Republic of Iraq Ministry of Higher Education and Scientific Research University of Babylon College of Materials Engineering Department of Polymer and Petrochemical Industries



Improving The Properties Of Bag Factory Waste And Recycling It In Prouduction

Final year project report submitted in partial fulfillment of the requirements for the degree of bachelor of science polymers and Petrochemicals in the faculty of Materials Engineering / Babylon University

By

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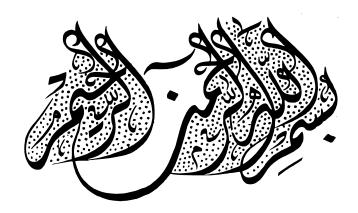
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Firstly, all the thank and praise be to **ALLAH** in enabling me to achieve this study.

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Dedicate

I dedicate this study to God (Glory be to Him) and then to the parents of this nation, **Muhammad Abdullah Abd al-Muttalib**, may God bless him and his family, and **Ali Ibn Abi Talib** (peace be upon him).

Then, to every **student**, **researcher**, **or lover** of the field of recycling plastic materials and improving their properties, I dedicate this

study to you, which will give you the cornerstone

Supervisors Certification

I acknowledge that this research is titled. "Improving The Properties Of Bag Factory Waste And Recycling It In Production ". It was prepared by the two students "Muhammad Ali Muslim "Hisham start jabber under my supervision at the University of Babylon / College of Materials Engineering / Department of Polymers and Petrochemical Industries, in fulfillment of the requirements for obtaining a bachelor's degree in Materials Engineering / Department of Polymers and Petrochemical Industries.

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Abstract

This work focuses on enabling plastic bag factories to use their waste again in production. When recycling low-density polyethylene, it loses 35% of its basic properties, as its melting point and the length of its internal chains decrease, and it becomes less dense, and the movement of its hydrocarbon chain increases. This study includes improvement Mechanical and rheological properties of recycled low density polyethylene. Where granules of pure low-density polyethylene were added in different proportions to the recycled granules of the same type, as well as corn starch was also added to it in different proportions, and the effect of this addition was studied on the physical and mechanical properties of the application of producing plastic bags.

In the first mixture, a mixture of pure low-density polyethylene (LDPE) granules with recycled low-density polyethylene (rLDPE) granules was prepared by twin-screw extruder at the ratios of 20%, 30%, and 50%. In the second mixture, a mixture of rLDPE granules was prepared.) with a natural polymer that is corn starch in specific proportions of 30%, 40%, 50% by weight of starch. rLDPE was used () as a base material, the material with the highest percentage was used (pure LDPE) and corn starch to improve the properties of (rLDPE).

Several tests were carried out to characterize and characterize the properties of the mixture such as tensile strength, hardness, scanning electron microscope, infrared spectroscopy and differential thermal analysis of the prepared mixtures.

The tensile and hardness test showed an increase in tensile strength and hardness resistance than that of (rLDPE) without addition. An evaluation of the thermal and rheological properties was carried out using DSC for the mixtures and compared with pure LDPE. DSC measurements indicate that LDPE \rLDPE mixtures are miscible in the crystalline phase and pure LDPE can increase the melting point

The results of DSC show that the pure low density polyethylene will enhance the natural flow of the recycled and improve the movement of the parts of the inner chain, and thus the rheological properties have improved and the ability to form and process increased compared to the smoothness and formability of the recycled when it is without addition. In the second mixture, the addition of starch led to an increase in tensile strength. The results of the SEM microscopy showed that the interfacial interaction was weak at 40% and 50% starch. The mixtures prepared using recycled rLDPE with starch were of the same tensile strength as pure LDPE, where starch was the agent. The main influence on the properties of the mixture. The use of this type of mixing with recycled polymer granules gives us two advantages, the first

Improving the rheological properties and tensile strength of the recycled polymer. Also, corn starch is a biodegradable polymer when mixed with inert recycled polymers such as rLDPE. Or the film that contains the remaining inactive components and thus disappears, in addition to that the starch will be an alternative to using the pure granules in the first improvement method, and thus the cost will decrease on the laboratories that use the returned granules because the starch is cheaper than the pure granules.

In this work, we have obtained desirable properties in the production plants, and these results are considered encouraging data from the manufacturing point of view.

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List of Abbreviations

Abbreviation	Meaning
ASTM	American Society for testing materials
LDPE	Low-density polyethylene
rLDPE	Recycle Low-density polyethylene
DSC	Differential scanning calorimetry
FTIR	Fourier transform infrared
EPDM	Ethylene Dimine Propylene Monomer
SEM	Scanning electron microscope
SALS	Small angle light scattering
SANS	Small angle neutron scattering
SAXS	Small angle X-ray scattering

PET	Polyethylene terephthalate
rPET	Recycle Polyethylene terephthalate
HDPE	High density polyethylene
rHDPE	Recycle High density polyethylene
UCST	Upper critical solution temperature
UV-vis	Ultraviolet-visible spectra
XRD	X-ray diffraction
PICS	Phase-induced critical scattering
SALS	Small angle light scattering

Symbols	Meaning	Unit
ΔG _{mix}	Free energy of mixing	J/K.mol
ΔH_{mix}	Enthalpy of mixing	J/g
ΔL	Change in length	mm
ΔS _{mix}	Entropy of mixing	J/K
A	cross- sectional area	m ²
Dc	Degree of crystallinity	Dimensionless
E	Young Modulus	GPa
F	Tension force	N
L	Final length	mm
Lo	Initial length	mm
Tg	Glass transition temperature	°C
T _m	Melting temperature	°C
٤	Failure strain	Dimensionless
б	Tensile strength	MPa

List of Symbols

1.1 Introduction

Plastic waste is an environmental disaster spread around the world. Resorting to the disposal of this waste has become an urgent and necessary goal at the present time. Here we will discuss one of the most famous processes used to get rid of plastic waste .that science the environment Ecology he that He studies relationship Human environment surrounding with it [1].

and its effect on him And he has showed studies and research that pollution This is amazing the environment Threatens life Humanity and even objects other. Although efforts exerted, the the situation environmental now worst from him before twenty years though from some Achievements own to limit from this deterioration [2]. that hangover Solid Solid waste Prepare from the problems that suffer Of which communities Humanity So I cared studies and research Scientific treat This is amazing the problems in a manner Penny And considerate safety the environment and health. According for anyone institutes French Lost reach out consumption the world Entire For the year2000 from Materials to(120) million tons paper and cardboard(70)million tons from Material consumerism(40)million tons glass(20)million tons utensils metallic,(22)million tons wood will be limited Our conversation on Materials PVC and offal. used Materials PVC in various aspects life service to the human being in Different domains, And she dissolved Shop a lot from Materials other So it increased its consumption and its production in a form big very in all around the world. that This is amazing Materials become Remnants after use it as goods So I became form a burden bear responsible its results communities Humanity expendable commodities This is amazing Materials. lhave I became hangover PVC form Danger increasing because get rid of Of which not command easy, She is not met him to compress And resist it High factors decomposition natural from air And a sun and humidity and rain, and not capacity objects live minute or enzymes on Analyze it. rate loopback any twirling may be no exceed(20%)So does maybe to imagine backlog Whopper that Effect on practical balance environmental And a reason glitch clear[3].

And let's give some examples:

Example(1)

Consume bottles water PVC on level worldwide more from million and a half tons from plastic that he from derivatives oil And often from polycarbonate Ethylene(PE).Although from that Somaya This is amazing Materials few unless that manufactured to produce plastic transparent Lead to emit Gases toxic Draw one hundred weakness when emitted from production amount similar from the glass. that fate This is amazing bottles after consumption water the packer with it he Garbage. what the solution Note that hangover PVC not met him to decompose And her capacity on Persistence and survival in the environment.

- burning plastic: Produce about him pollutants invasive serious like(dioxin)carcinogen.

- buried plastic: Produce about him material phthalates bad the influence which leak to water subterranean (one Sources Main for water packaged with this bottles) [4].

Example(2)

Manufactured bags plastic Generally from material the (PE) Which will be our work dedicated to this type of plastic which she series long from atoms carbon hydrogen, Which not met him to decompose by dozens or favorable years no Done re manufactured that it Evil and danger must from him, Which addition to that Its residues cause harm[4] :

- distortion beauty nature.

- Hinder growth the plant where prevent the sun and air about her.

- it causes death the animals that swallow it for her thought that it food and this is Lead for blockage the channel alimentary she has.

- Contains plastic bags Material chemical Melt in food And cause diseases in liver and lung She indicated one studies Scientific on Existence antiquities Material manufacturing in blood Human and performer for diseases malicious, As for Pregnancy foods hot in bags It is considered more severe Danger[5].

- in rivers And the seas lead to to die Preparation big very from fish Because she block Gills breathing, and lead sometimes to disable Machines and equipment Operating ships if they are encountered.

There are three ways get rid of from Remnants plastic and its damages[6]:

1- landfill or burial to the ground:hangover PVC not met him to decompose inside the soil Which Stay present causative pollution the soil Firstly, then that Leak some Materials Dakhla in installation plastic may be impact in water subterranean.

2- cremation : that to burn Remnants Materials PVC effects Destroyer on Human and plant and climate where He causes pollution air with materials carbonaceous severe Harm and emitted from That fires, which effect in layer ozone.

Example on that burn refills the(PVC)escalate fumes phosgene and acid hydrochloride captivity for poisoning as well vehicles dioxin Dioxinssevere severity.and with that the burn hangover PVC with a picture Rank And by means Scientific Modern may be be acceptable in some cases.

3- Recycle (Re manufacturing): that practical recycle and use for a hangover Materials PVC It is the only way to save economical solutions and environment and design for countries the world Advanced where she could overcome on the problems of waste accumulation.

As for in Countries developing the three quarters hangover Complete bury it and not rotate it. that recycle she method that Availability Benefit Economic addition to get rid of from hangover PVC. And count This is amazing method from better ways followed currently to process effects hangover Plastic in our world It must be noted that plastic materials in the recycling process are divided into: two cases from Materials [6]:

<u>A</u>-Material Thermoplastic plastic(TP):Which Same interaction inverse any maybe you give birth to it more from Once thermoplastic like ABS, PS, PP, PE, PVC.

