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Morphological and mechanical characterization of Thermoplastic polymers and their Use in packaging Applications

A Graduation Project

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University of Babylon in Partial Fulfillment of the Requirements for the
Bachelor's Degree of Science in Materials Engineering/ Polymer**

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بِسْمِ اللَّهِ الرَّحْمَنِ الرَّحِيمِ

((وَمِنَ النَّاسِ وَالْأَنْعَامِ مُخْتَلِفٌ أَلْوَنُهُ كَذَلِكَ ۖ إِنَّمَا

يَخْشَى اللَّهَ مِنْ عِبَادِهِ الْعُلَمَاءُ ۗ وَإِنَّ اللَّهَ عَزِيزٌ غَفُورٌ))

صَدَقَ اللَّهُ الْعَلِيِّ الْعَظِيمِ

سورة فاطر: الآية ٢٨

Dedication

To The One Who Worthy Praise and Thanks.....

The one who Revives the skies by His Throne and Decorates the Universe by his Mention.....

Allah

To our great Prophet Mohammad and his Relatives (Peace and Blessings of Allah be Upon Him and Them)

To the one who embraced me like my mother.....

The one who is my light in life.....

My dear father

To everyone who taught us a letter in this mortal world

To everyone who thinks and searches for the advancement of science everywhere, we dedicate this humble effort

To all the Teaching staff .. for helping and providing advice and rich information

We also thank everyone who helped us from near or far, even with a good word or prayer

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Also, we would like to thank the Department of Polymer Engineering and Petrochemical Industries and all members of the laboratories

Finally, we would like to thank Our families ; Our friends and all those who helped us accomplish the current work.

Supervisor Certification

certify that this project entitled **(Morphological and mechanical characterization of Thermoplastic polymers and their Use in packaging Applications)**

was prepared

By: **Shafag Haref Theban and Nabaa Ahmed Hussein**

under our supervision at Babylon University / Collage of Material Engineering / Department of Polymers Engineering & Petrochemical Industries , in partial Fulfillment of requirements for the Award Bachelor Degree of Science in Material Engineering , Polymer& Petrochemical Industries..

We Recommend that this project be forwarded for examinations in accordance with the regulation of the University of Babylon.

Supervisor: Assist. Prof. Dr. Ali Salah Hassan

ABSTRACT

LDPE is a major type of thermoplastic that is why it is used in many industries, many plastic displays and toys. The most common use is plastic bags and cans made from it for packaging juices and milk.

In this work, a polymeric compound was studied and prepared from a polymer LDPE, and a filler material of ZnO nanoparticles was added for uses in packaging applications.

Different samples of the compound were prepared with different inoculation ratios (45g LDPE +5g ZnO) and (40g LDPE+10g ZnO). At three different temperatures for each percentage were these temperatures (180,200,220) respectively, preparing these compounds by means of a twin-screw extrusion machine and homogeneous fish, and studying their mechanical properties from (tensile, hardness, shock) tests, where the results showed a decrease in the tensile values and the modulus of elasticity due to the movement of the chains and an increase in the flexibility of the material and an increase in the shock values due to the strength of the compound and a decrease in the values of hardness due to decrease in stiffness and increase in toughness.

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chapter One

1.1 Polymer Science: History

Polymers were the product of post war renaissance in chemical industry driven by the promise of inexpensive petroleum derived feed- stocks, The fifties and sixties saw the introduction of many polymers that changed the face of human civilization. From early curiosities polymers became an indispensable part of our daily living and so ubiquitous that we no longer realize how addicted we are to polymer materials. No new polymers has entered the market since the early nineties. The last ones were Poly(propylene terephthalate) by DuPont (PTT), Poly(ethylene Naphthalate) by Teijin (PEN) and Nature Works Poly (Lactic Acid)s by Cargill.

Several new polymers developed in the last fifteen years have been abandoned after market introductions. Example, Carilon by Shell, Questra (syndiotactic polystyrene), PCHE (hydrogenated polystyrene), Index (ethylene -styrene copolymers by Dow). The rate of growth of markets of the new polymers introduced after the nineties have been painfully slow.

Global Issues That Impact The Polymer Industry Energy consumption, driven by demands of emerging economies, is increasing exponentially Greater than 85 % of global energy demand is met by non renewable fossil fuels It is generally recognized that fossil fuel production has reached its peak and the era of "cheap oil" is over. Price of oil will be demand not supply driven Increase in oil prices will spiral into increase in the cost of feedstocks and polymer costs which cannot be passed on to consumers Polymers consume only 7 % of the fossil fuel; yet human addiction to oil as an energy resource will take precedence, making the cost of feedstock for polymers derived from

oil unsustainable Difficulties in creating value and high entry barriers for new product introductions [1].

1.2. Introduction of polymer

The structure of polymers consists of a great number of repeating units and high molecular mass compounds. polymer materials are very important in our daily life that entity of human life depends on these polymers. Polymer materials have wide range of properties that can be used in different applications. There has been increasing public concern over the harmful effects of petroleum-based polymer packaging materials specially polyolefins in the environment after the usage. These petroleum – based polymers create significant amount of waste after the usage and that generates the critical environmental issues. Recycling, recovery and disposal of plastic waste are some of the options available. These methods have certain disadvantages and not hundred percent practical. An important alternative to minimize the polymer waste is the introduction of biodegradable polymers, which can be degraded through the action of naturally occurring microorganisms [2].

There is a considerable interest in replacing some or all of the synthetic plastics by natural or biodegradable materials in many applications. Since the food industry uses a lot of plastics, even a small reduction in the amount of materials used for each package would result in a significant polymer reduction, and may improve solid waste problems. It is clear that the use of biodegradable polymers for packaging offers an alternative and partial solution to the problem of gathering of solid waste composed of synthetic inert polymers [3].

1.3. Properties of Polymers

A polymer's properties fall into two wide categories:

1. Material properties relate primarily to the polymer's nature.
2. Specimen properties are mainly implications of the size, shape and design of the completed samples prepared from that polymer as well as the method used to prepare them.

A material properties of the polymers can be roughly divided into two general types:

- a) Fundamental properties such as a quantity of Vander Waals, cohesive energy, heat capacity, molar refraction and molar dielectric polarization are directly linked to some very fundamental physical factors.
- b) Derived properties such as the temperature of the glass transition, density, solubility parameter and modulus, are more complicated manifestations of the basic characteristics and can be expressed in terms of their combinations [4].

Based on their thermal processing conduct, all polymers can be split into three significant groups: thermoplastics and thermosets.

1.3.1. Thermoplastics Polymer

These are linear or slightly branched long-chain molecules that can be softened continuously when heated and hardened when cooled. These polymers have intermolecular forces between elastomers and fibers of intermediate attraction. Polyethylene, polystyrene, polyvinyl, etc. are some common thermoplastics [5]. Thermoplastics are rarely processed by themselves. To generate pellets, powders or flakes for use in a subsequent

processing processes, they are often mixed or compounded with other materials. The compounds may contain fillers, dyes, other polymers, flame retardants, reinforcements, stabilizers, and various handling aids. Simply refer to the compounded polymers as plastics. Polymer processing is concerned with converting raw polymeric materials into "value added" finished products and may occasionally require chemical reactions in relation to compounding and shaping. Several processes are used for thermoplastics processing, the most common being extrusion (the largest in volume), injection molding, extrusion and injection blow molding, film blowing, film and sheet casting, calendaring, fiber spinning, compression molding and rotational molding. A popular feature of these procedures is that the polymer is processed and it flows [6].



Figure (1.1): Structure of thermoplastic [7].

1.3.2. Thermoset Polymer

are polymers with a combination of mechanical, thermal, electrical and chemical resistance properties that allow them to compete with metals, ceramics and thermoplastics. Almost all thermosetting compounds lack the strength and hardness of metals, and contain fibrous particles or reinforcements. Fillers, such as calcium carbonate, glass chips, and wood flour are added to reduce cost and increase the hardness of the treated product. Fibers such as glass, carbon, and polyaramid increase their strength,

toughness, and cost. The amount of composite fillings varies between 45 and 75%.

Compared with metals, thermosite has corrosion resistance, is lighter in weight, has better insulating properties, and can be processed at lower pressures and temperatures. The flow properties of uncured thermoplastics can be used to form large and complex shapes in a single mould , allowing for parts integration and eliminating processing costs. The advantages of metallic materials are high temperature performance, thermal and electrical conductivity, isotropic properties, ductility, and dimensional control.

