SURFACE MODIFICATION OF LINEAR LOW-DENSITY POLYETHYLENE BY GRAFT COPOLYMERIZATION WITH MALEIC ANHYDRIDE AND ITS BLEND WITH POLYAMIDE 6

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ABSTRACT: *The Linear Lower Density Polyethylene surfacing characters were changed by Maleic Anhydride grafts by adaptation of counter-rotation of both screws extrudes in the occurrence of Benzoyl Peroxide. The linear low-density polyethylene PA 6 and linear low-density polyethylene-g-MAH/ PA 6 blend were made by counters rotations both screws Extruders. The blends were categorized by Differential Scanning Calorimeter, Scanning Electron Microscopy, capillary rheometer, and Fourier Transform Infrared Spectroscopy. Factors influencing grafting degree and rheological behavior of the (LLDPE) were evaluated, such as variations in an initiator and Maleic Anhydride concentrations and temperature. The presence of Maleic anhydrides onto (LLDPE) backbones were treated by Fourier Transform Infrared Spectroscopy. From a mechanical analysis study, it is found that the tensile stresses of the blends rise with rising Polyamide-6 contents. The co-continuous structure of LLDPE/ PA 6 blends has been shown by scanning electron microscopy (SEM) results identifying the best interfacial adhesions and improvement of mechanical characteristics. In this paper, we used Linear Low-Density Polyethylene as a continual phase, but the Polyamide as for dispersing phases. Furthermore, the addition of the home synthesized melted Linear Low-Density Polyethylene presented the chances to be adopted for the compatibilizations of Linear Low-Density Polyethylene or PA6 blends.*

Keywords: Grafting copolymers, Infrared spectroscopy; Polyethylene; Morphology; Viscosity;

1. . INTRODUCTION

The developing of multi phases plastic commonly present the track to combine various characters of only one of the polymer component which may be obtained from the mixture of two or greater numbers of polymers leads to forming the polymer blend. The chances of compound Linear Low-Density Polyethylene with the polymerized polar like Polyamide has resulted in the development of the occurrence of Linear Low-Density Polyethylene or the blend of polymers [1-4]. The survey of literature in this respect reveals some significant characteristics of polymer blends. The starch and P.E are not the homogenous mixtures due to their variances in polarities; the starch and P.E are the hydrophilic and hydrophobic respectively. It is quite important to add the compatibilizers for inducing the interactions in the reduction and phases tensions of the interfaces which raise the adhesions in the blend component and promote reducing the sizes of the dispersing phases that make it homogenous [5-11]. The compatibility may be promoted via grafts or blocks of the polymers. [12]. In general, the methods used for the functionalization consist of grafting Maleic Anhydride [13-17], but the polymerization in the molten state has been widely adopted that is commonly performed in the occurrence of the Radical Initiators [18]. The methods for Polyethylene the prominent side's reactions are the crossed linkages and for Polypropylene the method of reactions is scission chains [19, 20]. The cross-linking and chain scission in ethylene propylene rubber can be significant and leads to wider distributions of chain structures from greater degrading short chain to lesser crossed linkage chain and a plateau value of gel contents [21-22]. Interfacial characters are greatly improved with the rising of moiety polar and the other reactions can surely alter the religious natures and proceeding characters with mechanical characteristics of the function polymers [23-25]. Furthermore, some studies show the surface modification of LLDPE through grafting nonsaturated fertilizers like Maleic Anhydride and Acrylic Acid has achieved great deals with the attention of the last few decades [26-28]. Various researchers attempted to prepare HDPE–g-MAH Copolymers adopting various methods [29-31]. In this study, the graft polymers were prepared by greater degrees of grafts by counter-rotation with screws extrudes, used for the compatibilization of LLDPE/PA6 blends. In this work, we first used Linear Low-Density Polyethylene as continual phases and PA6 as dispersing phases to improve the properties of LLDPE, while in literature polyamide 6 was used as a continuous phase. The variety of thermoplastic by counter-rotation both extrude techniques could be achieved to produce the latest material which is cheaper and fast to achieve the latest and important commercial polymer. Counter-rotating both extrudes acts as a consistent flow reactor for a polymer that plays a significant role in raising and producing higher performance thermoplastics [32].

In this experimental work, the broad methods of the functionalization of Linear Low-Density Polyethylene ave been exploited. The counter-rotating both extrudes is employed to cram the grafting of Maleic Anhydride onto the Linear Low-Density Polyethylene chain. FT-IR and scanning electron microscopy techniques are adopted to evaluate the reaction of grafting; grafting degrees and other objectives of this work are to examine the combining effects of Maleic Anhydride, Benzoyl Peroxide and temperatures on graft degrees, melts viscous and tensile strengths of reactive products. The results of the morphological, thermal, and rheological characterization were compared with those of   LLDPE.

2. EXPERIMENTAL
2.1. Materials
The materials employed in this experimental work were

commercial Linear Low-Density Polyethylene granule (LLDPE) 5000S (Sinopec Beijing Yansan Petrochemicals company Ltd, Beijing, China). It had a density of 0.95 grams/cm^3, melting point 120℃, and a melt flow index of 3.5 grams per ten minutes. Polyamide 6 was supplied by U.B.E industries Ltd Japan having density 1.12 gram per cm^3 and point of melting at 220 ^{o}C and M.F.I 220 ^{o}C, 2.16 kilogram is equal to 2 grams per ten minutes. The Benzoyl Peroxide (ninety-eight percent) was purchased from BDH Chemical Ltd Poole England and also Maleic anhydride obtained from BDH Chemical Ltd Poole England of reagent grades and used except more purifications. Reagent grade was Acetone and Xylene used as received in all experiments.