<u>B</u>- Materials thermoset (Ts): Which Same interaction not inverse, any form once one thermoplastic whether She was Solid Mother liquid or on appearance powder. that This is amazing Materials out on Domain recycle unless in cases especially in the rate of around (20) from Remnants This is amazing Materials where Complete grind it well And used as materials Filling or stuffing mostly to reduce cost production for commodities. He should that point out to necessity non Use plastic The resulting on recycle in production products Dealing with her Human in a way directly to avoid Damages health

resulting on that unless in cases Especially without exaggerating this matter, because there are methods that can make it suitable for human food use, including the highly efficient cleaning method. I headed research Scientific and studies in the world to production Material and refills easy get rid of Of which after usage on road decomposition biological Biodegradable plastic, whether using objects live minute or on road enzymes, or plastic Materials (chips)with materials membership Like starch And cellulose in order to help the processes of biodegradation and the annihilation of these wastes[7].

The number of plastic waste recycling facilities in one of the major countries increased significantly from 1990 by 923 facilities to 1999 by 11677 facilities. Because it is the ideal solution to get rid of these wastes. In our research, we are working to improve the quality of the returned granules and make them more efficient[7].

<u>1.2 Aim of the study</u>

A study of improving the mechanical and rheological properties of recycled low-density polyethylene (meaning improving the properties of recycled plastic waste .

Where pure low-density polyethylene was added in proportions to the returned granules, as well as corn starch was added automatically, and the effect of this addition on the physical and mechanical properties of the .application of producing plastic bags was studied

2.1 Introduction

Polymer blend is an innovative way for providing materials with full set of desired specific properties at the low cost, for example a combination of strength and solvent resistance, strength and toughness, and so on. Blending likewise helps the manufacturer by offering improved processability, product uniformity, quick formulation changes, plant flexibility and high productivity, that's mean polymer blending is one of the well-known techniques utilized for developing new polymeric materials[8].

A polymer blend or polymer mixture is a part of a class of materials in which at least two polymers are mixed together to make another material with different physical properties. They combine the properties of the alloying components as an advantage and in some cases; the properties of the blend are superior to those of the individual components [9]. The properties of numerous plastics can be adjusted by mixing or alloying at least two polymers. These terms are frequently used reciprocally, however technically, blends are mixtures that are not fully compatible, and alloys are mixtures that are fully compatible [10]. The principal cost of polymer alloys and blends relies on the price of the main polymeric components and the interface modifier, as well as compounding costs:

Where W_i and C_i are weight fraction and cost in dollars per kilogram of ingredient i, and K is the compounding cost per kilogram of blend [11].

One of the issues associated with blends and alloys is how to make the components adhere together. One approach is to present a compatibilizer that makes interpenetrating polymer networks by providing a physical link between semi-compatible materials. Most compatibilizers are block copolymers and they create an interwoven matrix of physically inseparable, however chemically distinct polymers [10]. The general relation between blends and alloys is shown in Figure (2.1). Polymer alloy constitutes a particular sub-class of polymer blend; virtually all high performance engineering blends are alloys. The properties of polymer alloys are reliant on the properties of each of the components, the compound structure and the phase separation structure [11].

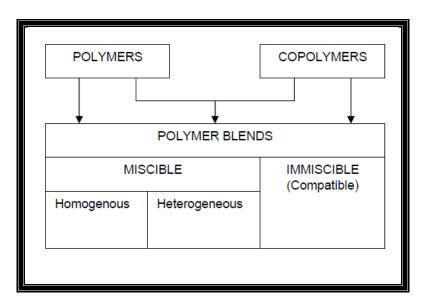


Figure (2.1): Interrelations in polymer blend nomenclature [11].

The quality of morphology in polymer blends are directly related to the rheological properties of the component of the blend, on the processing condition used to obtain the blend and the compatibility between the polymers forming the blend which is governed by the interfacial tension between the component forming the blend and on the composition of the blend[7]. The ultimate behavioral pattern of polymer blends is dependent on Interaction between the component polymers, Nature of the phase provided by the matrix material, Character of the dispersed phase and Extent of phase separation [9]:-

There is no doubt that economy is the primary reason for blending, compounding and strengthening. The material remain competitive if it can be manufactured at a lower cost with properties meeting the required specification. In general, the following economyrelated reasons can be recorded [11]:

- 1. Better processability, so improved product uniformity and scrap reduction.
- 2. Product tailorability to specific customer needs, thus better customer satisfaction.
- 3. Quick formulation changes, so better plant flexibility and high productivity.
- 4. Blending reduces the number of grades that need to be manufactured and stored, so savings space and capital investment.
- 5. Recyclability of blends achieved by control of morphology.

2.2 Polymer blends types:-

There are two types of polymer blends: - miscible and immiscible. By far most of polymer blends are immiscible with each other. There are few commercially important polymer blends based on miscible and partially miscible (miscible within a low range of concentration) polymer pairs. It is sometimes at least two polymers are mixed to make a blend with useful properties. It is important to devise a particular procedure for compatibilizing the mixture to accommodate for ideal physical performance and long-term stability when preparing a new polymer blend from immiscible resins [12].

2.2.1 Miscible polymer blend:-

Miscible polymer blends are an expensive, easy-to-use approach for investigating and refining polymer combinations. It facilitates combinations that work and some that don't. Miscible polymer blends behave similarly to what is expected of a single phase system. Their properties are a combination of properties of pure components. The characteristics of components affecting the proper-ties of miscible blends are their chemical structure and molecular weight, their concentration and intermolecular interactions. While miscible blend system is of considerable scientific and practical interest, it should not be concluded that miscibility is always preferred with respect to properties [13].

2.2.2 Immiscible polymer blend: -

More than two phases are displayed in this blend. At certain range of temperature, pressure and composition immiscibility may be restricted. One remarkable property of immiscible blends is that one made from two amorphous polymers has two glass transition temperatures, since the two components are phase isolated, they and their T_g separate. To see weather if the blend is miscible or immiscible, researchers frequently measure the T_g of the blend. If two T_g are discovered, then the blend is immiscible. If only one T_g is observed, then the blend is miscible. An immiscible blend is called compatible if it is a useful blend wherein the inhomogeneity brought on by the different phases is on a small enough scale not to be apparent in use (compatible blends are that are miscible in certain useful ranges of composition and temperature, but immiscible in others). Most blends are immiscible and can be made compatible just by an assortment of compatibilization techniques [15]. Also there are another types of polymer blend, such as homologous and isomorphic

2.2.2.1 Homologous polymer blend:-

Mixture of two or more fractions of the same *polymer* each of which has different molecular weight distribution such as polystyrene PS with different fractions [14].

2.2.2.2 Isomorphic polymer blend:-

Polymer blend of two or more different semi-crystalline polymers are miscible in the crystalline state as well as in the molten state. This type of blend displays single composition-dependent glass-transition temperature T_g and single composition-dependent melting point T_m . This behavior is extremely rare such as PVF/PVDF [14].

Figure (2.2) indicates idealized expected property combinations from blending two polymers that are either miscible (center line), immiscible and compatibilized line), (top or immiscible and uncompatibilized (bottom line). One can only hope to acquire an average of properties relying upon the proportion of each polymer present in the blend when polymers are miscible in all extents. In a typical case, as the higher T_g polymer increases in proportion in the blend the T_g of the blend will differs straightly from that of the lower T_g polymer to that of the higher T_g polymer. One generally obtains a mixture with physical properties more awful than those of either individual polymer when two immiscible polymers are blended without compatibilization. Generally, since there is no mechanism for stabilizing a dispersion of one polymer in a matrix of the other, such a blend has poor structural integrity and poor heat stability [16].

The poor stress transfer between the phases of the immiscible blend are the main reason of poor stretching and impact-strength properties. Since low molecular attractive forces between the blend components are in charge of immiscible-phase behavior, low attractive or adhesive forces between phases are to be expected, and it is believed that this poor interfacial adhesion causes immature failure under stress as a result of the usual crack-opening mechanisms [17].

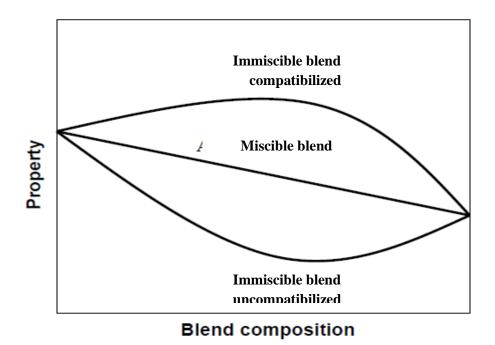


Figure (2.2): Potential effect on polymer blend properties with component concentration [16].

2.3 Blend Preparation Methods

Most polymer pairs are immiscible, and subsequently, their blends are not formed spontaneously. Additionally, the phase structure of polymer blends is not in equilibrium and relies on upon the process of their preparation. Five distinct methods are utilized for the preparation of polymer blends: melt mixing, solution blending, latex mixing, partial block or graft copolymerization, and preparation of interpenetrating polymer networks [28, 29]. It ought to be mentioned that due to a high viscosity of polymer melts, one of these methods is required for size reduction of the components (to the order of μ m), even for miscible blends.

Melt mixing is the most widespread method of polymer blend preparation in practice. The blend components are mixed in the molten state in extruders or batch mixers. Preferences of the method are welldefined components and universality of mixing devices—the same extruders or batch mixers can be utilized an extensive varity of polymer blends. Disadvantages of the method are high energy consumption and conceivable unfavorable chemical changes of blend components[22].

Solution blending is often as possible utilized for preparation of polymer blends on a laboratory scale. The blend components are dissolved in a typical solvent and intensively stirred. The blend is isolated by precipitation or evaporation of the solvent. The phase structure formed in the process is a function of blend composition, interaction parameters of the blend components, type of solvent, and history of its separation. Preferences of the process are rapid mixing of the system without large energy consumption and the possibility to unfavorable chemical reactions. On the other hand, the method is restricted by the need to find a typical solvent for the blend components, and specifically, to remove huge amounts of organic (frequently toxic) solvent. Therefore, in industry, the technique is utilized only for preparation of thin membranes, surface layers, and paints.