Compared with ceramic materials, refractory materials offer lighter weight, better toughness, and easier handling. Ceramic materials provide improved high temperature Performance, excellent chemical resistance and toughness. For many applications, both thermoplastic and engineering resins are suitable candidates. The choice of the preferred material depends on a specific set of required properties and processing characteristics. Therma+1 composites offer advantages in terms of reduced creep and improved solvent/crack resistance to stress. 3D polymer networks in heat aggregates also improve machinability, provide low rates of gas permeability, low mold shrinkage, and high temperature performance. The low initial viscosity of thermosetting compounds allows the use of large amounts of fillers or fibers [8].

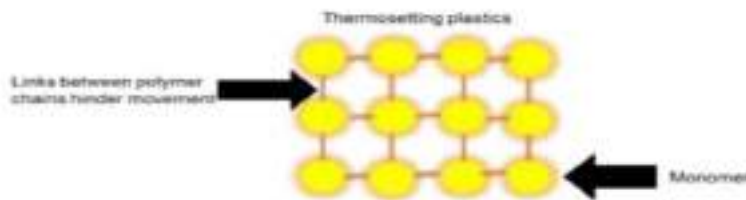


Figure (1.2): The degree of cross linking in thermoset polymers [9].

1.3.3. Elastomer Polymers

Elastomeric polymers have continuous chains but are less branched and are flexible even at low temperatures, so these materials have a low modulus of elasticity. All types of rubber belong to the label (elastomer), and elastomers have special characteristics such as their ability to stretch, contract and elongate by pressure. Between plastic and elastomer [10], the polymeric chains are free during local movement. Elastomeric polymers are divided into natural, such as natural rubber, and synthetic, such as Krylonitrile-Butadiene rubber, which are produced by various polymerization processes.

Rubber materials have been used since ancient times, when the Native Americans turned rubber after extracting it from its trees into rubber balls that they used in their games. In 1839 AD, the process of vulcanizing rubber was discovered by scientists Hancock from England and Goodyear from America, and this process made rubber a moldable material, so they used it in the manufacture of old car tires and was also used to cover fabrics and clothes resistant to water and rain, and in 1888 AD the scientist invented Dunlop pneumatic car tire and discovered that carbon black is a hardening substance when added to rubber with organic materials that accelerate the vulcanization process and as a result the rubber industry has developed dramatically. Rubber trees were grown in Malaysia and Indonesia more than they were grown in South America, but the spread of synthetic rubber production began in the period between World War I and II, especially in Germany and the United States of America. During this period of time, thiokol rubber and rubber were produced Neoprene [11,12].

Any material that suffers deformation in its shape as a result of exposure to an external force, even if it is small, and that regains its original shape strongly and quickly after the removal of the force applied to it is expressed. by the word “rubber”. Rubber is classified. In general to :

1 - Natural rubber

2- Synthetic rubber

Elastomers are defined by the American Society for Testing Materials (ASTM) as polymeric materials that can stretch at least twice their original length (within the elastic limit) at room temperature, and after the stress is removed they return to their original length. The elastomers are distinguished by two characteristics [13,14]:

1-The glass transition temperature (T) has a lower temperature than its use.

2-Its molecules are closely related to each other.



Figure (1.3): Structure of elastomers [15].

Chapter Tow

2.1. Polyethylene(PE)

The experimental development and testing of polyethylene polymers in the 1930s for use as a high frequency insulation for radar cables during World War II gave impetus to its commercial production. Polyethylene is available in a range of flexibilities, depending on the production process. High density polyethylene (HDPE) is the most rigid of the three basic types of PE resins (HDPE, low density polyethylene (LDPE), and linear low density polyethylene (LLDPE)).

HDPE can be formed by a wide variety of thermoplastic processing methods and is particularly useful where moisture resistance and low cost are required. Polyethylene is limited by low end use temperature characteristics.

There are three basic manufacturing processes for making HDPE. The slurry particle reactor process (the most widely used method), the gas phase process, and the new metallocene catalyst technology. This last process also may be used with standard catalysts to combine the advantages offered by both polyolefin polymerization catalysts. A new class of catalysts for polyolefins based on nickel and palladium reportedly can produce a very broad range of molecular weights and branching. Ability to incorporate a wide variety of polar and nonpolar comonomers allows the production of EVA and EMA copolymers in low pressure processes. Combinations of reactors also are used to make HDPE. The tandem reactors produce what are called bimodal molecular weight HDPE.

The slurry process and gas phase process can also be used to make HDPE.

Unlike LDPE, LLDPE lacks a long chain branching. Another difference is that the ethylene is copolymerized with butene, octane or hexene comonomers in the reactor. LLDPE has a narrower molecular weight distribution than LDPE. A final distinction is that LLDPE exits the reactor in a powder form and the granules are compounded with additives in an extruder and pelletized.

Blow molding uses the largest amount of HDPE. About 35% is used to make blow molded products. Extruded products consume about 30%, and injection molding accounts for about 20%.

High molecular weight (HMW) and HDPE resins have high strength; they are used in packaging films, sheets, pipes, and large blow and rotation molding items.

Low density polyethylene (LDPE) is the second largest PE capacity in the US. Extrusion is the dominant process used with LDPE resins. Extruded products, principally films, account for 75% of LDPE sales. LDPE remains the dominant PE resin for extrusion coating and for food packaging films. LDPE is also used for injection molding, blow molding, and rotational molding.

High molecular weight (HMW) LDPE film grade resins produce high gloss, high clarity film that exhibit good toughness and heat sealability.

High pressure, high temperature polymerization reactors, both tubular and autoclaves, are used to make LDPE. The molecular structure of LDPE is characterized by long side branches that give the resins their combination of flexibility, clarity, and processability [8].

General Properties of Generic Unfilled HDPE and LDPE Polymers		
	HDPE	LDPE
Specific gravity	0.94	0.91
Tensile modulus @ 73 °F (Mpsi)	0.2	0.05
Tensile strength @ yield (Kpsi)	3.75	2.25
Notch Izod impact @ 73 °F (ft-lb/in)	No break	No break
Thermal limits service temp. (°F)	158–176	140–167
Shrinkage (%)	1.1–1.4	1.1–1.4
Vicat point (°F)	255	–
T _g (°F)	–150	–150
T _m (°F)	257–275	212–230
Process temp. (°F)	400–535	360–530
Mold temp. (°F)	50–140	50–140
Drying temp. (°F)	150	150
Drying time (h)	3.0	3.0

Figure (2.1): shows the properties of HDPE and LDPE

2.2. Introduction of LDPE

The recycling and reuse of plastic waste films created from greenhouses is a global environmental problem. Agricultural greenhouse cover films and mulch films are typically made from low-density polyethylene (LDPE) [16–17]. Therefore, every year a large amount of LDPE film waste is found in agricultural waste streams. Recycling LDPE films from agriculture is a promising solution to reduce the amount of material discarded. However, since recycled LDPE exhibits moderate mechanical properties, which are further affected by an aging of the product, it is not really interesting for specific applications. New outlets for recycled LDPE could be developed if their low mechanical properties are improved by adding other materials. In

fact, recycled LDPE has been widely used with virgin polymers to improve its mechanical properties. This is an effective way to reuse recycled LDPE. The addition of ethylene-vinyl acetate (EVA) to LDPE has been used commercially to increase resistance to environmental stress cracking, toughness, and resistance to film tearing [18–19].

LDPE is a thermoplastic, produced by a free radical polymerization method under pressure from 150–350 MPa and a temperature from 80–300 °C. The key characteristics of LDPE are low-temperature impact toughness, low-temperature impact resistance, good resistance to chemicals, and good creep resistance [20,21]. However, LDPE has some disadvantages such as low compatibility with additives, and high flammability.

2.3. Low-density polyethylene

LDPE falls within the thermoplastic polymer classification and is produced under high pressure (82 - 276 MPa) and high temperature (405- 605 K) with a free radical initiator (such as peroxides and oxygen). The LDPE contains some long chain branches (LCB) that could be as long as the backbones of the chain and short chain branches (SCB) [22]. The structure of LDPE shown in Figure (2.1)

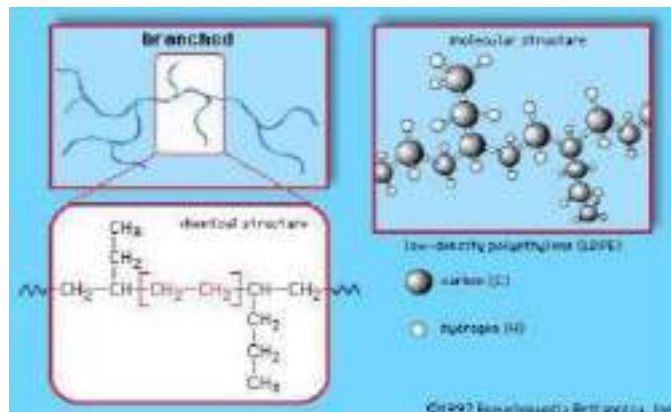


Figure (2.1) Represent structures of low-density Polyethylene [23]

Low density polyethylene (LDPE) is so called because these polymers contain substantial branch quantities that hinder the process of crystallization, leading in comparatively low densities. The branches are primarily ethyl and butyl groups with some length chain branches. The ethyl and butyl branches are frequently grouped together, separated by long runs of unbranched backbone, due to the nature of the elevated pressure polymerization method by which low-density polyethylene is manufactured. Long chain branches happen along the length of the primary chain at random intervals. In turn, the long chain branches can be branched. The various branches properties of polyethylene molecules with low-density inhibit their ability to crystallize, decreasing resin density relative to polyethylene with elevated density. Low-density polyethylene resins typically have densities of about 0.90–0.94 g / cm³ [24].

