as shown below:

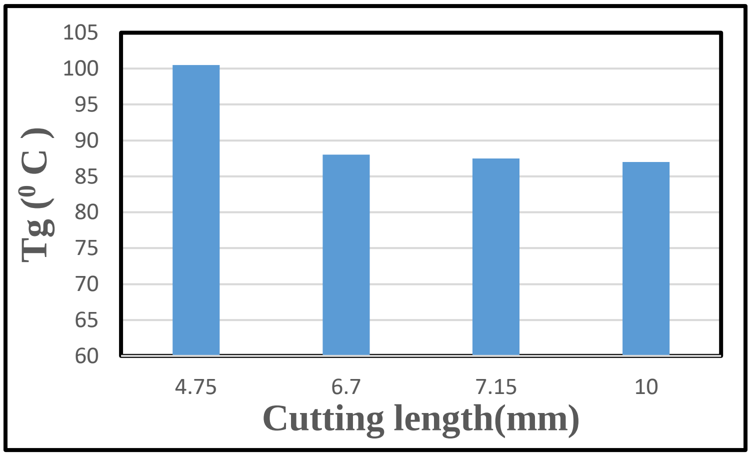


Figure (4.2): The relation between glass transition temperature and the cutting length of all extruded sheets resulted from PET bottles cut in different pieces for recycling.

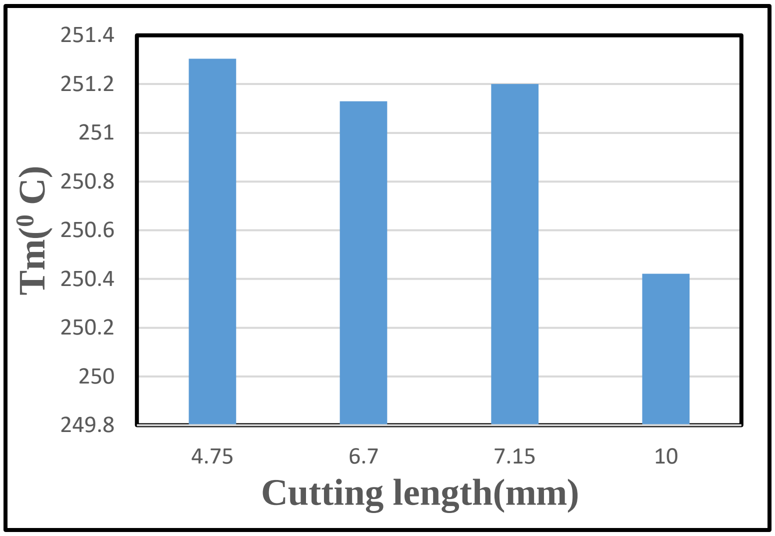


Figure (4.3): The relation between melting temperature and the cutting length of all extruded sheets resulted from PET bottles cut in different pieces for recycling.

4. 3. The Effect of Crumbling Length on Energy Consumption:

The energy consumption was calculated by the current indicator in the twin-screw extruder. It was observed during the extrusion process that the smaller pieces consumed less electrical energy, compared to the rest of the other different pieces as shown in Figure (4.4)

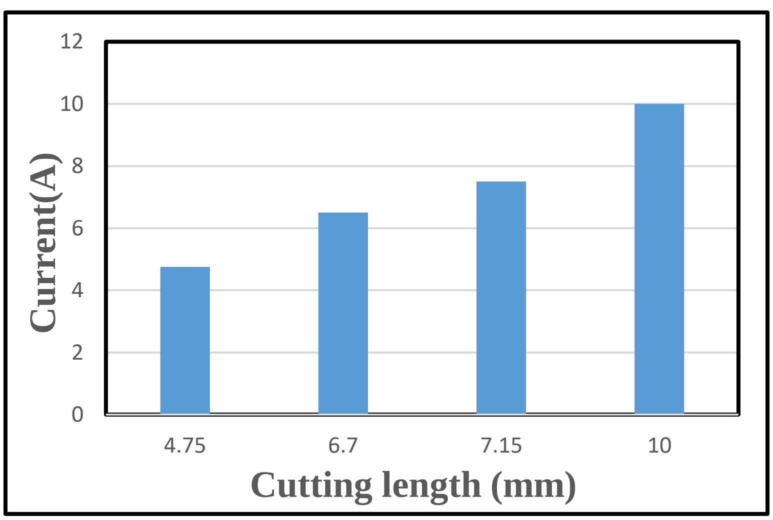


Figure (4.4): The relation between energy consumption and cutting length during the extrusion process.

4. 4. Mechanical Tests:

4. 4. 1. Hardness Test:

The hardness test is done using shore "D" type used with thermoplastics are performed on all samples resulting from the extrusion process of different cutting lengths of recycled PET bottles. However, the hardness value drops noticeably with increasing the cutting length. For better comparison among the samples, all tests were done using the same conditions, preserving the hardness scale. As shown in Figure (4.5).

Table (4.2): The hardness of recycled PET with different cutting length.

Cutting length(mm)	4.75	6.7	7.15	10
Hardness (shore D)	62.33	48.77	57.46	36.1

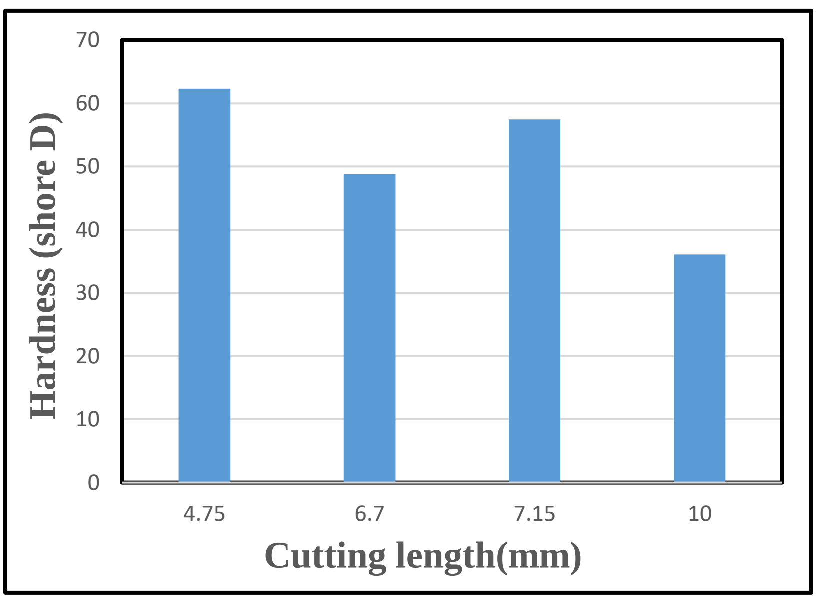


Figure (4.5): The hardness of all extruded sheets resulted from PET bottles cut in different pieces for recycling by using (Shore D).