A blend with heterogeneities on the order of 10 μ m can be set up by mixing of latexes without utilizing organic solvents or large energy consumption. Significant energy is required just for removing water and eventually achievement of finer dispersion by melt mixing. The entire energy balance of the process is normally better than that for melt mixing. The need to have all components in latex form confines the utilization of the process. Since this is not the situation for most synthetic polymers, the application of the process in industrial practice is constrained[23].

In partial block or graft copolymerization, homopolymers are the essential product. But, an amount of a copolymer adequate for achieving good adhesion between immiscible phases is formed. In most cases, materials with better properties are set up by this method than those formed by pure melt mixing of the corresponding homopolymers. The drawback of this process is the complicated and costly startup of the production, in comparison with other methods, e.g. melt-mixing.

Another method for synthesis of polymer blends is by formation of interpenetrating polymer networks. A network of one polymer is swollen with the other monomer or prepolymer; from that point onward, the monomer or prepolymer is cross-linked. In contrast to the preceding methods it is utilized for thermoplastics and uncross-linked elastomers, blends of react plastics are set up by this method.

2.4 Thermodynamics of polymer blend:-

At equilibrium, a mixture of two amorphous polymers may exist as a single phase of intimately mixed segments of the two macromolecular components or separate into two distinct phases comprising basically of the individual components [17]. It is necessary to note that amorphous polymers form glassy phase upon adequate cooling and that a homogeneous, or miscible, polymer blend displays a single, composition- dependent glass- transition temperature Tg, though an immiscible blend has isolate glass transitions related with each phase. Equilibrium-phase behavior of mixtures is represented by the free energy of mixing ΔG_{mix} [11, 17].

And how this amount, consisting of enthalpic (Δ H) and entropic (Δ S) parts, is affected by composition and temperature T. If two polymers are mixed, the most frequent result is a system that displys a complete phase separation due to the repulsive interaction between the components (i.e. the chemical incompatibility between the polymers). Complete miscibility in a mixture of two polymers requires Δ G_{mix} which must be negative and that the accompanying condition is satisfied [30]:

$$\Delta G_{\text{mix}} = (\Delta H_{\text{mix}} - T\Delta S_{\text{mix}}) < 0 \dots (2.3)$$

For a stable one-phase system, criteria for phase stability of binary mixtures of composition φ at fixed temperature T and pressure p are:

$$\Delta G_{mix} < 0$$
, and $\left(\frac{\partial^2 \Delta G_{mix}}{\partial \phi^2}\right)_{P,T} > 0$ (2.4)

Miscible polymer blend is a polymer blend which is homogeneous down to the molecular level and associated with the negative value of the free energy of mixing and the domain size is comparable to the dimensions of the macromolecular factual segment. The value of T ΔS_{mix} is always positive since there is an expansion in the entropy on mixing. Therefore, the sign of ΔG_{mix} dependably relies on upon the value of the enthalpy of mixing ΔH_{mix} . The polymer pairs mix to form a single phase just if the entropic contribution to free energy exceeds the enthalpic contribution, i.e.

$\Delta H_{\text{mix}} < T \Delta S_{\text{mix}} \cdots (2.5)$

For most polymer blends the miscibility increments with increasing pressure. The effect depends on the magnitude of the heat of mixing ΔH_{mix} . For $\Delta H_{mix} < 0$, the miscibility is enhanced by compression, though for those with $\Delta H_{mix} > 0$ it is decreased.

The composition range over which the two polymers phaseseparate is not constant. It can change with temperature. Figure (2.4 a) demonstrates the dependence of the free energy of a mixture on composition at three temperatures. At T_1 , the above two conditions are fully satisfied, and miscible, single–phase mixtures occurs for all compositions at this temperature.

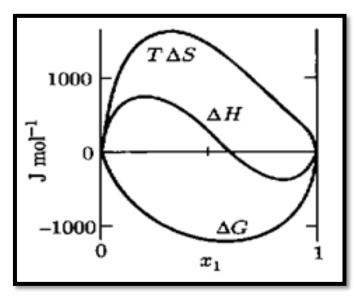


Figure (2.3): Assessment of ΔG_{mix} of polymer blend. ΔH_{mix} can be positive or negative quantity:

If A-A and B-B interactions are stronger than A-B interaction, then $\Delta H_{mix} > 0$ (unmixed state is lower in energy). If A-B interactions are stronger than pure component interaction then $\Delta H_{mix} < 0$ (solution state is lower in energy).

At T₂, equation (2.4) is not satisfied for all compositions, and mixtures between the points B and B' isolate into two phases with these compositions as this results in a total free energy falling on the dashed line, which is lower than that of the homogenous phase (solid line). The curve for an intermediate temperature T_c has been drawn in a manner satisfying the conditions of a critical point at c.

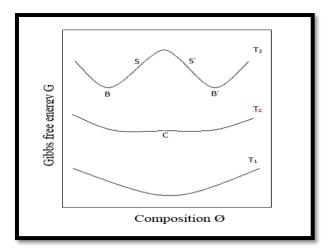
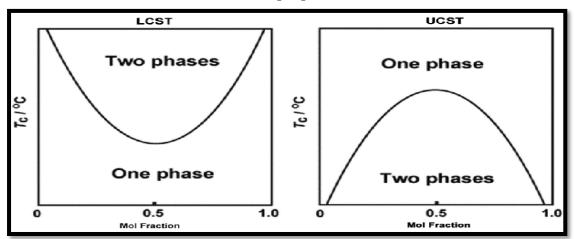
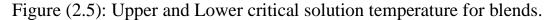


Figure (2.4): Phase diagram of mixtures composition dependence of the free energy of the mixture (on an arbitrary scale) for temperatures above and below the critical value [17].





For some polymer pairs that range of immiscibility gets smaller as temperature increments. Eventually, if one heats such a pair sufficiently high, which range of immiscibility, will turn out to be so small that it will disappear. The temperature at which this happens $(T_1>T_2 \text{ and } T_c)$ is known as the *upper critical solution temperature or UCST* Figure (2.4 b). The upside –down parabola is *the* boundary between those temperatures and compositions at which there is one phase, and those at which there is phase separation. However, in some cases the opposite happens. For some polymer pairs the range of immiscibility decreases with decreasing temperature. If one cools such a pair enough, in the end, the temperature is reached at which the range gets so small that it disappears. This temperature ($T_2 > T_1$ and T_c) is known as *the lower critical solution temperature or LCST*. If one plots the range of immiscibility versus temperature Figure (2.4 c), the plots look like an inversion of the UCST plot. These temperaturecomposition ($T-\Phi$) planes characterize the liquid-liquid phase diagram for a two-component mixture. Most bicomponent blends display upper critical solution temperature (UCST) behavior, in which they stay homogeneous at elevated temperatures (in the melt) and phase–separate at low temperatures.

UCST and LCST points are possible in diagrams that are more complex. A schematic phase diagram is shown in Figure (2.6). There are three regions of various level of miscibility:-

1. Miscible region single-phase between the two binodals.

2. The four fragmented metastable regions between binodals and spinodals.

3. The two-phase separated regions of immiscibility, bordered by the spinodals.

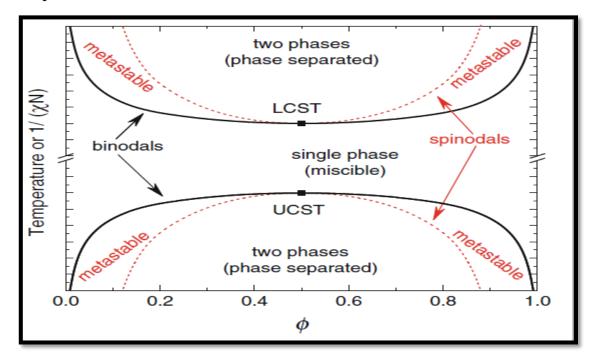


Figure (2.6): Phase diagram for liquid mixtures with the upper and lower critical solution temperature, UCST and LCST, respectively [30].

The demonstrate shows two critical solution temperatures, the lower LCST (at higher temperature), and the upper UCST (at lower temperature). The phase diagram with two critical points is a rule for mixtures of low molar mass components, though the polymer blends typically indicate either LCST (most) or UCST. Both UCST and LCST can be observed in polymer blend. UCST: low molar mass with only dispersive forces, LCST: polymers with either specific interaction or free volume. In principle, every miscible blend is bounded by upper (LCST) and lower (UCST) temperature limits. However, in reality the LCST may be higher than the degradation temperature of the blend and UCST may be lower than the T_g and hence can not be determined.

The binodals in Figure (2.6) separate miscible (one-phase) and metastable region, the spinodals separate metastable and two-phase region. The thermodynamic conditions for phase separations are given by:

Spindal:-
$$\left(\frac{\partial^2 \Delta G_{mix}}{\partial \phi^2}\right)_{P,T} = 0$$
(2.6)
Critical point:- $\left(\frac{\partial^2 \Delta G_{mix}}{\partial \phi^2}\right)_{P,T} = \left(\frac{\partial^3 \Delta G_{mix}}{\partial \phi^3}\right)_{P,T} = 0$ (2.7)

The phase separation happens when a single-phase system suffers a change of composition, temperature or pressure that forces it to enter either the metastable or the spinodal region. At the point when the system enters from single-phase region into the metastable region, the phase separation happens by mechanism resembling crystallization – slow nucleation followed by growth of phase separated domains. By difference, when the system is forced to jump from a single-phase into

the spinodal region of immiscibility the phases separate spontaneously by a mechanism called spinodal decomposition [31].

2.5 Compatibility in Polymer Blends:-

Compatibility is defined as the capability of the individual component substances in either an immiscible polymer blend or a polymer composite to exhibit interfacial adhesion. It is often established by the observation of mechanical integrity under the expected conditions of use of a composite or an immiscible polymer blend [15].