2.4. Production Processes for LDPE

The free-radical Polymerization of Ethylene at high pressures to LDPE can be performed by two processes, either the tube reactor process or the autoclave process.

Production Processes of LDPE
Autoclave (1300-2200 bar/155-320°C/Conversion 15-20%)
or
Tube (O₂ or O₂+Peroxides) (2200-3000 bar/130-330°C/Conversion 15-30%)

A tube reactor (figure 2) consists of a high number of high pressure tubes; the total length of these tubes can be up to 2000 m. Repeated dosage of the initiator (organic peroxides or Oxygene) and fresh Ethylene will be performed at various segments of the tubes.

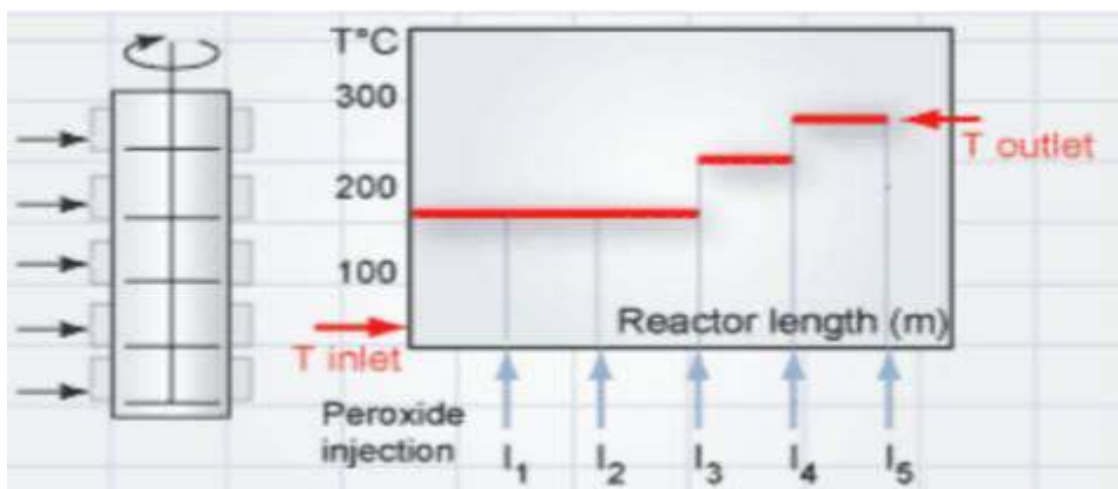
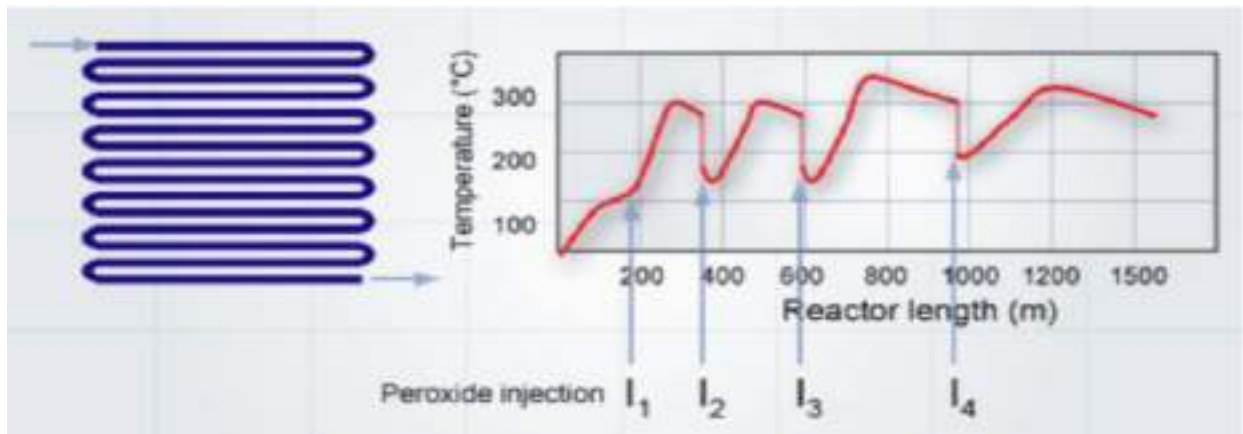


Figure (2.2) Tube Reactor [23]

The size of an autoclave reactor (figure 5) is up to 2000 l. Multi-chamber types are often used. Repeated dosage of initiator and Ethylene will be performed in order to achieve a certain temperature profile in the reactor.

Both processes are bulk processes. The main criterion for the selection of a suitable initiator will be the polymerization temperature. In addition the physical properties of the LDPE, like degree of chain branching and therefore the density and crystalline structure are also determined by the polymerization temperature. The temperature profile ranges from approx. 130° C up to 330° C. At higher temperatures there is a risk of Ethylene decomposition; lower

temperatures than 130 ° C can be not maintained due to the initiator activity and the low reactivity of the Ethylene. The typical reactor holding time of the Ethylene is about 1 min., the degree of conversion approx. 10 - 35 %. Due to the high viscosity of the LDPE one cannot obtain higher degrees of conversion [24].

2.5. The applications of low-density polyethylene

1. It is widely used for manufacture of different containers, dispensing bottles, washing bottles, tubing, computer component, plastic bags, and various molded laboratory equipment.
2. Trays and containers of general use.
3. Work surfaces resistant to corrosion.
4. Parts requiring welding and machining.
5. Wraps of plastic
6. To produce agricultural covers [25].

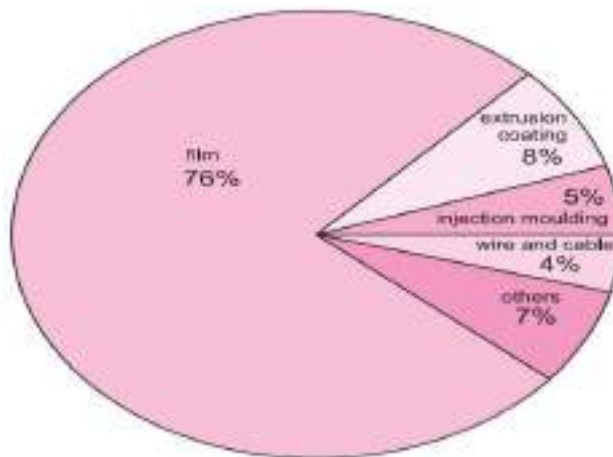


Figure (2.3) Uses of low density polyethylene [25].

2.6. Preparation and Application of LDPE/ZnO Nanocomposites

1- for extending shelf life of fresh strawberries strawberries have a very short post-harvest life mostly due to their relatively high water content, intense metabolic activity and susceptibility to microbial rot. Antimicrobial low-density polyethylene nanocomposite films containing znO nanoparticles at different mass fractions were prepared by melt mixing and followed by compression moulding using a hot press machine. Fresh strawberries were packed in nanocomposite films and stored at 4 °c. Their microbial stability, ascorbic acid content and titratable acidity were evaluated after 0, 4, 8, 12 and 16 days of storage. Microbial growth rate was significantly reduced up to 16 days as a result of the use of nanocomposite packaging material containing znO nanoparticles. By increasing the znO nanoparticle mass fraction to 5%, the antimicrobial activity of the film increased. All packages containing the znO nanoparticles kept the microbial load of fresh strawberries below the level that affects shelf life (5 log cfu/g) up to 16 days. The lowest degradation of ascorbic acid content (6.55 mg per 100 g), and loss of acidity (0.68%) were observed in packages containing 3% of znO nanoparticles with 10% polyethylene-grafted maleic anhydride [26].