That may indicate the relation between the hardness of the samples with the cutting length. In addition to that when comparing the hardness results with crystallinity values, there is a direct relationship among them, so hardness increases with increasing crystallinity. The arrangement of chains and fibrils of crystals decreases the free volumes between the chains they leave a small percent of voids that decrease the mechanical properties of the material [98, 99].

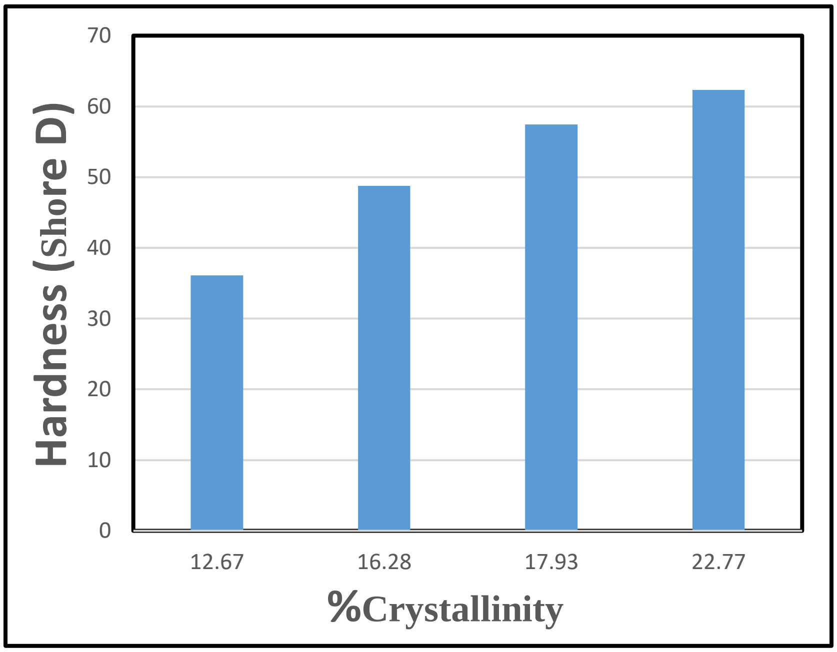


Figure (4.6): The relation between the crystallinity and the hardness of all extruded sheets resulted from PET bottles cut in different pieces for recycling.

As expected, the results show that the hardness is directly related to the crystallinity a linear relationship is sufficient to correlate the variables.

4. 4. 2. Tensile Test:

The tensile test is carried on a universal test machine on all samples resulting from the extrusion process of different cutting lengths of recycled PET bottles to investigate the tensile properties of the specimen, as shown in Figure (4.7).

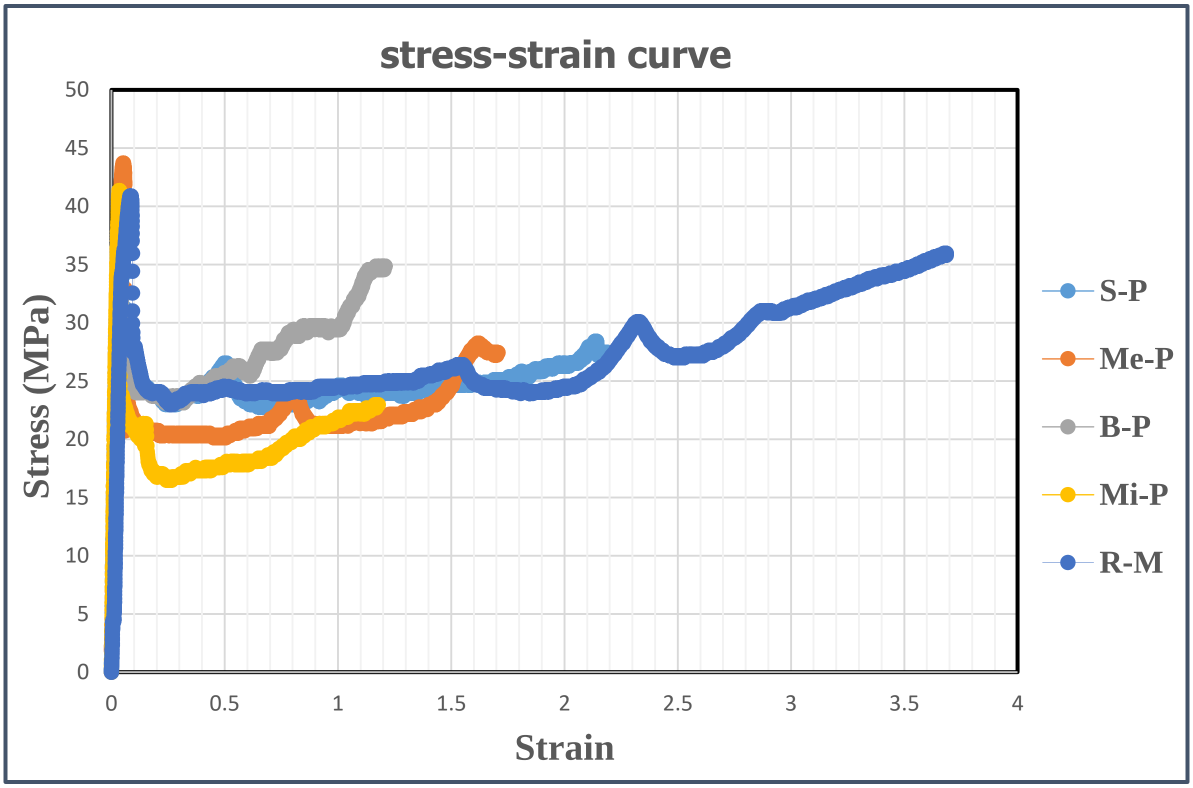


Figure (4.7): The stress-strain diagram of all extruded sheets resulted from PET bottles cut in different pieces for recycling with the raw material sample.

A testing machine (WDW-5E) is used for performing mechanical tests. This tensile test was performed with the speed of (10 mm/ min) and applied load (5 KN), which is recommended in standards for extruded polymers as in the study's case. All tests were carried out under the same conditions of humidity and temperature. The load should normalize the arrangement of polymer chains along the polymer samples, there for the higher degree of crystallinity should show higher tensile behavior. Fractures could have occurred by disentanglement of the chain, where the unbroken molecules are detached from each other that depend on the length of the chain and entanglement that are directly related to the cutting length and degree of crystallinity [100, 101].

Figure (4.8) shows that the samples with cutting length (7.15 mm) have the highest elastic modulus about 1630 MPa followed by (10mm). This means that the samples with a higher cutting length have a high modulus, on the other hand, the (4.75mm) samples show the highest tensile strength, as shown in Figure (4.9).