In general, the compatibility between the polymer phases decides the properties of a heterogeneous polymer blend [33]. The interface between the polymer phases in a polymer system is characterized by the interfacial tension which, when approaching zero, causes the blend to become miscible. In other words, if there are strong interactions between the phases then the polymer blend will be miscible in nature. Large interfacial tensions lead to phase separation, with the phase separated particles perhaps undergoing coalescence; this will result in an increased particle size and, in turn, decreased mechanical properties. The interfacial tension can be reduced by the addition of interfacial agents known as compatibilizers [33]; these are generally molecules with hydrophobic and hydrophilic regions that can be aligned along the interfaces between the two polymer phases, causing the interfacial tension to be reduced and the compatibility of the polymer blends to be increased. Compatibility results in a reduction of the dispersed particle size, an enhanced phase stability, and increased mechanical properties [33]. The physical properties of miscible, compatabilized and uncompatabilized blends can be characterized using techniques such as thermogravimetric analysis, dynamic mechanical thermal analysis, and universal testing machines.

Compatibilization is the process of modification of interfacial properties in immiscible polymer blends. This results in the reduction of the interfacial tension, stabilization of the desired morphology and improved interaction between phases of solid state. In order words, compatibilization results in the formation of polymer alloys. Compatibilization is accomplished by physical or chemical means. In the former case, the desired level of dispersion is generated by physical means, then physically stabilised (for example; by quenching, retardation, cross-linking or co-crystallization). In the later case, the morphology does not only depend on the level of mechanical mixing, but to a great extent, it is controlled by the compatibilizer [34].

Use of selected compatibilizers in the form of appropriate block copolymers or coupling agents as the silane or titanate coupling agents may significantly help in dispersion of additives in polymers by enhancing adhesion or by minimizing phase separation in binary or multicomponent polymer blends by playing the role of a surfactant.

Degree of compatibility is frequently based upon the mechanical performance of the composite, the interphase thickness, or the size of the phase domains present in the composite. It is a measure of the strength of the interfacial bonding between the component substances of a composite or immiscible polymer blend. The term *degree of incompatibility* is once in a while used instead of degree of compatibility. Such use is discouraged as incompatibility is related to the weakness of interfacial bonding [15].

2.6 Miscibility Determination in Polymer Blend Systems:-

It is hard to determine ΔG_m from experiment. Different methods have been utilized to determine miscibility and phase behavior of polymer blend systems. Common methods that are utilized include the determination of the glass transition temperatures, microscopy, light or radiation scattering, nuclear magnetic resonance spectroscopy, and infrared spectroscopy. Each experimental technique has limited sensitivity to detect a domain size. Therefore, the phase behavior determined from various measurement techniques may vary, and thus may not precisely reflect the thermodynamic phase behavior of the blend. According to reference [37, 38] the methods for determining polymerpolymer miscibility are summarized below.

(a) Miscibility Determination by Glass Transition Temperatures

The occurrence of a single T_g intermediate between that of the pure components suggests miscibility of the polymer pair. A few expressions have been proposed for the composition dependence of the glass transition temperature for miscible blends.

The determination of T_g can be done by calorimetric method. The sensitivity of differential scanning calorimetry (DSC) is reported only detecting a domain size approximately larger than 10 nm. This is a widely used technique because of its simplicity.

(b) Miscibility Determination by Microscopy

Electron micrographs taken through scanning and transmission electron microscopy can observe the phase segregates. The contrast of scanning electron microscopy (SEM) depends on differences of surface topography or texture [39]. This can be emphasized by breaking the specimen in its glassy state, or etching one phase away. When the domain-phase is chemically etched, dimples can be seen by SEM.

(c) Miscibility Determination by Scattering Methods

The methods can determine the average domain size in a blend. Small angle light-scattering (SALS) is less sensitive than small-angle xray scattering (SAXS), which means SALS cannot recognize small particles such as the case of blends with specific interactions. Techniques utilized are cloud-point methods through SALS, traditional light scattering method, phase-induced critical scattering (PICS), neutron scattering methods particularly small-angle neutron scattering (SANS), and SAXS.

(d) Miscibility Determination by Heat of Mixing by Calorimetry

A few reviews utilize DSC to measure the thermal energy released upon blending. They conclude that the exothermic reaction during blending indicate interaction in a blend.

(e) Miscibility Determination by Melting Point Depression

This method applies to the blend systems that contain crystalline polymers. The utility of the melting point depression permits the calculation of the interaction parameters between polymer species.

(f) Turbidity Measurements

The method consists of preparation of a series of mixtures of varied concentrations (near the phase separation condition) then causing the separation to occur, e.g., by ramping the temperature. The onset of turbidity is observed visually, using a photoelectric cell, or by a UVvisible spectrophotometer. The ensemble of the cloud points defines the cloud-point curves (CPC) that closely follow the system's binodal. The method can be extended to rigorous studies of phase separation by measuring the light-scattering intensity. For polymer blends, the CPC is usually determined by preparing films under conditions of miscibility, in a wide range of compositions. The films are then heated through the cloud point at a rate not exceeding 0.1 C/min. Depending on the rate, type of system, and polydispersity, the hysteresis (difference between CPC on heating and cooling) can be significant[40].

(g) Ultrasonic Velocity

For homogenous systems, the ultrasonic velocity is related to the ratio of modulus to density. Thus, one may expect that any method that determines density changes with adequate precision can provide a measurement or an indication of miscibility. The ultrasonics can also be used as a fast screening method for the optimization of processing and its parameters, for example, employed as online characterization of polymers blends. The compressive ultrasonic velocity (6 MHz, at room temperature) was used to study cast blend films of PMMA/PVAc, PMMA/PS, PVC/CR, and PS/EPDM. A linear correlation between the sound velocity and the composition was observed for miscible blends, whereas immiscibility, viz, in PMMA/PS blends, the same dependence was irregular. Phase separation in PVC/CR was detected at w ¼ 70 wt% of CR, indicated by a sudden departure from linear correlation. The ultrasonic absorption versus composition gave even stronger evidence of immiscibility [40].

(h) Transmission Electron Microscopy TEM

There has been a tremendous development in transmission electron microscopy (TEM) since its discovery by Ruska. To study the polymer morphology by TEM, a sample must be thin, usually thinner than about 200 nm. However, using field emission guns (FEG), thicker specimen ($\leq 2 \mu m$) can be used. Since polymers are mainly composed of C, H, N and O atoms, the electron density difference between polymers is not large enough to achieve sufficient contrast in heterogeneous materials. Thus staining techniques are usually necessary. For most cases staining with osmium or rhutenium tetroxide (OsO₄ or RuO₄, respectively) yields sufficient results. OsO₄ reacts with isolated double bonds (C=O, C=C) such as the double bonds in polyisoprene and poly butadiene, but it does not react with conjugated double bonds. The mechanism of RuO4 staining seems to be different from OsO₄. It does not react directly with chemical species, but rather it forms clusters. It stains most polymer but to a different degree dependent mostly on the diffusion rate. The diffusion of RuO4 into the polymer is most important. For crystalline polymers, RuO4 diffuses preferably into the amorphous regions and stains them, whereas the lamellar crystals remain unstained. The double staining technique facilitates a clear contrast for more complicated systems [40].

(i) Spectroscopic Methods: NMR

For the studies of interactions in polymer blends, nuclear magnetic resonance (NMR) is of principal significance. The NMR methods for the studies of polymer blends are well described in textbooks as well as in several reviews. The NMR parameters used in the determination of polymer/polymer miscibility are mainly the times related to the half-life of the spin relaxation, such as the spin–lattice relaxation time (T_1), the spin-spin (T_2), and the spin–lattice in the rotating frame (T_{1r}). The shorter the relaxation time, the broader is the NMR line width (for solid samples the lines are broad, a manifestation of slow reorientation of bonds, whereas for liquids and solution they are narrow, as expected from faster bond reorientation). The position of the lines, i.e., the precessional frequency of the nucleus, depends on its chemical environment, spatial configuration, and interactions. The position of the peak or the so-called chemical shift (usually quoted as d in parts-per-million, ppm) is a reflection of the energetic state of the nucleus, while the line intensity is that of its population [40].

2.7 Materials Used In the Research

2.7.1 Low density polyethylene and Recycle Low density polyethlen

Low-density polyethylene (LDPE) is so named because such polymers contain substantial concentrations of branches that hinder the crystallization process, resulting in relatively low densities. The branches primarily consist of ethyl and butyl groups together with some long chain branches [44].

Due to the nature of the high pressure polymerization process by which low density polyethylene is produced, the ethyl and butyl branches are frequently clustered together, separated by lengthy runs of unbranched backbone. Long-chain branches occur at random intervals along the length of the main chain [44].

The long-chain branches can themselves in turn be branched. The numerous short-chain branches found in low density polyethylene reduce its degree of crystallinity well below that of high density polyethylene, resulting in a flexible product with a low melting point. Long-chain branches confer desirable processing characteristics, high melt strengths coupled with relatively low viscosities. Such characteristics eminently suit it to the film-blowing process, products of which are its principal outlet, accounting for more than half of all its use. Major applications include low load commercial and retail packaging applications and trash bags [44].

Other uses include diaper backing, shrink-wrap, vapor barriers, agricultural ground cover, and greenhouse covers. Low-density polyethylene can be coated onto cardboard to create a waterproof and heat-sealable composite widely used in fruit juice and milk cartons. Minor uses include wire and cable insulation and flexible pipe. Injection-and blow-molded items made from this resin are flexible and reasonably tough, suiting them for such applications as squeeze bottles and food storage containers. Low-density polyethylene resins typically have densities falling in the range of approximately 0.90–0.94 g/cm³ [44].

LDPE has light-weight, good impact resistance, extremely high flexibility, no moisture absorption and has high chemical and corrosionresistivity. It is used mainly for the blown and cast film, extrusion products as well as coatings, rotational and injection molding [45], Figure (2-13) shows different structures of LDPE.