2- UV-protected transparent composite films made from a dispersion of virgin zinc oxide nanoparticles in low-density polyethylene

This work proposes an approach to fabricate a flexible ultraviolet (UV) transparent shielding film by casting method, which uniformly disperses virgin zinc oxide nanoparticles (NPs) in low-density polyethylene (LDPE). Critical conditions for film fabrication, such as casting temperature, LDPE

concentration in solution, melting time, NP concentration, and post-hot press cooling processes, are systematically studied. It is found that the casting temperature should be close to the melting temperature of LDPE, that is, 115 °C, so that the formation of a transparent film without cracks can be ensured. NP agglomerates are suppressed if the polymer concentration is controlled below 6%. For good dispersion of NPs, the LDPE should be swelled or not crosslinked enough in solution (approximately 200 hours dissolution time), then NP agglomerations can be reduced due to diffusion of NPs in the polymer gel (322 hours dissolution time) . When the NPs are well dispersed in the LDPE film, the film can completely shield UV light while allowing high transmission of visible light. As the concentration of NPs in the film increases from 4 to 6%, the film transition decreases, the tensile strength increases, and the tensile failure strain decreases [27].

2.7. The Properties of low density polyethylene

- 1- LDPE has a high degree of short and long chain branching.
- 2- Density ranges from (0.910 -0.940 g/cm³).
- 3- At room temperatures, it is not reactive.
- 4- It can withstand temperatures of 80 °C continuously and 95 °C for a short time.
- 5- It is quite flexible, and tough but breakable.
- 6- Lower tensile strength and higher resilience.
- 7- Good resistance to aldehydes, ketones and vegetable oils (minor attack).

8- Poor resistance and not recommended to use with halogenated hydrocarbons [25].

9- Low density polyethylene (LDPE) has a great flexibility and resilience [28].

2.8. Properties usually consider disadvantages

1- Applications requiring high rigidity and high tensile strength may not be suitable for LDPE.

2- Low strength to oxidizing agents, aliphatic solvents, aromatic solvents, polar liquids, chlorinated solvents, low softening point, low scratch resistance, low permeability of gas and moisture.

3- Relatively lower resistance to stress crack compared to other polyethylene types.

4- Under shear conditions, LDPE undergoes thermal degradation and chain extension at high temperatures [29].

2.9. Extrusion Process

Extrusion molding: It is a method that depends on continuous annealing of polymeric materials in the annealing system, pushing them forward and then extruding through a channel called Die. (The screw) and the thrusting system and the extrusion head (Istanba). The idea of extrusion started in 1879 by the inventor: M. Graye in England/UK. Extrusion is basically the transformation of the raw material into a specified shape product by forcing it through a die. Many different materials can be formed through an extrusion process, such as metals, ceramics, clays, foodstuffs, and plastics either in the

molten or solid state. Extrusion is a high volume manufacturing process in which raw plastic material is melted and formed in to continuous profile [30].

Extrusion process for polymers Extrusion is a high-productivity manufacturing (forming) process, and this process is usually continuous to produce large lengths, the polymeric material is molten by the influence of temperatures applied to it through heaters and is extruded through a die to obtain the desired shape, and the method of work is summarized by the movement of the snail The screw is constantly inside the cylinder, as it derives its circular motion from an electric motor by means of a belt that is attached to the gearbox, to control the speed through several gears of various sizes sitting on the shaft and the bearing chair, and the “helix” is responsible for mixing, homogenizing and pushing the material The molten into the mold, for the annealing system, is the same as in the injection process except for the presence of additional fans to regulate the heating process. At the end of the cylinder there is a filter, and in the cylinder there is a relief valve to expel excess gases and vapors. The important parameters (variables) in the extrusion machine are temperatures and speed, when The exit of the product falls into a basin of water for cooling, and there are other ways to cool the product, for example: air fans, the extrusion process is easy, but difficult for thermally hard materials. The extruded material takes the shape of the mold according to the section to be produced. It is the ideal way to manufacture shapes of standard sizes such as bars, tubes, strips and plates, and it is also suitable for thermally

hard polymeric materials (Thermosets), such as manufacturing (pipe,films) [31].

The drive system in extrusion machines must meet the following :

- wide limits of rotational speed change = 10: 1 with high efficiency
- good stability of the rotating speed of the screw when changing loads from (0-100),
- stability or little change of circular torque in Indication of circular speed
- ease of maintenance and repair and low costs.

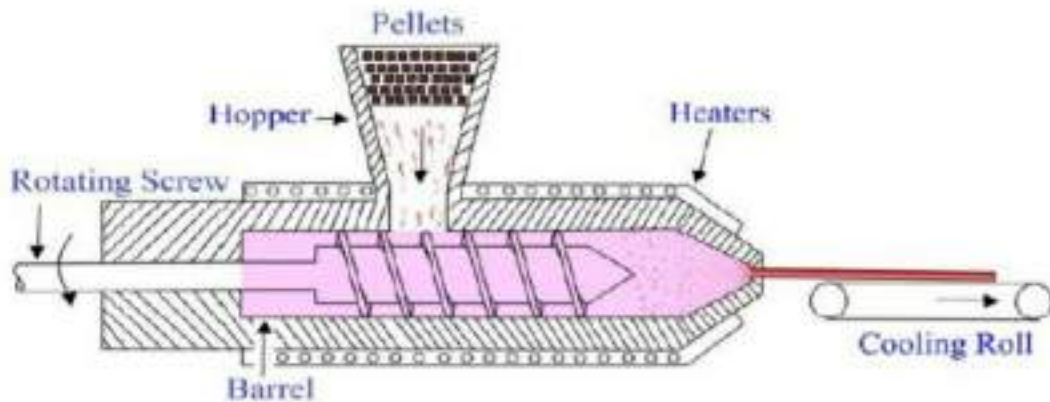


Figure (2.4) : showing a section of a single screw extruder for thermoplastic polymer materials.[31]

2.10. Twin Screw Extruder

A twin screw extruder is a machine with two screws which can be categorized by the intermeshing structure, the rotation direction in to (co-rotating and counter- rotating), by the functions of screws designed to perform, or by screw speed. Two main areas of application for twin screw extruders are profile extrusion of thermally sensitive materials (e.g. PVC), and specifically polymer processing operations, such as compounding, chemical reactions [30].

The most important process parameters are melt temperature and pressure.

Other parameters related to the extruder are : ‘-

1 Screw Speed.

2 Motor Load.

3 Barrel Temperatures.

4 Die Temperatures.

5 Cooling Rate [30].

There are many parameters affecting the resulting structure, such as annealing temperature, polymer molecular weight, feed rate, screw configuration, and screw speed. All of them direct or indirectly affect the shearing intensity and the residence time during processing, and consequently the structure of the obtained product [30].

The advantage of twin screw extruder is low energy consumption, greater tolerance to difficult material to process and flexibility for producing small quantities of multiple items [32].

Chapter Three

3.1 The materials used in the preparation

3.1.1. Low Density Polyethylene

The first grade polyethylene was produced by imperial chemical Industries(ICI) in 1933 from free radical polymerization using a high pressure process. LDPE has a short and long branched chains with amorphous structure. The amorphous structure and weak intermolecular forces tend to reduce its density and tensile strength [33]. LDPE is widely used for manufacturing of various containers, plastic bags, tubing and molded laboratory equipments .

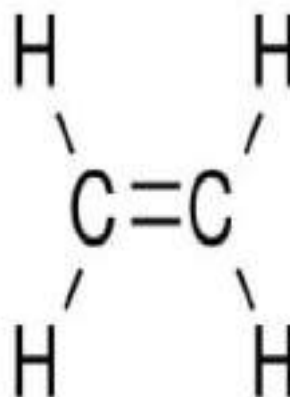


Figure (3.1) : Low Density Polyethylene

3.1.2 Zinc Oxide Nanoparticles (ZnO)

Zinc oxide (ZnO) is an inorganic compound known as zincite, which happens occasionally in nature, generally in a crystalline form. Because of the presence of manganese impurity, it is usually orange or red. It generally appears as a

white crystalline powder that is nearly insoluble in water and is widely used in many materials and products, including rubbers, plastics, ceramics, glass, cement, lubricants [34].

Owing to its prominent properties like chemical stability, low- dielectric constant, high luminous transmittance, high catalyst activity, effective antibacterial and bactericide, high ultraviolet and infrared absorption, high heat capacity and heat conductivity, low thermal expansion, high melting temperatures (1900 °C) and non-toxic, ZnO is one of the multifunctional compounds with increasing attention in recent years. The advanced of ZnO nanoparticles could improve mechanical and optical properties of polymer matrix [35].

Although it is possible to produce nanoparticles of many different types of materials, issues of compatibility with living cells limit the types of nanomaterials being considered for use in biomedical applications. ZnO is considered a "GRAS" substance (usually known as safe) [36]. Science shows overwhelmingly that, when applied to a skin in the lotion or cream based product, zinc oxide particles greater than 30 nm are not absorbed into the body, do not enter the blood stream and do not pose a threat to human health [37, 38]. ZnO nanoparticles can provide a better option for various biological applications and are widely used in the cosmetic industry, typically in sun screams and facial creams, due in particular to their easy manufacturing, environmentally friendly nature and non-toxic material. A variety of antimicrobial, food packaging applications is also encouraged by their recognized antibacterial properties [39]. The structure of zinc oxide shown in Figure (1.3).