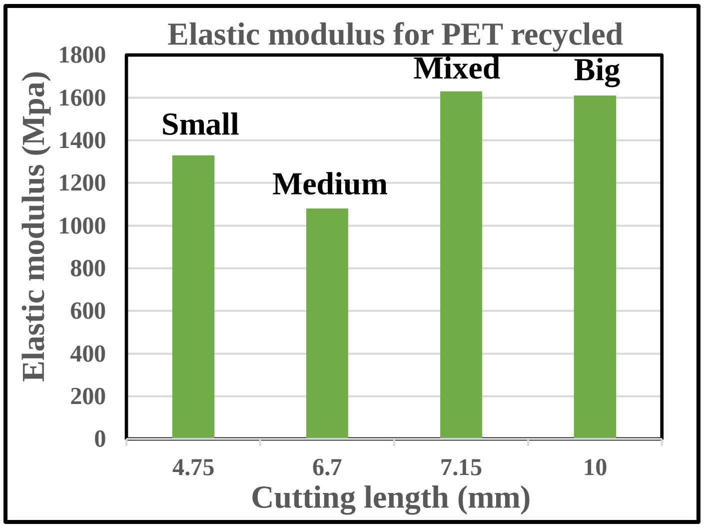


Figure (4.8): The modulus of elasticity of all extruded sheets resulted from PET bottles cut in different pieces for recycling.

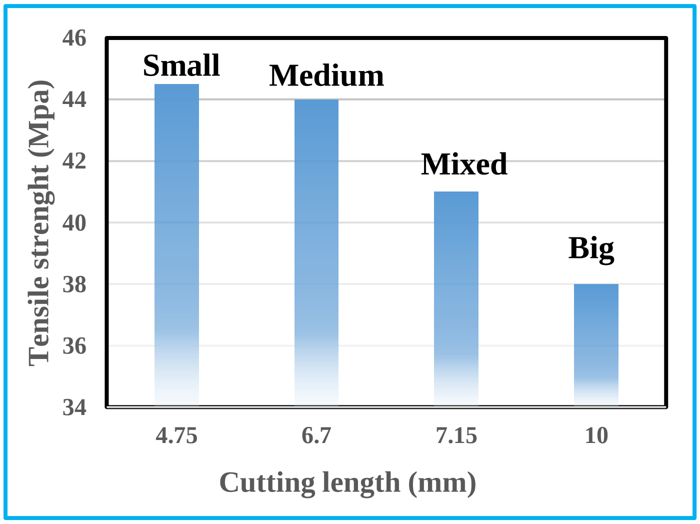


Figure (4.9): The tensile strength of all extruded sheets resulted from PET bottles cut in different pieces for recycling.

Polymer sample tends to show shear cracking deformation in which the chains slip pass over each other under external stresses, in semi-crystalline polymers fracturing accrue by the expansion of the voids or bubbles that are previously formed by gases or inhomogeneity between the fibrils or crystalline zones, as the voids expanded the bridges between them are necked and chains are aligned until the crack propagates and fracture occurs, there for theses defects reduces the mechanical properties of the sample, as shown in Figure (4.10) [100, 101].

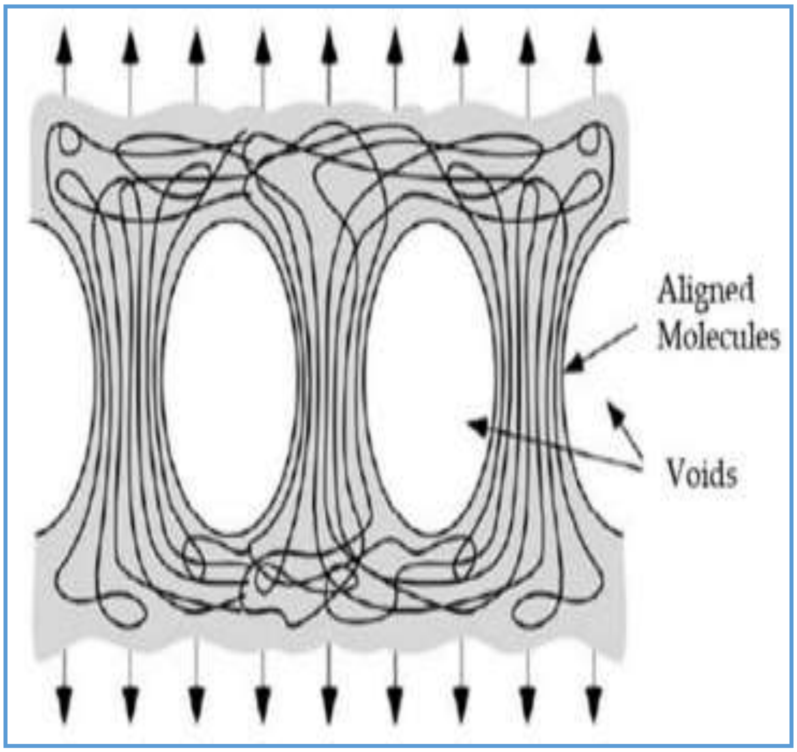


Figure (4.10): Shows voids shape between coordinated molecular chains [102].

When the tensile test result compared with the cutting length result shows that the elongation has an inverse relationship with the cutting length, as shown in Figure (4.11).

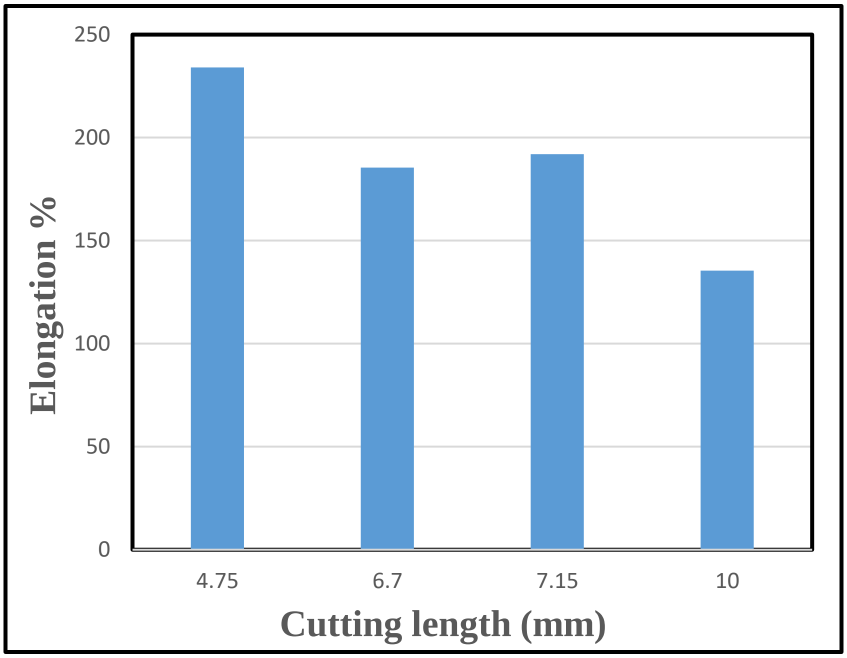


Figure (4.11): The elongation of all extruded sheets resulted from PET bottles cut in different pieces for recycling.