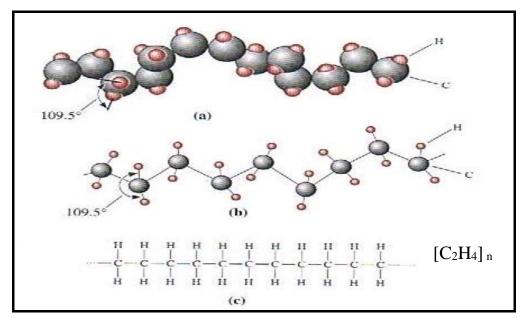


Figure (2.13): Three ways to represent the structure of PE, (a) solid three dimensional model, (b) a three dimensional "space" model and (c) a simple two dimensions model [46].

2.7.2 coner starch

Corn starch is a white, tasteless, odorless powder, used in food processing, papermaking, and the production of industrial adhesives; it is also a component of many cosmetics and oral pharmaceutical products. It has been used as a lubricant in surgical gloves.

Molecular formula	C12H22O11
Molecular Weight (g / mol)	342.30
Color	white
Density (g/ ml)	1.5

2.8 Mechanical Properties tests

2.8.1 Tensile Property

The tensile test is one of the most important mechanical tests through which many important engineering information can be obtained that determine the mechanical behavior of materials during use, including [56]:-

- A. Yield stress.
- B. Ultimate tensile strength.
- C. Modulus of elasticity.

When a particular load is applied on a test sample so that it conforms to the it's longitudinal axis, it will undergo a certain elongation depending on the amount of force applied. So that get geometric curve called stress-strain curve. The stress can be expressed in terms of the following relationship [56]:

$$\sigma = \frac{F}{A} \tag{2-8}$$

Where:

 σ : the stress and it's unit (N/m²).

F: the force applied on the sample to cause elongation along the axis of the sample and it's unit is (N).

A: is a cross-sectional area of the sample and it's unit (m^2) .

While strain denoted by ϵ and is expressed in the following relationship [57]:

$$\epsilon = \frac{\Delta L}{L_o} = \frac{L - L_o}{L_o} \tag{2-9}$$

Where:-

L: elongation (final length of the sample).

Lo: initial length of the sample (primary).

 ΔL : amount of change in length.

Strain is divided into two types (elastic strain and plastic strain), and the ratio between the stress applied on the material and the resulting strain in the elastic zone of the stress – strain curve. It is constant ratio called modulus of elasticity or young's modulus, which has the symbol (E). It is measured in units of (N/m^2) and is expressed in the following relationship [58]:

$$E = \frac{\sigma}{\epsilon} = \frac{P/A}{\Delta L/L_o} = \frac{PL_o}{\Delta LA} \qquad \dots \dots (2-1)$$

10)

2.8.2 Hardness

Hardness of material is generally measured by its resistance to local deformation or indentation by rigid body of a specified dimension under a specified condition. Hardness is a complex property and it is related to such mechanical properties of materials as modulus, strength, elasticity, and plasticity, even though the relation is not quite straightforward. In a given class of plastics and rubber materials, higher modulus and strength usually result in higher hardness. Hardness is a relative concept and it cannot be absolutely defined [59].

Hardness evaluation is commonly used as a simple and rapid means of production and quality control in the plastics and rubber industries in the processing sector. Hardness testing is particularly useful in getting an indication of degree of cure in thermosetting resin moulding and rubber vulcanization.

Durometer Hardness Test (Shore D) This test is described according to ASTM D 2240 (Figure (2-15)), for measuring the indentation hardness of materials ranging from soft rubbers to hard rubbers and plastics. These equipment measure the depth of indentation under load when hardness steel indenter is forced into a specimen by means of calibrated spring. The scale has a graduation from 0 to 100 hardness numbers and has an inverse relationship to indentation [59].

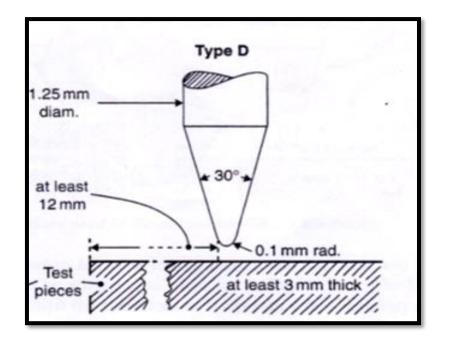


Figure (2-15): Indenters used in the durometer (shore hardness) [59].

2.9 Miscibility Tests

2.9.1 Infrared Spectroscopy

Infrared (IR) spectroscopy is a popular method for characterizing polymers. This technique is based on the vibrations of the atoms of a molecule. An infrared spectrum is obtained by passing infrared radiation through a sample and determining which fraction of the incident radiation is absorbed at a particular energy. The energy, at which any peak in an absorption spectrum appears, corresponds to the frequency of vibration of a part of the sample molecule. Most infrared spectroscopy is carried out by using Fourier-transform infrared (FTIR) spectrometers. This method is based on the interference of radiation between two beams to yield an interferogram, i.e. a signal produced as a function of the change of pathlength between the two beams. The two domains of distance and frequency are inter-convertible by the mathematical method of Fourier transformation. The basic components of an FTIR spectrometer are shown schematically in Figure (2-16). The radiation emerging from the source is passed through an interferometer to the sample before reaching a detector. Upon amplification of the signal, in which high-frequency contributions have been eliminated by a filter, the data are converted to a digital form by using an analog-to-digital converter and then transferred to the computer for Fourier transformation to take place.

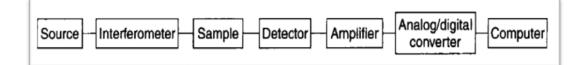


Figure (2-16): Schematic of a typical Fourier-transform infrared (FTIR) spectrometer [60].

The output from an infrared instrument is referred to as a spectrum. Inverse wavelength units (cm⁻¹) are used on the x-axis - this is known as the wavenumber scale. The y-axis may be represented by % transmittance, with 100% at the top of the spectrum. It is commonplace to have the choice of absorbance or transmittance as a measure of band intensity. The transmittance is traditionally used for spectral interpretation, while absorbance is used for quantitative work. The infrared spectrum can be divided into three regions, namely the farinfrared (< 400 cm⁻¹), the mid-infrared (400-4000 cm⁻¹) and the nearinfrared (4000- 13000 cm⁻¹). Most infrared applications employ the midinfrared region, although the near- and far-infrared regions can also provide specific information about materials. The near-infrared region consists largely of overtones or combination bands of fundamental modes appearing in the mid-infrared region. The far-infrared region can provide information regarding lattice vibrations. Spectrum interpretation is simplified by the fact that the bands that appear can be assigned to particular parts of the molecule, thus producing what are known as group frequencies [60]. The mid-infrared spectrum may be divided into the following four regions:

- X-H stretching region (4000-2500 cm⁻¹)
- triple-bond region (2500-2000 cm⁻¹)
- double-bond region (2000-1500 cm⁻¹)
- fingerprint region (1500-600 cm⁻¹)

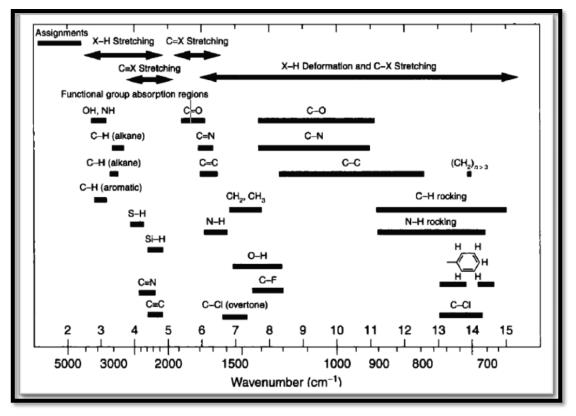


Figure (2-17): A typical correlation table for the infrared modes of polymers [60]

2.9.2 Differential Scanning Calorimetry (DSC)

A great deal of information about the glass-transtion temperature, crystalline melting point, degree of crystallinity, and reactions (degradation, cross-linking, etc.) of a polymer may be determined by thermal analysis. In this technique, small quantities of a sample and refrence material are loaded into identical cells. The refrence material (often powder aluminum) should have known specific-heat-versustemperature characteristics and should exhibit no thermal transformations in the temperature range of interest. The sample and refrence cells are then subjected to a ramp increase in temperature (often 10 °C/min). A control circuit apportions power to the sample and reference to keep them at exactly the same temperature [61].

The difference in power inputs is plotted versus temperature. When the sample undergoes an endothermic transition (such as crystalline melting), the difference in power will be increased because additional power is necessary to provide the heat of melting (approximately the inverse of the heat of crystallization) while maintaining equal temperatures[61].

 T_g , T_m and degree of crystallinity (D_c) can all be determined from a single DSC analysis. In addition, if degradation or cross-linking occurs at high temperature, the enthalpy of these reactions will be reflected as changes in differential power over the range of temperature over which these reaction occur[61].

2.9.3 Scanning Electron Microscope (SEM)

The scanning electron microscope is one of the most versatile instruments available for the examination and analysis of the micro structural characteristics of solid objects [62].

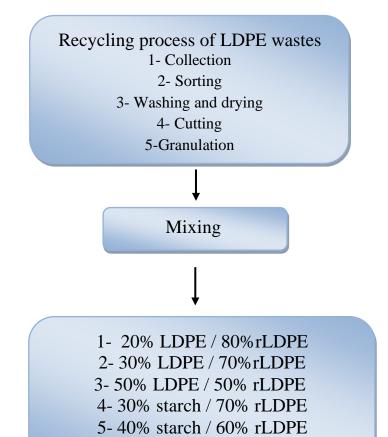
Samples suitable for SEM measurements include most solids which are stable under vacuum (metals, ceramics, polymers, minerals).

Samples must be less than 2 cm in diameter. Non-conducting samples are usually coated with a thin layer of carbon or gold in order to prevent electrostatic charging [63].

A major reason for the SEM's usefulness is the high resolution which can be obtained when bulk objects are examined; instrumental resolution on the order of 1-5 nm (10-50A) is now routinely quoted for commercial instruments. Specimens can be observed in high vacuum, low vacuum and in environmental SEM specimens can be observed in wet conditions. The basic components of the SEM are the lens system, the electron gun, the electron collector, the visual and photorecording cathode ray tubes (CRTs), and the associated electronics [62].