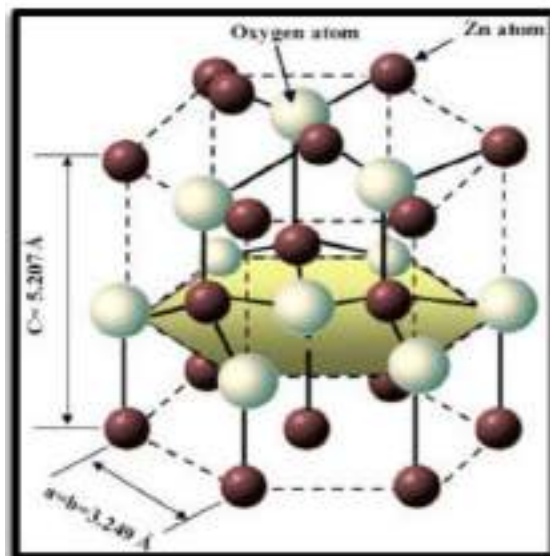


Figure (3.2) The hexagonal wurtzite structure model of Zinc oxide [40]

3.1.2.1 The Applications of Zinc Oxide Nanoparticles (ZnO)

- 1- Electronics industries
- 2- Textile industries
- 3- Pharmaceutical and cosmetic industries
- 4- Photocatalysis
- 5- Rubber industries
- 6- Miscellaneous applications
- 7- Vibration isolation
- 8- Microwave absorption and antibacterial influence [41]

3.1.2.2 The Properties of (Zinc Oxide Nanoparticles (ZnO))

- 1-Zinc oxide (ZnO) in nature occurs as a mineral zincite.
- 2-Water and alcohol insoluble, but acid and alkali soluble.
- 3- For maximizing the physical properties of the powder, the particle shape is important.
- 4- Depending on the manufacturing process zinc oxide particles may be spherical, acicular or nodular.
- 5- Conductive ZnO is known to have a lower resistance to heat since it is exposed to oxygen atmosphere for a long time [41].

3.2. Sample preparation

In this work, sheets of low density polyethylene polymer found in the laboratories of the Department of Polymer and Petrochemical Industries were prepared,

At different temperatures (180,200,220) °C respectively using a twin-screw extruder with a homogeneous thickness in an acceptable ratio. Samples filled with zinc oxide were prepared with an addition ratio of 5% and 10%.

Where the weight of the polymer was 45 to 5 zinc oxide And 40 to 10 zinc oxide at the same temperatures.

Table (3.1): Table showing details of samples.

Sample	Ratio	Temperature	Name
LDPE/ZNO	45/5	180	A1
LDPE/ZNO	45/5	200	A2
LDPE/ZNO	45/5	220	A3
LDPE/ZNO	40/10	180	A4
LDPE/ZNO	40/10	200	A5
LDPE/ZNO	40/10	220	A6

3.3. Mechanical Properties of Polymer:

3.3.1. Tensile test

Tensile test is a measurement of the ability of a material to applied forces tending to pull it apart and observe the extent of material stretches before breaking. Different types of plastic materials are often compared based on tensile property data (i.e. strength, modulus, and elongation data) [42]. The tensile test is one of the important mechanical tests through which it is possible to obtain many engineering information that determines the mechanical behavior of materials during use, including: [42,43]

- A.** Yield Stress.
- B.** Ultimate Tensile Strength.
- C.** Modulus of Elongation.
- D.** Percentage Elongation.
- E.** Percentage Reduction Area.



Figure (3.3): Indicates the tension device

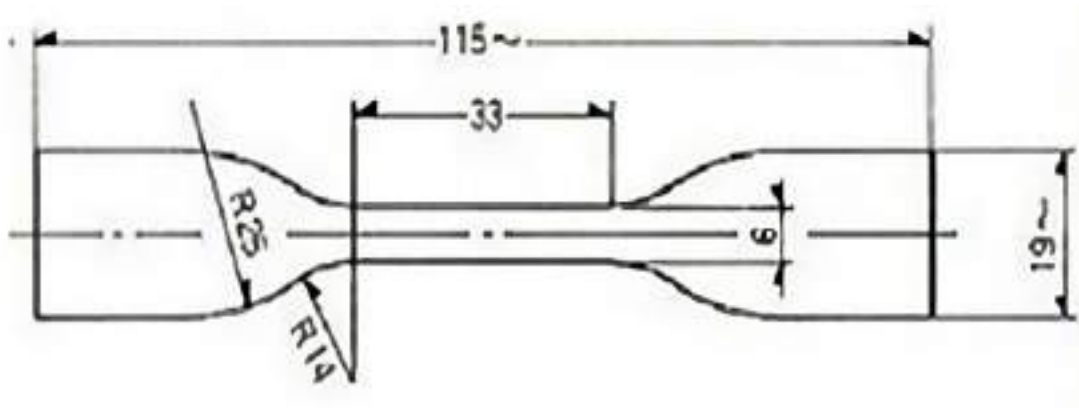


Figure (3.4) : Indicate the shape of the tensile sample with dimensions (ASTMD 638IV).

When a specific load is applied to a test sample so that it applies to its longitudinal axis, the sample will suffer from a certain elongation according to the amount of force applied to it, and thus we get a geometric curve called (stress-strain curve), (Stress-Strain Curve) [42, 44], as shown in the figure (4).

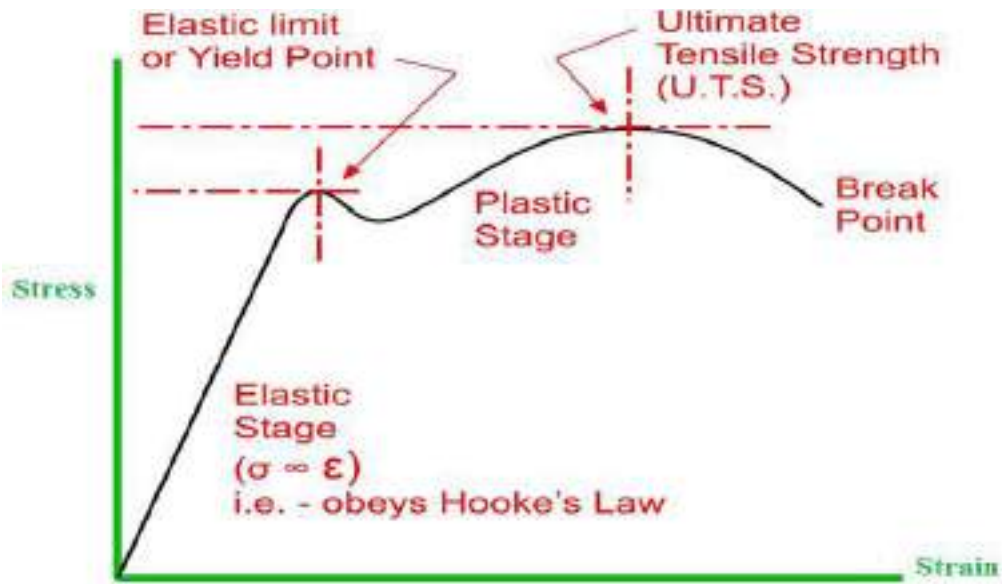


Figure (3.5): Typical Stress Strain Curve for Polymer Testing [45]

Where stress can be expressed by the following relationship: (42, 46).

$$\sigma = \frac{P}{A} \quad \text{-----(1)}$$

Stress is denoted by the symbol and its unit is N/m², where:

- P: is the force applied to the sample to cause elongation and is along the sample axis and its unit (N)
- A: is the cross-sectional area of the sample and its unit (m²)

As for the strain, it is symbolized by the symbol (ε) and it is expressed by the following relationship [45] :-

$$\epsilon = \frac{\Delta L}{L_o} = \frac{L - L_o}{L_o} \text{-----(2)}$$

Whereas:

- L = Elongation (final length of the sample).
- L_o = the initial length of the sample (initial)
- LΔ = the amount of change in length.

The strain is of two types: Elastic Strain and Plastic Strain [46], and the ratio between the stress applied to the material and the resulting strain in the elastic zone of the stress-strain curve is a fixed ratio called the modulus. Elasticity (Modulus of Elasticity) or Young's Modulus, which is symbolized by the symbol (E) and measured in units (N/m²) and expressed by the following relationship:

$$E = \frac{\sigma}{\epsilon} = \frac{P/A}{\Delta L/L_o} = \frac{PL_o}{\Delta LA} \text{-----(3)}$$

The importance of knowing Yunk's modulus lies in calculating the change in the dimensions of samples manufactured from homogeneous flexible materials under the influence of tensile or pressure loads, i.e. knowing the extent to which samples bear elongation or compression. Of great importance when choosing a material for a particular engineering application.