That may indicate the elongation behavior of the samples decreases with increasing the cutting length.

4. 4. 3. Impact test:

Impact test was performed on the samples to investigate the effect of cutting length as well as recycling process on the impact response of samples with different cutting lengths (4.75, 6.7, 7.15, and 10 mm) using the standard method (ASTM D-22885). The result shows that the impact resistance decreased sharply with increasing the cutting length, as shown in Figure (4.12) this decay in impact strength may correlate to the increment of the cutting length due to the presence of voids, cracks, and impurities that may accrue during the processing stage that may act as stress risers that may accelerate the fracture of material as well as the fracturing under lower loads as expected this means the small cutting length the more homogenous and higher impact strength. This may give an indicator of the effect of cutting length on the final product properties.

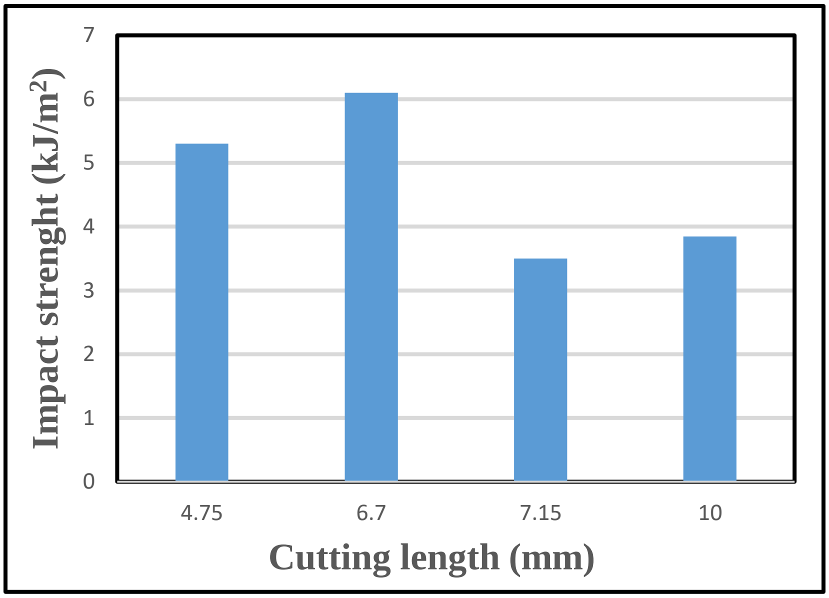


Figure (4.12): The impact strength of all extruded sheets resulted from PET bottles cut in different pieces for recycling by using (Charpy method).

As expected, when the result is compared with the crystallinity values obtained from the DSC test, the result shows the increase in the impact strength with the increment in the crystallinity values.

4. 5. Optical Test:

4. 5. 1. Digital Microscope Inspection:

Figure (4.13) shows the digital microscopy images for all samples after extrusion of PET bottles cut into different pieces for recycling:

(a) Represent the small pieces which have the surface to be smoother, more homogenous, and low bubbles formed during the extrusion process, while,

(b) Represent the medium pieces that have a low surface finish and a moderate level of bubbles while,

(c) Represent the mixed pieces that have non-homogeneous regions which also may lead to fracture under loading due to stress concentration while,

(d) Represent the big pieces that have a high density of bubbles as well as a low surface finish due to the formation of cracks and bad surface finish that leads to fast fracture due to stress localization.

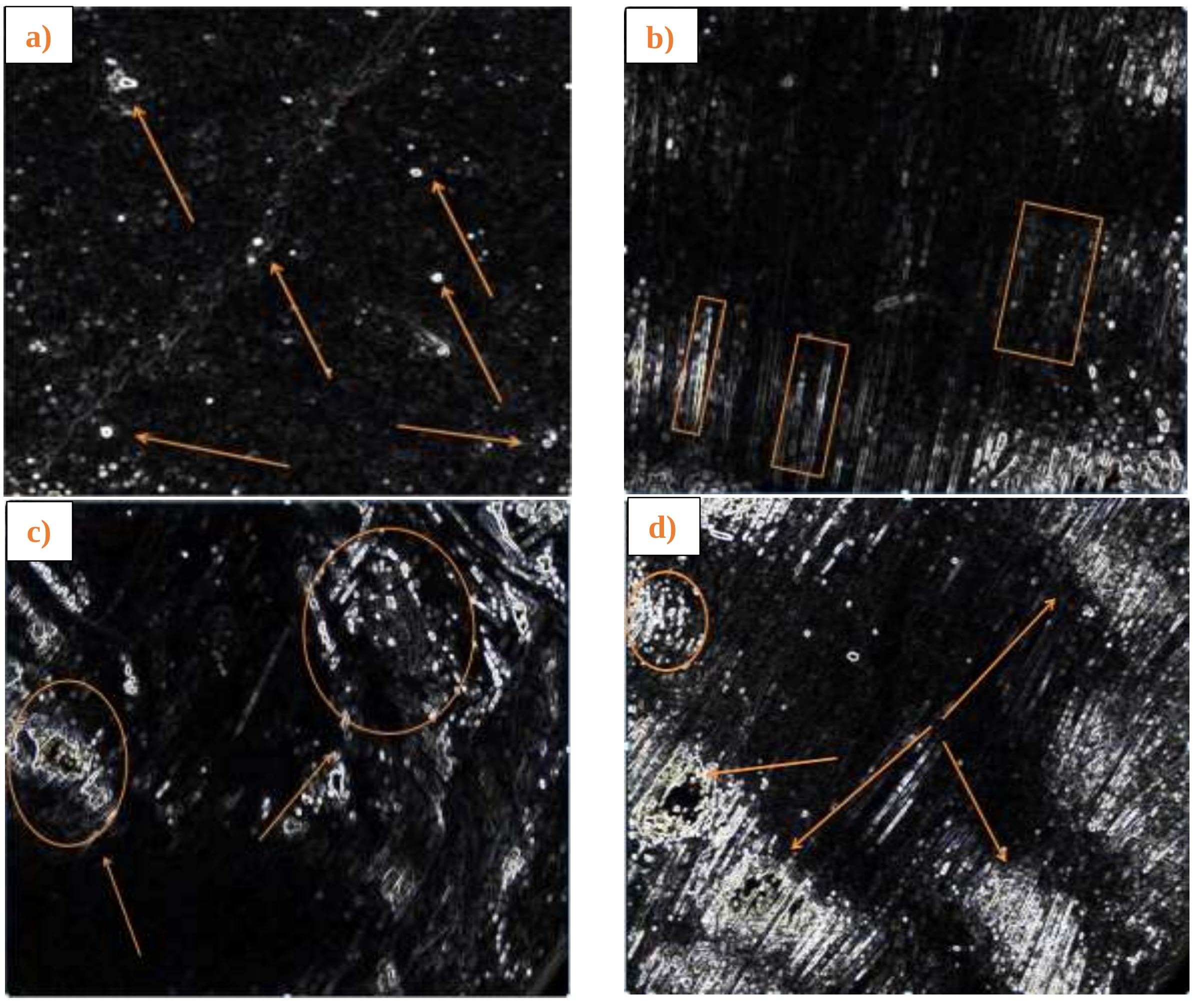


Figure (4.13): The digital microscope results a) small pieces b) Medium pieces c) Mixed pieces d) Big pieces.