3.1 Introduction

In this chapter, a detailed description of the experimental methodology is presented, which includes, materials selection, sample preparation methods and detailed description of test instruments used. The flow chart (3-1) shows experimental part steps:



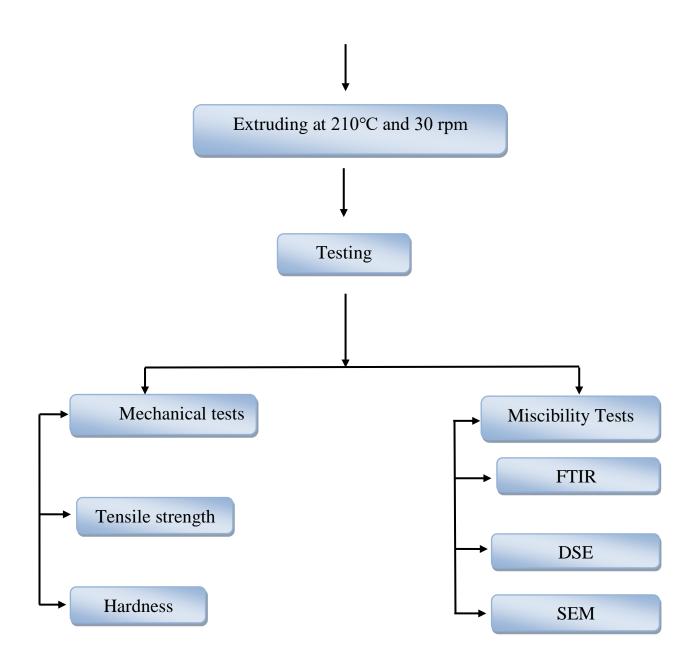


Figure (3-1): Flow chart of experimental part steps

3.2 The Materials Used:

Three materials are used in this study which included Low density polyethylene pellets, Recycle Low density polyethylene , and cornstarch

3.2.1 Low Density Polyethylene (LDPE):

LDPE was obtained from Sabic Petrochemical Company as pellets, Kingdom of Saudi Arabia . Some properties of LDPE shown in table 3.1

Properties	Unit	Value
Density	g/cm ³	0.923
Hardness	Shore D	46-51
Tensile strength	Мра	11
Melting point	°C	125

Table 3.1: Properties of LDPE

3.2.2 Recycled Low Density Polyethylene (rLDPE)

Recycled low density polyethylene (rLDPE) from agricultural covers waste was provided from Najwin factory _Bagdad as pellets, as shown in the figure (3.1).



Figure (3.1): Recycled LDPE.

3.2.3 coner starch:

Corn starch is a white, tasteless, odorless powder, used in food processing, papermaking, and the production of industrial adhesives; it is also a component of many cosmetics and oral pharmaceutical products. It has been used as a lubricant in surgical gloves.

Molecular formula	C12H22O11
Molecular Weight (g / mol)	342.30
Color	white
Density (g/ ml)	1.5

Table 3.2 Properties of starch.

3.3 Polymer blend preparation:

3.3.1 Weighting:

In the first mix rLDPE material pellets, LDPE pure pellets, were weighted according to selected ratios to prepare the batches for Extrusion.

In the second mix rLDPE material pellets, cornstarch powder, weighted according to selected ratios to prepare the batches for Extrusion.

3.3.2 Mixing:

. For optimum distribution in the first mix, rLDPE material, LDPE mixed together according to the ratios that shown in Table (3-3) in dry condition at room temperature by hand mixing in abowl.

For optimum distribution in the second mix, rLDPE material, cornstarch mixed together according to the ratios that shown After

melting and drying Table (3-3) in dry condition at room temperature by hand mixing in abowl

(rLDPE/LDPE)%	(80/20)	(70/30)	(50/50)
(rLDPE/starch)%	(70/30)	(60/40)	(50/50)

Table 3.3 Polymeric blends percentages

3.3.3 Extrusion:

Forming process was carried out using a twin-screw extruder model (SLJ-30A) which is available in laboratory of Materials Engineering collage /University of Babylon. The mixed materials were fed from a hopper on the screw. The materials then conveyed along the barrel where it is heated by conduction from the barrel heaters and shear due to its movement along the screw flight. The depth of the twin-screw channel is reduced along the length of the screw so as to compact the material. At the end of the extrusion, the melt passes through a die in the form of sheet. The extruder operation conditions were 35 rpm in the beginning then increased to 50 of screw speed. The temperatures used for zones 1 and 2 were 150 and 165°C respectively.



Figure (3.2): Co-rotating twin screw extruder machine.

3.3.4 Rolling and cutting:

After the material melts and out from the extruder, it passes through two co-rotating rollers that pressurize the material to give high molecular orientation and eliminate from any voids.

The result sheets was prepared for cutting and mechanical machining by using electrical saw with very soft teeth to ensure that samples would not vibrate. Cutting was done according to international standard specifications (ASTM) for hardness test as shown in table (3-4), while tensile test samples were cut by die press.

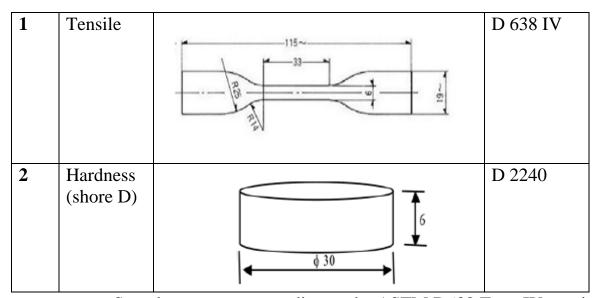
3.4. Tests:

3.4.1 Fourier transform infrared spectroscopy analysis:

Fourier transform infrared spectra technique is used for the characterization of the prepared samples. Instrument, type IR Affinity-1 (made in Japan), which is available in laboratory of Materials Engineering collage /University of Babylon, was used for this purpose. In order to measure a sample, calibrate the device using the KBr, and then prepare powder of the sample to be examined, and mixed with KBr (mixing ratio 99% KBr). The mixing process achieved thoroughly then pressed as tablet-shaped semi-transparent to the possibility of penetrating radiation.

3.4.2 Tensile test

No.	Property	Sample	ASTM
-----	----------	--------	------



Samples were cut according to the ASTM D638 Type IV specimen dimensions as shown in Table (3-7). The machine used for the testing of tensile properties is micro computer controlled electronic universal testing machine model (WDW-5E) China, located in the laboratory of Materials Engineering collage /University of Babylon. The test was conducted at applied load (5KN) and speed value (10mm/min) for all the sample at room temperature. Tensile stress was applied until the failure of the sample and stress-strain curve was obtained.

Table (3-4): Sample test dimension

3.4.3 Hardness test:

Shore D hardness device model (TH 210 FJ) made in Germany, which is available in laboratory of Materials Engineering collage /University of Babylon. Cylindrical samples were prepared according to the ASTM D 2240 as shown in Table (3-7). Shore instrument is similar to compass containing needle placed in a position perpendicular to the sample and it takes waiting (0.5 min) to read the value and to have some accuracy an average of three readings have to be taken in different locations and at different points for each sample .

3.4.3 Scanning electron microscopy test

Analytical Scanning Electron Microscope (SEM), model (INSPECT S50) which found in located in the laboratory of Materials Engineering collage /University of Babylon. The sample used in the testing was cut into small pieces (1x1 cm) to fit into the device. To obtain good electric conductivity, all samples were first coated with thin layer of gold has been made from the surface along the edge using sputtering device.

3.4.4 Differential scanning calorimetry test

Differential scanning calorimetry measurements (DSC) measurement were carried out by using DSC i-series (CW-05G) instrument under inert gas atmosphere which located in the laboratory of Materials Engineering collage /University of Babylon. The prepared samples with weight of $(8-10)\pm 0.5$ mg was mounted in aluminum pans and heats up from 25 to 250 C° with a heating rate of 10 C°/min.

4.1 Introduction

This chapter includes all the experimental results that are obtained from the mechanical and physical tests for the polymer blends under investigation.

The experimental tests and the results of blends systems were used to investigate, the mechanical properties including (tensile strength, modulus and hardness) and physical properties including (Fourier transforms spectroscopy (FTIR) and differential scanning calorimeter DSC) and finally the most important morphology for polymer blend were performed by using Scanning electron microscopy (SEM).

In this chapter, the results of all samples that have been selected for polymer blend systems are presented:

- rLDPE/LDPE
- rLDPE/cornstarch

In the current chapter, the results that have been obtained from the tests mentioned in chapter three are discussed.

4.2 FTIR analysis result

To prove that the mechanical recycling process causes deterioration of polymer properties, spectral analysis was used to determine the percentage and amount of recycled polyethylene degradation relative to pure polyethylene. Carbonyl formation is an indication of increased degradation and degradation of polymers and their oxidation. Pure LDPE and rLDPE were examined using FTIR spectroscopy. Determination of the carbonyl index at the values of the range 1715 cm⁻¹ based on a relative table. There was no degradation in pure LDPE, while the carbonyl index of recycled polyethylene showed a certain amount of polymer degradation, as peaks appeared in the range of 1715-1740 cm⁻¹ due to the carbonyl group as well at 1640 cm⁻¹ due to weak bonds and bonding[72].

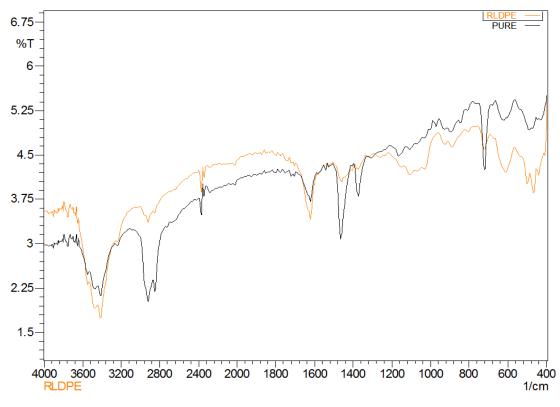


Figure (4-1): FTIR spectrum for LDPE, rLDPE.