3.3.2. Hardness Test

Hardness is one of the basic mechanical characterization engineering materials. It can be defined as the resistance of the solid material to cut, scratching, wear, indentation, penetration and workability. In other words, it

is the resistance of the material to scratching by tools and machine harder than it. As compared with other characterizations hardness has the advantage of being easily to measure which can be determined from any piece of sufficient size [47].

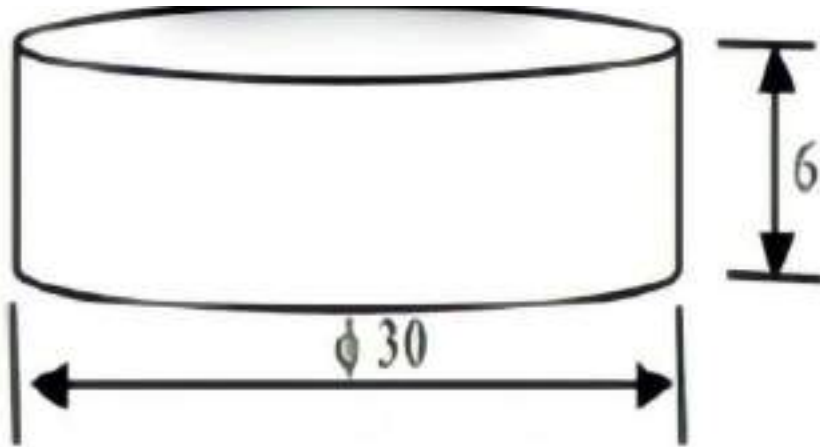


Figure (3.6): Indicate the shape of the hardness sample (shore D) with dimensions (ASTMD 2240).

Hardness has been well established in characterizing metallic material and ceramics for many years, but only recently it has been widely employed for characterizing polymers. In practice, hardness is measured in terms of the size of an impression made on a specimen by an indenter of a specified shape when a specified force is applied for a specified time; the indent being measured after the force has been removed. There are three principal standard testing methods for expressing the relationship between hardness and the size of the impression, these being Brinell, Vickers, and Rockwell. For practical and calibration reasons, each of these methods is divided into a range of scales, defined by a combination of applied load and indenter geometry. Shore (A and D) and Rockwell methods are applied for examining the hardness of polymers [48].



Figure (3.7): Indicates the hardness device (Shore D).

3.3.3. Impact test:

The impact properties of the polymeric materials depend mainly on the toughness of the material. Toughness can be described as the ability of the polymer to absorb applied energy. The molecular flexibility has a great significance in determining the relative brittleness of the material. Impact energy is a measure of toughness, and the impact resistance is the ability of a material to resist breaking (fracture) under a shock-loading [49].



Figure (3.8): Indicate the shape of the impact sample with dimensions (ASTMD 256-87).

Two basically different test methods, namely Izod and Charpy type, are used generally. In Izod type testing, the specimen is clamped vertically. to a cantilever beam and broken by a single swing of the pendulum released from the fixed distance from the specimen clamp [49] The impact strength is calculated by dividing the impact values obtained from the scale by the cross section area of the specimen. One point indicating the advantages of the Charpy test over an Izod test is that the specimen does not have to be clamped; therefore, it is free of variations in clamping pressures. Impact resistance can be obtained from the following relationship [50]:

$$\mathbf{I.S = UC/A \quad \text{-----}(4)}$$

Where -:

- I.S: impact resistance of the material (J/m²).
- UC: impact energy (J).
- A: cross-sectional area of the sample (m²).



Figure (3.9): Indicates the Impact device

3.3.4. Atomic Force Microscopy (AFM) Test:

AA3000 Scanning Probe Microscope is a very well device designed for use in research and industry, in which the inspector may do quick, easy analysis. Because the tip is placed into the base, there is no risk of being damaged by handling. tapping mode, contact mode, lateral force microscopy, all possible with the AA3000 Scanning Probe Microscope.

The main function of the standard unit is to see the area of samples prepared up to 10 microns by 10 microns. The device can be adjusted to inspect larger areas of the sample. With the existing “digital signal processor (DSP)” in the system, the device can treat more complex tasks effectively.

Chapter Four

4.1. Tensile test results:

When installing the prepared samples in a tensile test device type (5E/wdw) with a load (5kN) and a velocity value (10 mm/min) and conducting the test on it, a graph was obtained between the applied force (p) and the elongation occurring in the sample through the graph in the test device (ΔL), and from this diagram were extracted the stress strain values. The stress-strain curves were drawn for all the prepared samples and the figures below show the results of the tests.

Where the results showed when adding plasticizers, which are zinc oxide nanoparticles, will lead to a gradual decrease in the value of the modulus of elasticity as well as the values of tensile strength, and thus a decrease in its stiffness. The reason for this is due to the mechanical behavior of the plasticizers that increase the movement of the chains, thus reducing stacking, increasing elongation, and reducing chemical compatibility and physicist of matter.

As for stress, it decreases with increasing temperatures, and the strain and elongation of the material increase until the material reaches a state of failure.

Table (4.1): It shows elastic modulus and tensile strength of samples.

sample name	Elastic modulus (Gpa)	Tensile strength (Mpa)	Max Deformation (mm)
A1	0.06	8	31.581
A2	0.05	9	26.298
A3	0.03	2	3.583
A4	0.07	11	32.969
A5	0.06	12	24.281
A6	0.09	10	53.905

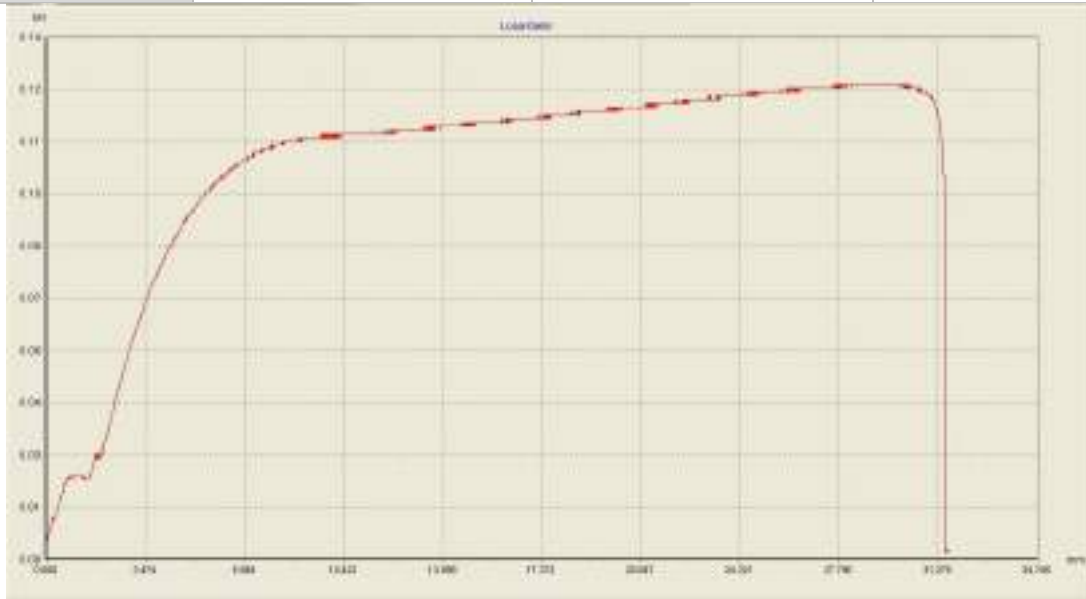


Figure (4.1): It Shows a stress-strain diagram for a sample (A1).