4. 6. Rheological Test:

4. 6. 1. Melt Flow Rate (MFR) Results:

Melt flow rate (MFR) in thermoplastics is the mass of a polymer (plastic) that exits the die in 10 minutes at a definite temperature and weight during a specific time. The MFR value is linked to molecular weight (MW) as well as, MFR is also inversely proportional to viscosity which is the material's ability to flow when under pressure. [103, 104]. In this test, the PET samples are filled in the heated barrel then a weight of 2.16 Kg is placed on the top of the device after a predefined heating period to extrude the polymer through the die. During this test, studied the effect of cutting size on the melt flow rate. The result shows that when cutting length is increased and the MFR is increased considerably, as shown in Figure (4.14) which shows the relationship between the cutting length and the melt flow rate (MFR) the test is carried at 255^{o}C using the same loading conditions.

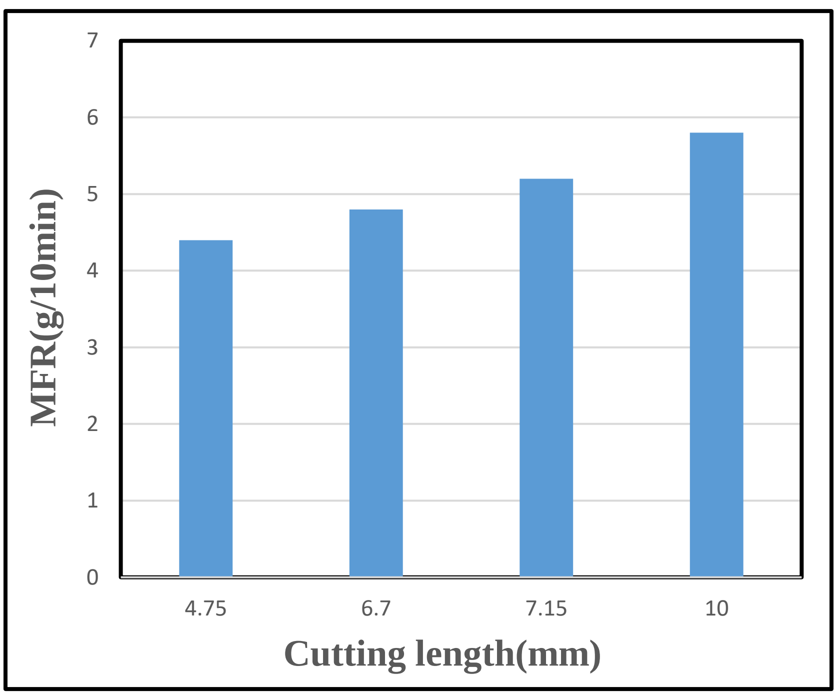


Figure (4.14): The relation between MFR and the cutting length of all extruded sheets resulted from PET bottles cut in different pieces for recycling.

This may give an indicator that the cutting process affects the chains lengths consequently on melt viscosity. An approximated model can be obtained from the relation between the MFR and the degree of the crystallinity obtained from the DSC test, as shown in Figure (4.15).

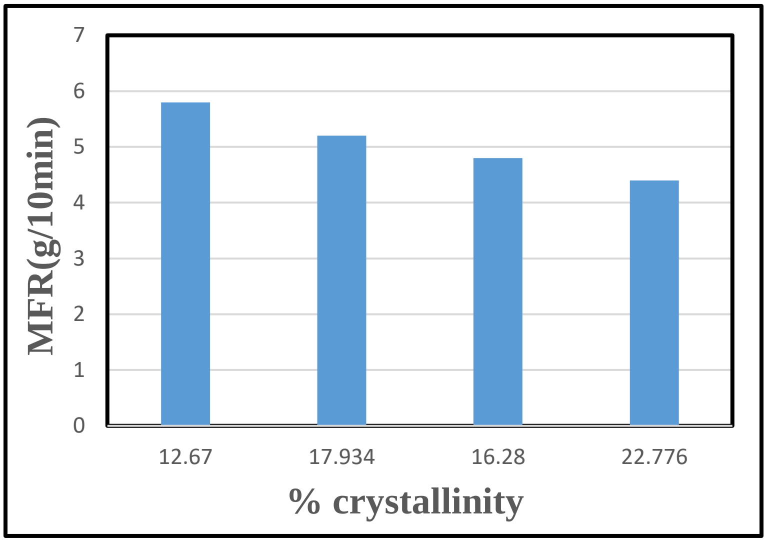


Figure (4.15): The relation between MFR and crystallinity of all extruded sheets resulted from PET bottles cut in different pieces for recycling.

4. 7. Density Test:

Polymer samples usually are unequal in shape especially after the cutting process, so their volumes are difficult to measure accurately. The difference in density may relate to crystallinity, plasticizer, and absorption of solvent, or different reason reasons. In this work, we use ASTM (D-792). According to this test, there are two methods:

- Method A - for analysis of solid polymers (plastics) using water.
- Method B - for analysis of solid polymers using liquids rather than water.

In our case, we use test method A. The obtained results show a decrease in the density with increasing the cutting length, as shown in Figure (4.16).