It also appeared in the test FTIR for LDPE shows many bands such as the bands at 2939 cm-1 and 2850 cm-1 for –CH2- stretching, bands at 1463 cm-1 for CH2 bending and the band at 721 cm-1 for CH2 rocking.

Table (4-1): The absorption bands of IR spectrum characteristic of LDPE and rLDPE.

Type of bond	LDPE standard[83]	rLDPE
CH ₂ stretching	2919 2851	2939 2850
CH ₂ bending	1473	1463
CH ₂ rocking	720-731	721

4.3 Tensile test

In the first mixture, Figure (1-4) shows the effect of adding pure LDPE on the tensile strength of rLDPE with and without additives The tensile strength of the rLDPE mixture increases with the increase in the content of pure LDPE[84].

Where, at the percentage of addition (20%-80%) and (30%-70%), its tensile strength may increase than that of rLDPE without addition. This increase is due to the interfacial adhesion strength and the ability of LDPE to improve the chain strength of rLDPE. The addition ratio (50-50%) showed a slight decrease in tensile strength due to the increase in the amount of the additive and a relative phase separation between them.

From the above result, the best additive that improves the tensile strength of the rLDPE/LDPE mixture is the addition ratio of 20%-80%) and also this confirms that the mixture is compatible[70].

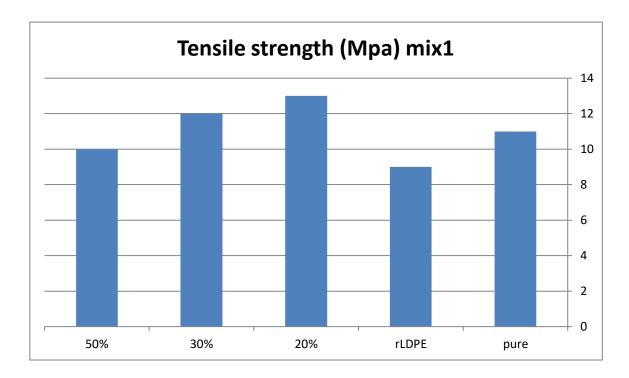


Figure (4-2): LLDPE amount effect on the tensile strength of rLDPE with and without additives.

In the second mixture, Figure (4-2) shows the effect of adding corn starch on the tensile strength of rLDPE with and without additives.

When adding corn starch by (30%-70%), the tensil strength of rLDPE is increased. This increase is due to the formation of cross-links and the. Ability of starch to enhance dispersion and improve chain strength, at the percentage of addition (40%-60%), the tensile strength decreases .This decrease in tensile strength is due to the weak interfacial adhesion between corn starch and rLDPE with different electrodes, which causes poor stress transfer between the matrix and the additive rLDPE at a high percentage of cornstarch.

At the percentage of addition (50%-50%), the tensile strength decreases by a greater percentage. This decrease is due to the weak interfacial adhesion between starch and rLDPE and the formation of larger starch agglomerates that lead to a decrease in tensile strength.

From the above result, the best additive that improves the tensile strength of the rLDPE/Starch mixture is the addition ratio, (30%-70%) and this mixture is not compatible[78].

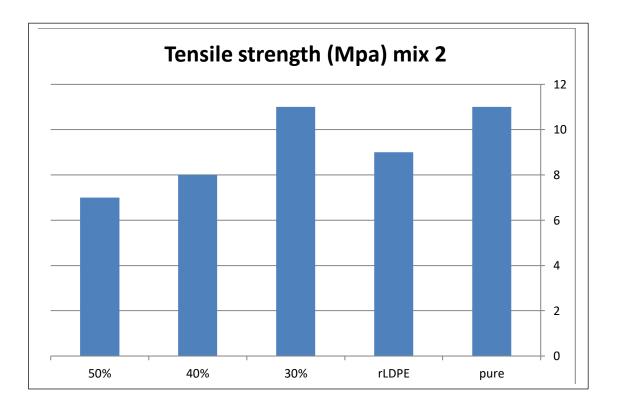


Figure (4-3): corn starch amount effect on the tensile strength of rLDPE with and without additives.

It must be mentioned here that the modulus of elasticity in the rLDPE/ starch mixture increased. The modulus of elasticity increased at the rate of addition of 30% as shown in Figure (4_4) and at 40% and 50% the modulus of elasticity decreased significantly. From the above result, the best modulus of elasticity for this mixture The incompatible is the ratio of 30% starch added to the matrix

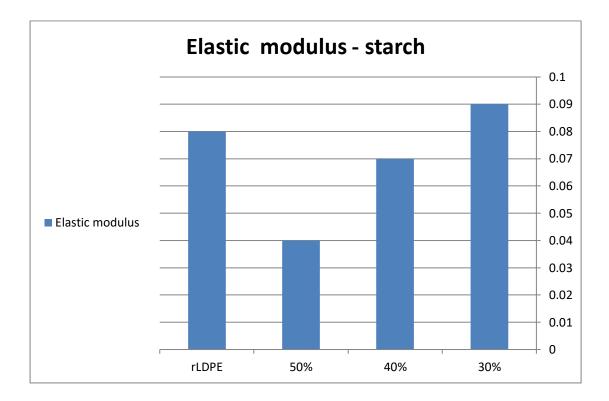


Figure (4-4): variation of Elastic modulus with pva amount LDPE/PVA blend with and without additives

4.4 Hardness test result

It is known that the hardness depends on the penetration resistance on the outer surface, and there are different ways to represent the hardness index in the first mixture. Figure (4_5) shows the hardness of the rLDPE/LDPE mixture with and without additives. The hardness increased at the addition rate of 20% because the matrix and the additive are compatible and from one type of Polymers, either at the percentage of addition 30% and 40%, the hardness decreased, and this indicates a separation in the phase by a small percentage, as the hardness decreased in the percentage of 40% by 1.3, and in the percentage of 50% it decreased by (1.9), and this means that the mixture is compatible and miscible with the crystalline phase. In the second mixture, Figure (4-6) shows the hardness of the rLDPE/STARCH mixture with and without additives. At the 30% addition rate, the hardness increased, but the hardness decreased at 40% and 50%. This decrease is due to phase separation between corn starch and recycled low-density polyethylene, in addition to the effects of agglomeration, which weakens the bonds between the rLDPE chains[87].

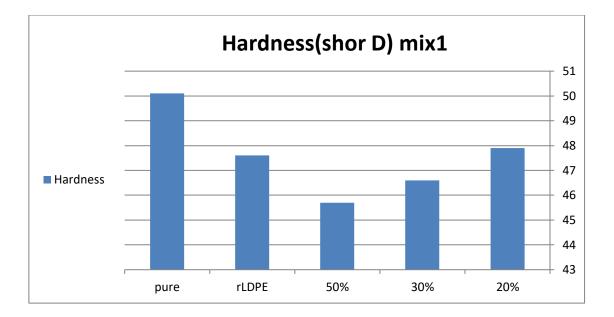


Figure (4-5): Shor D hardness variation with LDPE pure content in rLDPE/LDPE blend with and without additives.

In the second mixture, Figure (4-6) shows the hardness of the rLDPE/STARCH mixture with and without additives. At the 30% addition rate, the hardness increased, but the hardness decreased at 40% and 50%. This decrease is due to phase separation between corn starch and recycled low-density polyethylene, in addition to the effects of agglomeration, which weakens the bonds between the rLDPE chains[77].

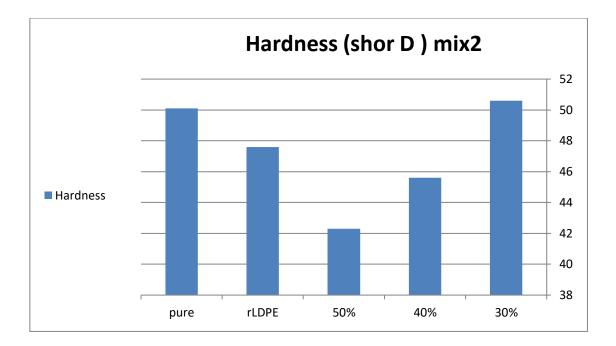


Figure (4-6): Shor D hardness variation with cornstarch content in rLDPE/starch blend with and without additives..

4.5 Scanning electron microscopy (SEM)

Figure (4-7) shows the scanning microscopic images of one sample mixed by the double screw extruder of the rLDPE/LDPE mixture at the ratio of addition (50%-50%). The surface is somewhat smooth and there are no holes and lumps, and this is because the mixture is of the same type of polymers[80].

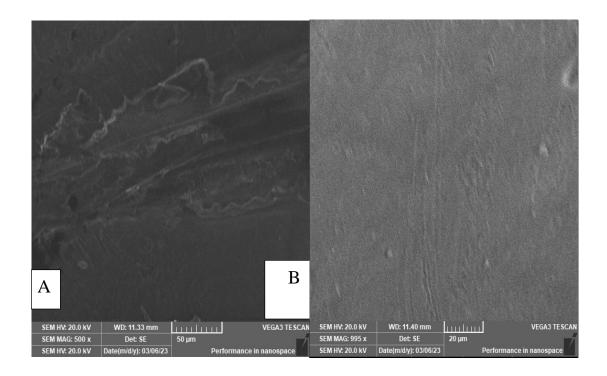


Figure (4-7): SEM images at 50 and 20 micrometer for: rLDPE /LDPE 50%-50% (A, B)

Figure (4-8) shows the scanning microscopic images of one sample mixed by the double screw extruder of the rLDPE/Starch mixture. At the addition ratio, large starch agglomerates appear due to the incompatibility of the mixture and the occurrence of phase separation between the faces of the mixture[81].

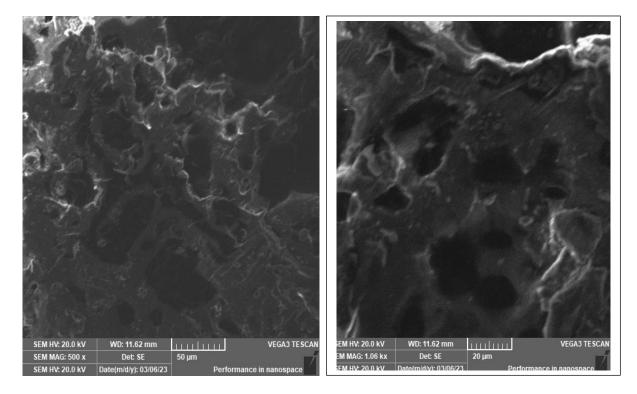


Figure (4-8): SEM images at 50 and 10 micrometer for: rLDPE/Cornstarch 50%-50% .