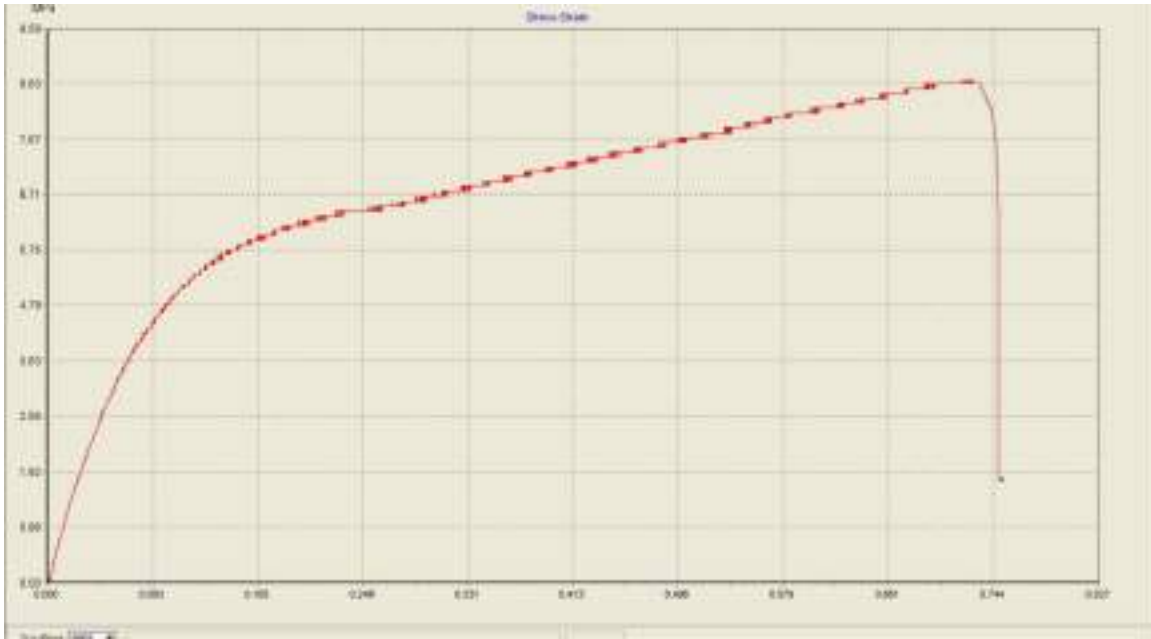


Figure (4.2): It Shows a stress-strain diagram for a sample (A2).



Figure (4.3): It Shows a stress-strain diagram for a sample (A3).

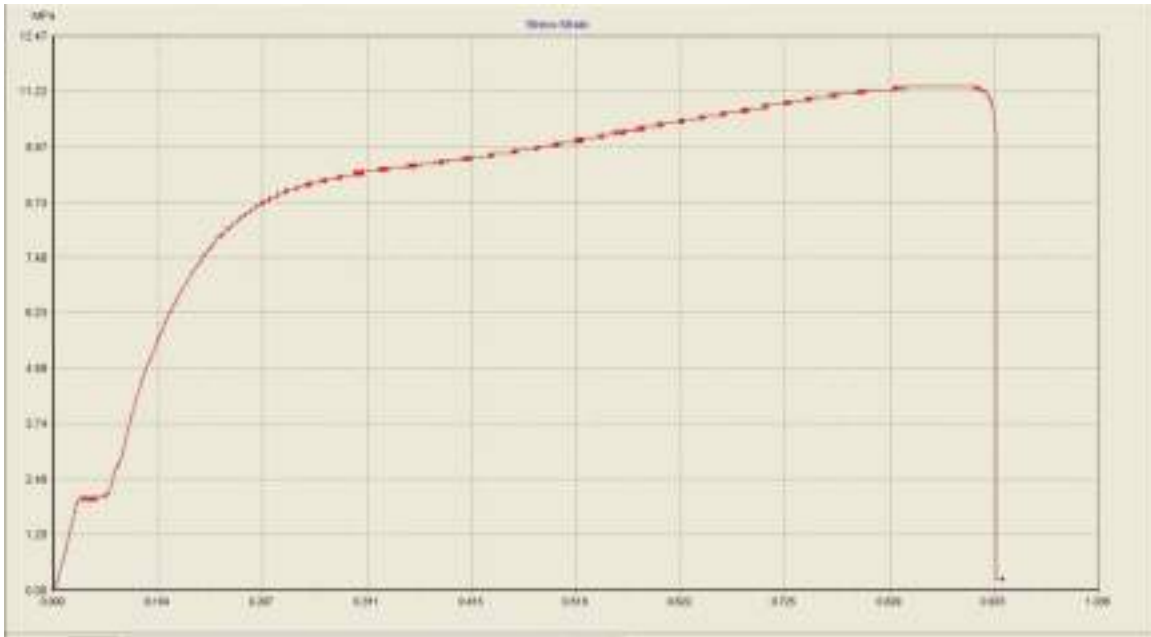


Figure (4.4): It Shows a stress-strain diagram for a sample (A4).

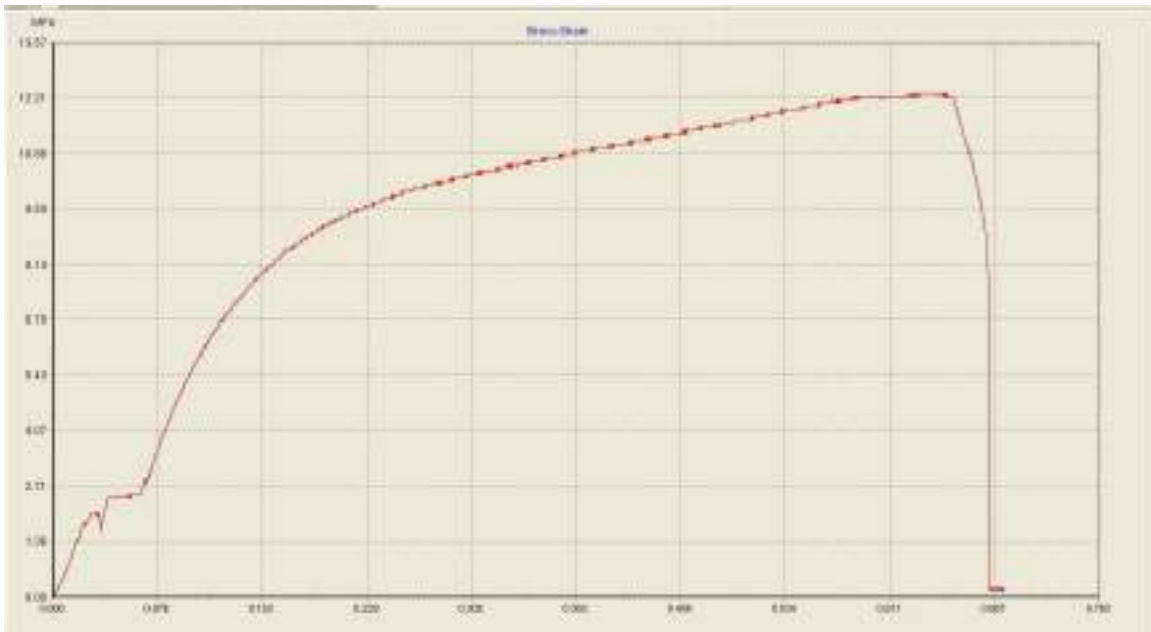


Figure (4.5): It Shows a stress-strain diagram for a sample (A5).

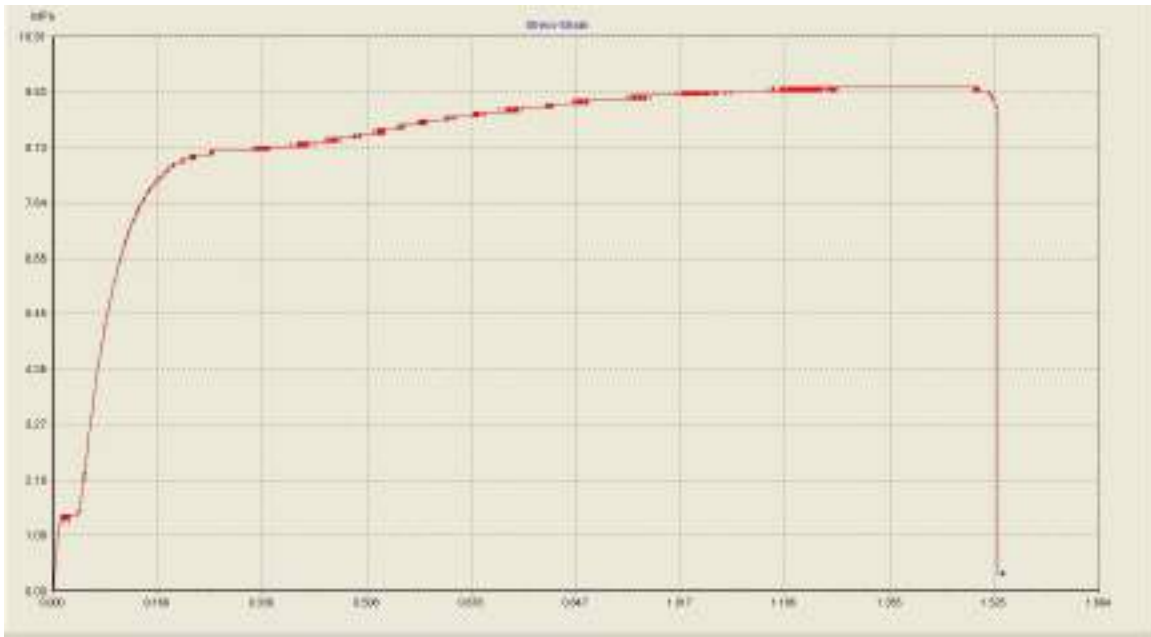


Figure (4.6): It Shows a stress-strain diagram for a sample (A6).