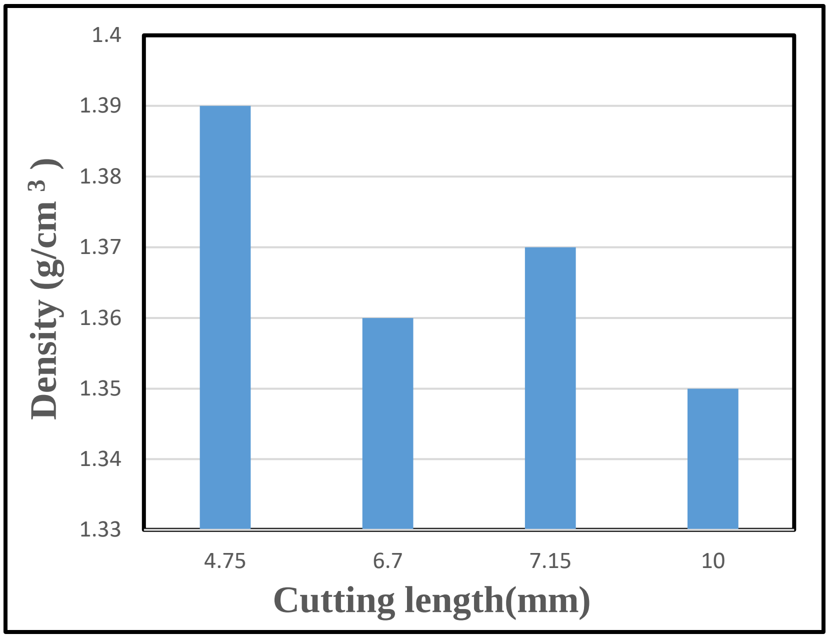


Figure (4.16): The relation between density and the cutting length of all extruded sheets resulted from PET bottles cut in different pieces for recycling.

Percent Crystallinity

We will adopt that a semi-crystalline polymer is a two-segment system and the total volume of the polymer is a summation of the volumes of the amorphous and crystalline regions [105].

$$V = V_a + V_c \qquad (4.1)$$

We will assume m, ma, and mc be the masses of the PET polymer, amorphous regions, and crystalline regions. Let D, Da, and Dc be related to densities. Then the equation is:

$$\frac{m}{D} = \frac{m_a}{D_a} + \frac{m_c}{D_c} \qquad (4.2)$$

Divided by the total mass (m) of the PET polymer.

$$\frac{1}{D} = \frac{P_a}{D_a} + \frac{P_c}{D_c} \qquad \text{(4. 3)}$$

Supposing that there are two phases with a percentage sum of 1.

$$\frac{1}{D} = \frac{1 - P_c}{D_a} + \frac{P_c}{D_c} \qquad \text{(4. 4)}$$

Crystallinity percentage will be:

$$P_c = \frac{\frac{1}{D} - \frac{1}{D_a}}{\frac{1}{D_c} - \frac{1}{D_a}} \qquad \text{(4. 5)}$$

The value D_a (g/cm^3) is the density of 100% amorphous phases, and Dc (g/cm^3) is densities of 100% crystalline, which obtained from special tables in our case polyethylene terephthalate (PET) D_a is equal to 1.336 (g/cm^3) and D_c is equal to 1.514 (g/cm^3). The percent crystallinity is approximately close to the degree of the crystallinity obtained from the DSC device as shown below:

Table (4.3): The crystallinity obtained by density results of all extruded sheets resulted from PET bottles cut in different pieces for recycling.

Cutting length (mm)	%Crystallinity
4.75	20.9855
7.15	15.44
6.7	14.4
10	13.35

Comparing the result of percent crystallinity obtained by different methods shows an average error of 1.5 %, as shown in Figure (4.17).

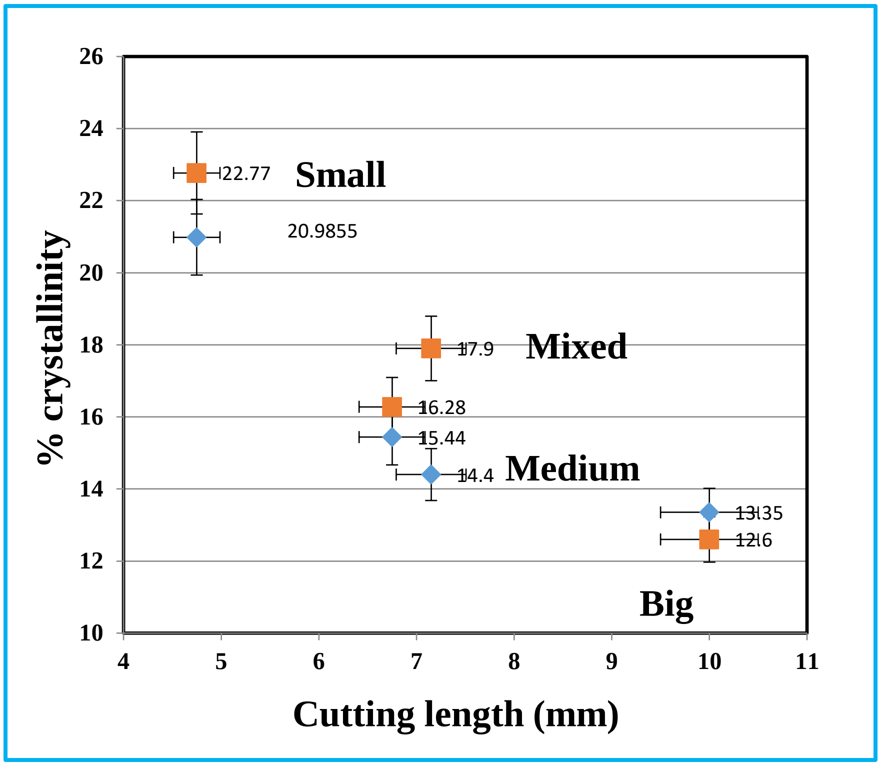


Figure (4.17): The result of percent crystallinity obtained by different methods of all extruded sheets resulted from PET bottles cut in different pieces for recycling.

4. 8. Infrared Fourier Transform Spectrometer (FTIR) Results:

FTIR test was implemented to investigate any change in the chemical structure of the PET samples with different cutting pieces. The result shows there is a small change in the structure of the different samples including C-H phenyl rings at 3055cm^{-1} that appears clearly in mixed dimensions, as shown in Figure (4.18). Other groups include C-H ethyl (2970cm^{-1}), H-C=O(2907 cm^{-1}), C=O ester (1725 cm^{-1}), CO ester, and C-H bending ethyl (731 cm^{-1}) highly appear in the medium dimension samples while others show convergence pattern [106, 107, 108].