4.6 Differential scanning calorimetry DSC

When the LDPE polymer is recycled, it loses 35% of its properties, as its melting point and the length of its internal chains decrease, it becomes less dense, and the movement of its hydrocarbon chain increases. In the first mixture, DSC was used to analyze the thermal properties of rLDPE and the effect of adding percentages of pure LDPE on the melting point of the first with and without additives, where the melting point of LDPE is taken as an indicator of the improvement of the mixture.

The melting point of LDPE is 125.14 °C and the melting point of rLDPE is 113.10°C For the rLDPE/LDPE mixture at the addition ratios of 20%, 30% and 50%, the melting point is[86]:

Table (4-2): DSC data for first blends with and without additives

LDPE Tm (oC)	LDPE Tm (oC)	rLDPE/LDPE	rLDPE/LDPE	rLDPE/LDPE
(Pure)	(Recycle)	(20%-80%)	(30%-70%)	(50%-50%)
125.14	113.10	122.52	116.51	113.31

From the above result, the best addition ratio is the addition ratio of 20% of LDPE pure to the 80% matrix which is rLDPE.

In the second mixture, which is rLDPE/cornstarch, DSC was used

To analyze the thermal properties of rLDPE and the effect of adding percentages of corn starch on the melting point of the first with and without additives, where the melting point of pure LDPE is taken as an indicator of improving the properties of the mixture. The melting point of LDPE is 125.14 °C and the melting point of rLDPE is 113.10°C

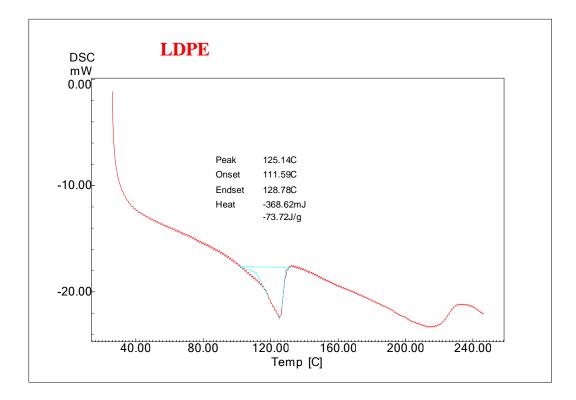
For the rLDPE/LDPE mixture at the addition ratios of 30%, 40% and 50%, the melting point is[85]:

Table (4-3): DSC data for second blends with and without additives

LDPE Tm (oC)	LDPE Tm (oC)	rLDPE/starch	rLDPE/starch	rLDPE/starch
(Pure)	(Recycle)	(30%-70%)	(40%-60%)	(50%-50%)

125.14	113.10	122.5	122.86	123.73

From the above result, the best addition ratio to improve the melting point is the ratio of 50% of corn starch to 50% of the matrix that is rLDPE.



rLDPE

20%LDPE 80% rLDPE

30%LDPE 70% rLDPE

50%LDPE /50% rLDPE

30% starch70%rLDPE

40% starch/60%rLDPE

50% starch/50%rLDPE



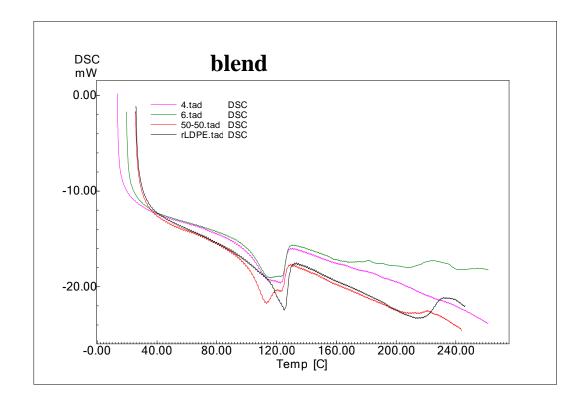


Figure (4-9): DSC curves for pure polymers (LDPE, rLDPE), and The proportions of the first and second mixture

5.1 Conclusions

From this work, the following conclusions can be summerized:

- FTIR results show degradation of recycled low-density polyethylene and an increase in oxidation by carbonyl (C-O) formation, and the bands prove that the material used is LDPE based on the standard bands.
- 2) In the first mixture, the pure 20% addition ratio improved the tensile properties such as tensile strength and modulus of elasticity, where they were the best percentages, while in the second mixture, the best percentage in which the tensile properties improved was the 30% addition of corn starch.
- 3) The hardness increases in the first mixture in the percentage of addition 20% of pure, and in the second mixture it increases in the percentage of addition of 30 of cornstarch.
- 4) The SEM results shows that the rLDPE/LDPE mixture was compatible, while the rLDPE/Cornstarch mixture was an incompatible mixture..
- 5) DSC thermographs show the amount of melting point improvement for all proportions of mixtures.

5.2 Recommendation

From the present study, the following recommendation can be helpful for further studies:

- 1) Using different mixing other type of recyclable polymers by mechanical method such as rHDPE/HDPE or rPET/PET.
- 2) Addition of a compatibility agent to the rLDPE/Cornstarch mixture such as EPDM agent to make the mixture compatible.
- Work to reduce the percentage of addition to the mixture of rLDPE/Cornstarch for 5%, 10%, 15%, by weight.

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الخلاصة

يركز هذا العمل على تمكين معامل الاكياس البلاستيكية من استخدام مخلفاتها مرة اخرى في الانتاج عند إعادة تدوير بوليمر البولي أثلين المنخفض الكثافة يفقد 35% من خواصه الاساسية، حيث تقل درجة انصهاره وطول سلاسله الداخلية، ويصبح أقل كثافة، وتزداد حركة سلسلته الهيدر وكربونية تتضمن هذة الدراسة تحسين الخصائص الميكانيكية والانسيابية للبولي إيثلين المخفض الكثافة المعاد تدويره. حيث تمت اضافة حبيبات من البولي اثيلين المنخفض الكثافة النقي بنسب مختلفة الى الحبيبات المعاد تدوير ها للنوع نفسة وكذلك اضافة نشا الذرة الية ايضا بنسب مختلفة ودراسة تأثير هذه الاضافة على الخواص الفيزيائية والميكانيكية لتطبيق انتاج الاكياس البلاستيكية.

في المزيج الأول تم تحضير خليط من حبيبات البولي اثيلين المنخفض الكثافة النقي (LDPE) مع حبيبات البولي اثيلين المنخفض الكثافة المعاد (LDPE) مع حبيبات البولي اثيلين المنخفض الكثافة المعاد (LDPE) مع حبيبات البولي ، 00% ، 50% أما في المزيج الثاني تم تحضير خليط من حبيبات (rLDPE) مع بوليمر طبيعي هو نشا الذرة بنسب محددة هي 30% ، 40% ، 50% ، 50% ، 50% بالوزن من النشا وقد استخدم (rLDPE) كمادة اساس ، المادة الأعلى نسبة واستخدم (LDPE) النقي ونشا الذرة لتحسين خصائص (rLDPE) .

أجريت عدة اختبارات لتوصيف وتمبيز خصائص المزيج مثل مقاومة الشد والصلادة والفحص بالمجهر الالكتروني الماسح ومطياف الاشعة تحت الحمراء والتحليل الحراري التفاضلي للخلائط المحضرة .

اضهر اختبار الشد والصلادة ازدياد مقاومة الشد ومقاومة الصلادة عن مقاومة DSC) بدون إضافة تم اجراء تقيم للخصائص الحرارية والريولوجية باستخدام

للخلطات ومقارنتها مع LDPE النقي . تشير قياسات DSC الى ان خلائط LDPE المحات ومقارنتها مع LDPE النقي زيادة درجة الانصهار rLDPE النقي زيادة درجة الانصهار

تظهر نتائج DSC ان البولي اثيلين المنخفض الكثافة النقي سيعزز التدفق الطبيعي للمعاد ويحسن حركة اجزاء السلسلة الداخلية وبالتالي قد تحسنت الخصائص الانسيابية وزادة قابلية التشكيل والمعالجة مقارنةً بانسيابية وقابلية التشكيل للمعاد عندما يكون بلا إضافة .

في المزيج الثاني أدت إضافة النشا إلى زيادة قوة الشد اظهرت نتائج الفحص المجهري SEM ان التفاعل البيني كان ضعيفا عند نسب 40% و50% نشا وكانت الخلطات المحضرة باستخدام (rLDPE) المعاد تدويره مع النشا بنفس قوة الشد (LDPE) النقي حيث أن النشا كان العامل الرئيسي الذي أثر على خصائص المزيج. ان استخدام هذا النوع من الخلط مع حبيبات البوليمر المعاد تدويرها يعطينا ميزتان اولهما

. تحسين خصائص الانسيابية وقوة الشد للبوليمر المعاد وايضا ان نشا الذرة هو بوليمر قابل للتحلل البيولوجي عند خلطة مع البوليمرات المعاد تدوير ها الخاملة مثل TLDPE يعمل على جعلة قابلا للتحلل حيث انه اذا كان المكون قابل للتحلل البيولوجي موجودا بكميات مناسبة تقوم البكتريا بإزالة هذا المكون وبالتالي يتفكك البلاستيك او الفيلم الذي يحتوي على المكونات الخاملة المتبقية وبالتالي يختفي بالإضافة الى ان النشا سيكون بديلا لاستخدام الحبيبات النقية في طريقة التحسين الاولى وبالتالي تنخفض التكلفة على المعامل التي تستخدم الحبيبات المعادة لكون النشا ارخص سعرا من الحبيبات النقية .

في هذه العمل لقد حصلنا على خصائص مرغوب بها في المعامل الانتاجية وتعتبر هذه النتائج بيانات مشجعة من وجهة نظر التصنيع.

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