Figure (4.7): Tensile test sample before and after measurement.

4.2. Impact test result:

When placing the prepared samples in the impact tester type German pendulum impact tester, gant company (HAMBURG), model WP 400 type Charpy. And conducting the test on it, the impact energy was obtained, and through the presence of the measured sample area, the impact resistance was obtained through equation (4) for the six samples prepared according to the table (4.2).

The results showed that the impact resistance increased with increasing the flexibility of the material, and with increasing temperatures, the flexibility of the material increased due to the increase in the movement and flexibility of

the chains. Thus, there is a space between the polymeric chains that allow shock absorption.

Table (4.2): It shows Impact Strength of samples.

Sample name	Impact Strength (N.M)
A1	0.51
A2	0.5
A3	0.54
A4	0.1
A5	0.1
A6	0.13

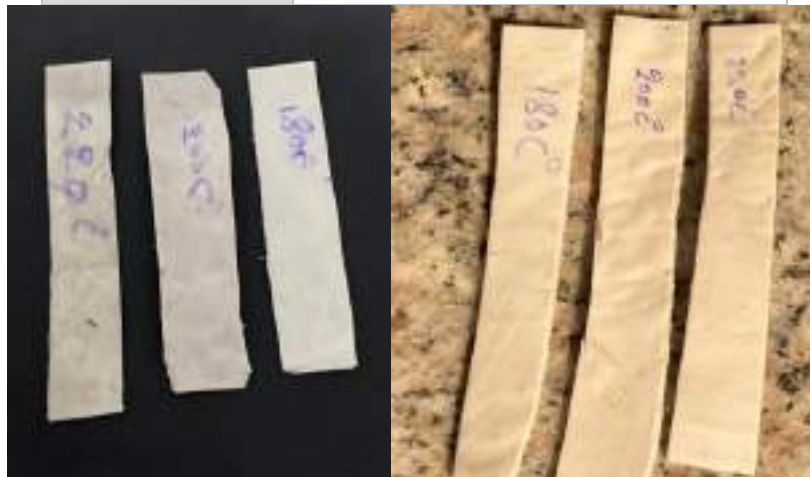


Figure (4.8): Impact test sample before and after measurement.

4.3. Hardness (Shore D) test result:

When the prepared samples were placed in the Shore De hardness tester and the test was conducted on them, three readings were measured for each of the six samples, and the final rate of hardness was taken to obtain the required accuracy according to the table (4.3). Where the results showed that the value of hardness is relatively lower when adding plasticizers, zinc oxide nanoparticles, which means that the hardness resistance is lower with increasing temperatures as well as the proportion of plasticizers. The hardness of materials depends on the type of forces that bind the atoms or molecules in the material. The higher the forces, the higher the hardness values. And vice versa.

Table (4.3): It shows value of hardness of samples.

sample name	Hardness
A1	48.2
A2	45.8
A3	47.6
A4	46.5
A5	42.8
A6	47.1



Figure (4.9): it Show the sheets of LDPE 45g+5g zno



Figure (4.10): it Show the sheets of LDPE 40g + 10g zno

4.4. AFM test result:

The 3-D AFM images and granularity accumulation distribution charts for samples are shown in Figure (4.11) ,(4.12) . LDPE has ball-shaped with good dispensability, homogenous grains and aligned vertically. By using special software imager, the estimated values of root mean square (r.m.s) of surface roughness average and average grain size are listed in Table (4.4). It is found that the grain size and the (r.m.s) of surface roughness increases when add increases [44].

Table (4.4): The grain size, roughness average and root mean square LDPE.

Different thickness(nm)	Grain size (nm)	Roughness average (nm)	Root mean square (nm)
A1	281.9	17.2	21.1
A4	301.1	22.2	29.2

This phenomenon can be attributed to nucleation and islet where the LDPE was growing. The compactness and homogeneity of the samples improved with the addition of ZnO, which led to the formation of large LDPE aggregates on the surface. Our results were in good agreement with [40]. The 3D images demonstrate that the grains are uniformly distributed within the scan area (500 × 500) nm with individual vertical grains extending upwards. This surface property is important for application in canning uses. Also, the AFM test showed the surface topography and grain size, which clearly increased with the addition of ZnO.

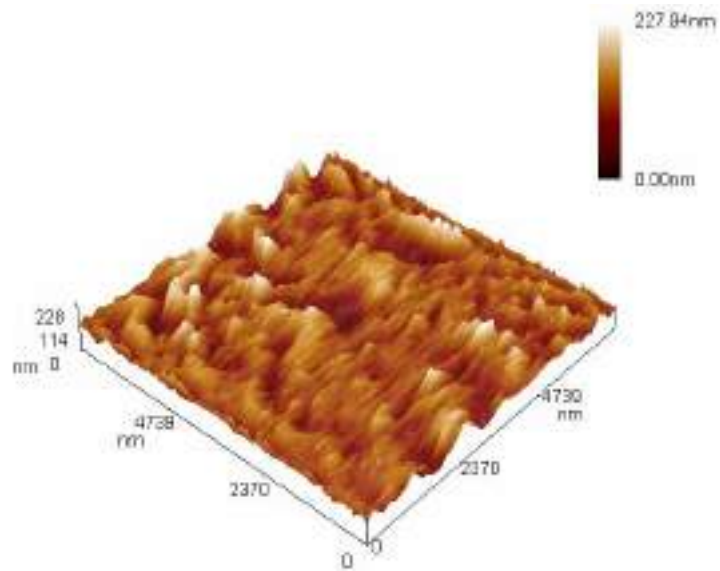


Figure (4.11): It Shows a stress-strain diagram for a sample (A1).

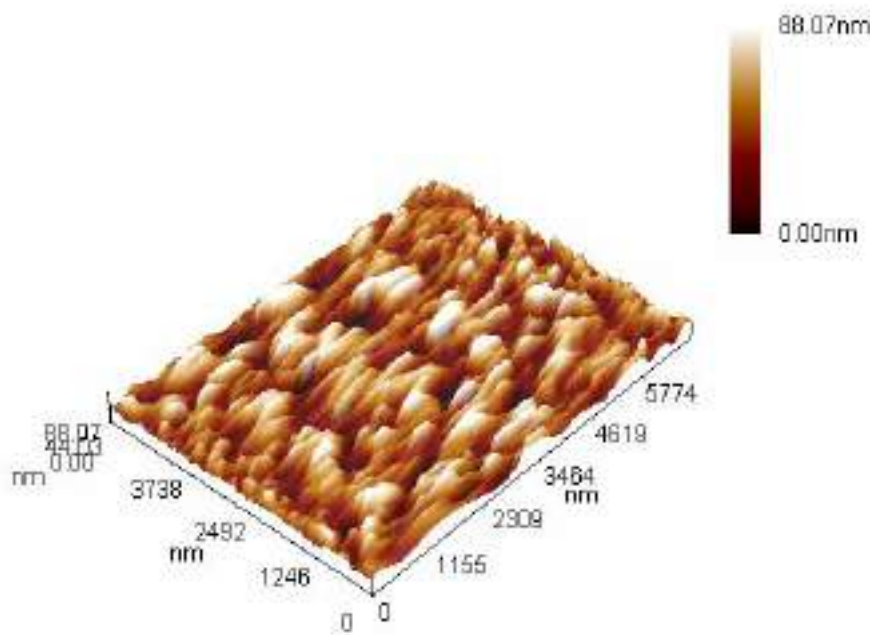


Figure (4.12): It Shows a stress-strain diagram for a sample (A4).

Chapter Five

5.1. Conclusions:

1- We noticed that the tensile values and the modulus of elasticity decrease, because the stress decreases due to the increase in the movement of the chains and their flexibility, and thus the elongation values increase.

2- We noticed an increase in shock resistance values due to the flexibility and movement of the chains, thus increasing their ability to absorb shock.

3- We noticed an increase in the addition rates of plasticizers as well as a decrease in the hardness resistance values due to the increase in the material's durability.

4- The percentages were (A2, A5) and the percentages were (45 LDPE + 5% ZnO) and (40 LDPE +10%ZNO)

at a temperature of 200 degrees Celsius is the optimum percentage in all tests.

5.2. Suggestion and Recommendations:

1. Characteristic Study of High Density Polyethylene Polymer.
2. Changing The Concentrations of Zinc Oxide Nanoparticles.
3. Study Of Structural And Optical Properties.
4. The Use of Zinc Sulfur Nanocomposite by Grafting High-Density Polyethylene And Low-Density Polyethylene Chains.

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