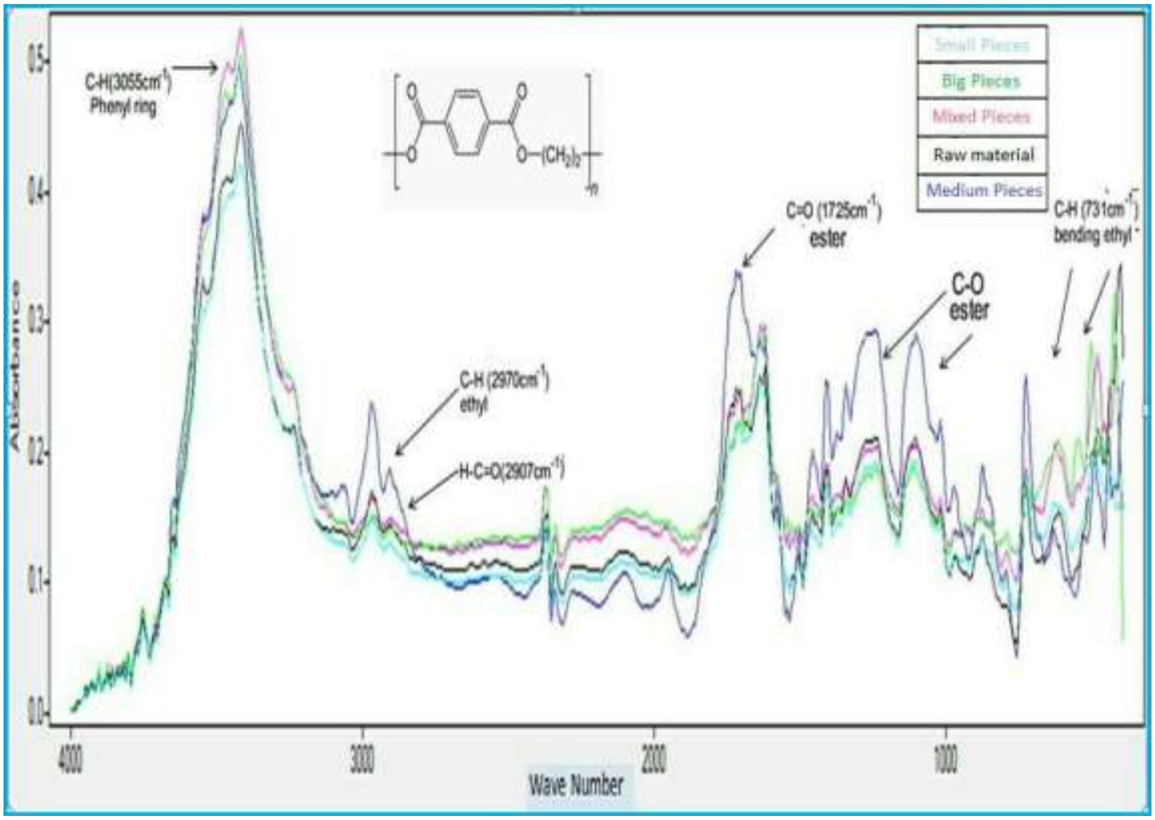


Figure (4.18): Shows the absorbance spectra of all extruded sheets resulting from PET bottles cut in different sizes for recycling with Auto Baseline correction using efficient FTIR software.

Chapter Five

Conclusions

&

Recommendations

5.1. Conclusion:

From the obtained results, it is concluded that:

1. The tensile properties such as elongation and tensile strength decrease with increasing the cutting length but the elastic modulus increases with increasing the cutting length.
2. The impact strength and hardness decrease with increasing the cutting length.
3. Crystallinity and melting temperature increased with decreasing the cutting lengths.
4. It was observed during the extrusion process that the small pieces consumed less electrical energy, except the raw PET (new material).
5. The degradation during the extrusion process starts at temperatures above 220^0 C.
6. The jammed occurs to raw material during the extrusion process when a large quantity was placed in the machine at once time.

5. 2. Recommendation:

Future researches are recommended to study the following aspect below:

1. The effect of the irregular cutting for recycled polyethylene terephthalate (PET) bottles on the molecular weight.
2. The use of the injection process instead of the extrusion process for recycled polyethylene terephthalate (PET) bottles with different cutting lengths.
3. Studying the relation between the cutting method with the piece length and uniformity.
4. The making of blends with other plastic types during the extrusion process to facilities the extrusion process and improve the properties of the product.

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الخلاصه:

يتناول هذا العمل دراسة تأثير التقطيع غير المنتظم لقناني البولي إيثيلين تريفثاليت (PET) المعاد تدويرها على عملية البثق باستخدام آلة بثق ثنائية اللولب وبالتالي تأثيرها على الخواص الميكانيكية للمنتج وكمية استهلاك الطاقة الكهربائية.

تم إجراء عملية البثق لقطع مختلفة من عبوات البولي إيثيلين تريفثاليت (PET) المعاد تدويرها (4.75 ، 6.7 ، 7.15 ، و 10 ملم) ، عند درجات حرارة تتراوح بين (200-205 درجة مئوية) بسرعة 50 دورة في الدقيقة. أظهرت النتائج أن طول القطع له تأثير مباشر على التبلور مما يؤثر على الخواص الميكانيكية والحرارية مثل الاستطالة ومقاومة الشد حيث تقل مقاومة الشد والاستطالة مع زيادة طول القطع ولكن معامل المرونة يزداد مع زيادة طول القطع. .

أظهرت نتائج اختبارات الصدم والصلادة أن مقاومة الصدمة تقل بنسبة (33.156٪) مع زيادة طول القطع كما تقل الصلابة بنسبة (23.88٪) مع زيادة طول القطع. لوحظ خلال عملية البثق أن القطع الصغيرة تستهلك طاقة كهربائية أقل بنسبة (52.5٪) ، مقارنة بالقطع الكبيرة من عبوات البولي إيثيلين تريفثاليت (PET) المعاد تدويرها ، باستثناء المادة الخام من (PET). أظهرت نتائج الفحص بالمجهر الرقمي أن كمية الفقاعات وكذلك تشطيب السطح السيئ يزداد مع زيادة طول القطع. تختبر نتائج قياس المسعر التفاضلي (DSC) أن درجة حرارة الانصهار والتبلور ودرجة حرارة التزجج تنخفض مع زيادة طول القطع. أظهرت نتائج اختبار معدل التدفق (MFR) زيادة معدل التدفق المنصهر بنسبة (19.696٪) مع زيادة طول القطع. تظهر نتائج اختبارات الكثافة أن الكثافة تتناقص مع زيادة طول القطع.

.

جمهورية العراق
وزارة التعليم العالي والبحث العلمي
جامعة بابل
كلية هندسة المواد
قسم هندسة البوليمر

دراسة التقطيع غير المنتظم للبولي إيثيلين تريفثاليت PET المعاد على الخصائص الميكانيكية والطاقة خلال عملية البثق

رسالة

مقدمة الى كلية هندسة المواد/ جامعة بابل كجزء من متطلبات نيل درجة الماجستير في هندسة المواد/ البوليمر

اعدت من قبل:

حسين علي عدنان عمران

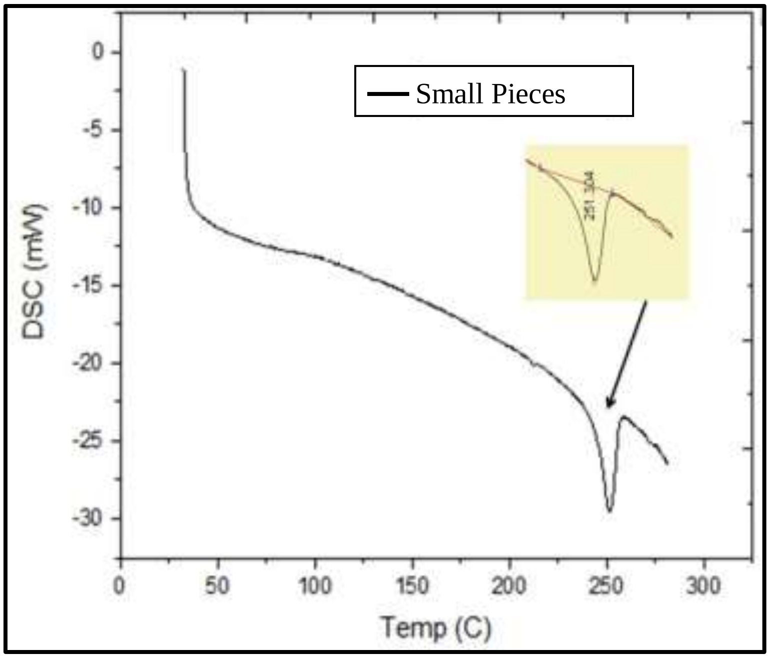
إشراف:

أ. د. علي عبد الامير الزبيدي

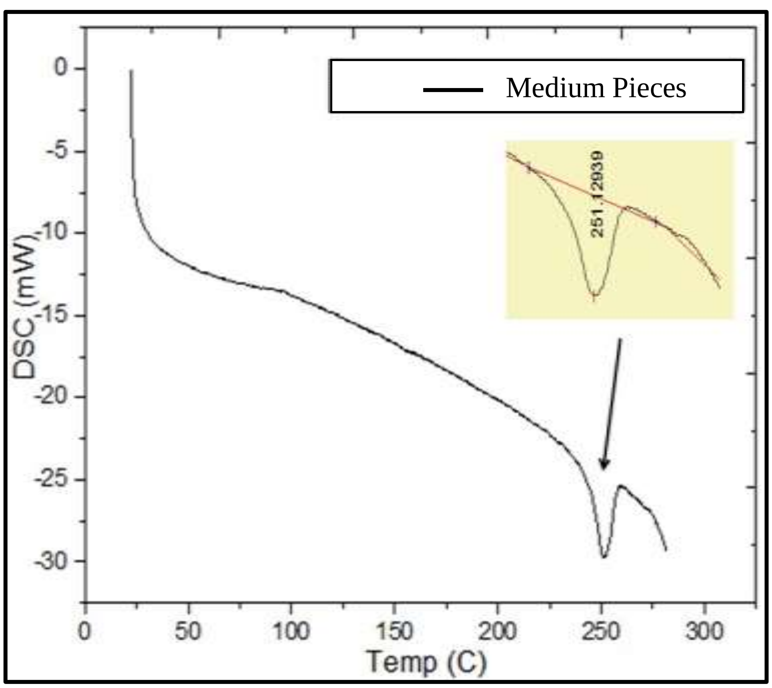
1442 هـ **2021م**

Appendix (A)

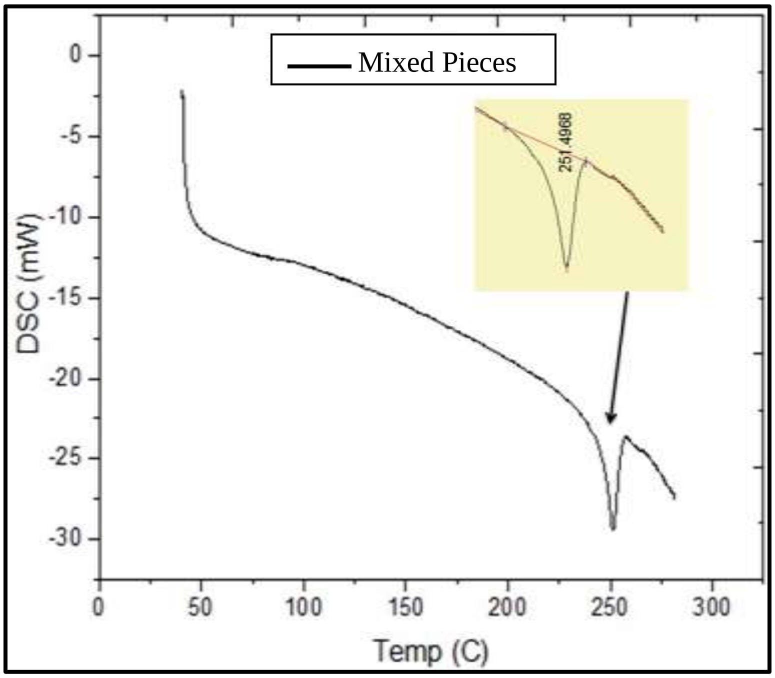
The DSC results anaylasis:



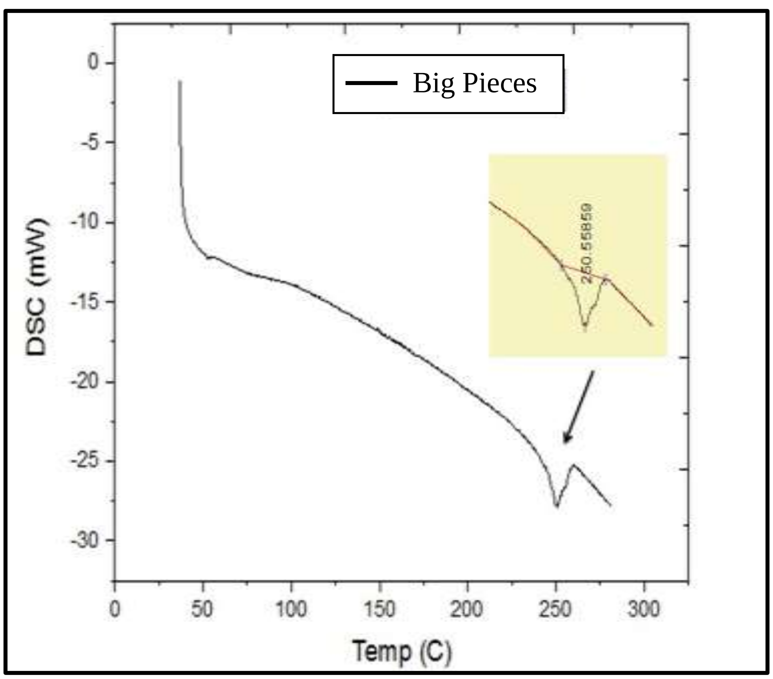
The DSC results analysis for small pieces of recycled PET bottles.



The DSC results analysis for medium pieces of recycled PET bottles.



The DSC results analysis for mixed pieces of recycled PET bottles.



The DSC results analysis for big pieces of recycled PET bottles.