2.2. Grafting procedure

Counter-rotating twin-screw extruder (Brabender Plastogaph Germany, screw diameter: 30mm, length: 706mm) was employed for the functionalization of LLDPE. MAH and BPO were dissolved in Acetone and then mix with LLDPE granule before extruding. After vaporizing the Acetone, the BPO and maleic anhydride adhere to the LLDPE granule consistently. The processing temperatures of the extruders from the feeding zones to die varies at 150/170/170/165℃ and the speeds of screws were kept constant at sixty revolutions per minute and the strips of polymers comes out of the extruders were quenched in the colder water baths.

2.3. Blending

Polyamide 6/ Linear Low-Density Polyethylene grafts Maleic Anhydride and these six pellets were dried in the vacuumed oven at 110 °C for 24h before processing. Counter rotation both screw extrude was used for the preparation of blends of LLDPE/PA 6 and LLDPE –g –MAH /PA 6 with the ingredient proportion of seventy: thirty wt/wt %. The processing temperature of extrudes varies at 150 over 220 ^{o}C and 220 over 215 ^{o}C. The pure PA 6 has been passed from extrudes to compare the material with a similar thermomechanical history and the strips from extrudes were quenched in cold water and granules longer about four millimeters were separated.

2.4. FT-IR analysis

The unreacted MAH was removed by dissolving functioned products in boiling Xylene followed by precipitating of solvent parts in Acetone. The grafting polymers were separated by the filtration, washes with Acetone, and after dries at 60oC in the oven to reaches the constant weigh. The formic acid and hotter Xylene were used as a solvent to purify the LLDPE-g-MAH/PA 6 blend samples. The blends specimens were treated with insoluble fractions of PA 6 and Formic Acid and separate by configuring the suspension, washes with purify methanol solvent, dries and weighs and the dry specimens were treated more with hotter Xylene to obtains the Xylene insolubility which are washes with Acetone and dries at sixty degree centigrade in oven and Xylene solubility fractions.

The purified products of MAH-g-LLDPE and obtained blends were compressed and molds in thinner films with the thinness of 0.05 millimeter for Fourier Transform Infrared characters using a (Thermo Nicolet Avatar 320 spectrophotometer USA) using Attenuated Total Reflection (ATR) technique. Grafting percentage was estimated in association with higher responsibility for asymmetry and symmetry stretches movements of C=O in Maleic Anhydride at the number of wave 1737.9 cm^{-1} in comparisons to –CH$_2$ rock movements in LLDPE at 719.6 cm^{-1}, that adopted in the internal references for the normalizations of the spectrum [16,33-35].

2.5. Morphological analysis

SEM micrographs were acquired by using the model 6380 of EO/JEOL, Japan scanning electron microscopy (SEM) and the specimens were fractures in the liquids of Nitrogen and the fractured surfaces were coated with thinner layers of the golds before scanning electron microscopy determination. The accelerating voltage was 10 kV.

2.6. Rheological characterization

The rheological behaviors of the graft copolymers were performed with Viscometer.

2.7. Thermal analysis

To evaluate thermal properties a METTLER TOLEDO instrument DSC 822^e Differential Scanning Calorimetric was used in the flows of Nitrogen, with heating rates of 10oC per minute from room temperature to 180℃, calibrates with Tin and Indium standards.
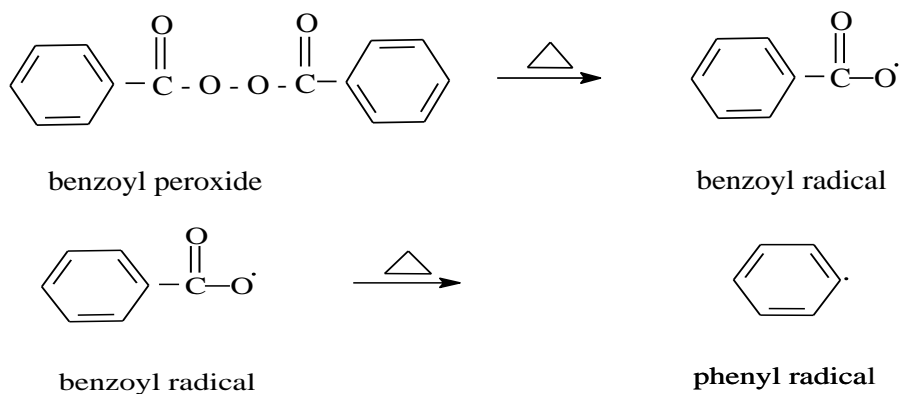
2.9. Tensile properties

Tensile measurements were determined on a computer-based material tested machine (Shenzhen Sans Model CMT4503 China) adopting the dumbbells samples with the standards of ASTM at a temperature of 20 ±3 ℃ with crossing heads rate of 50 mm per minute^{-1} and five tests of every data, were presented.
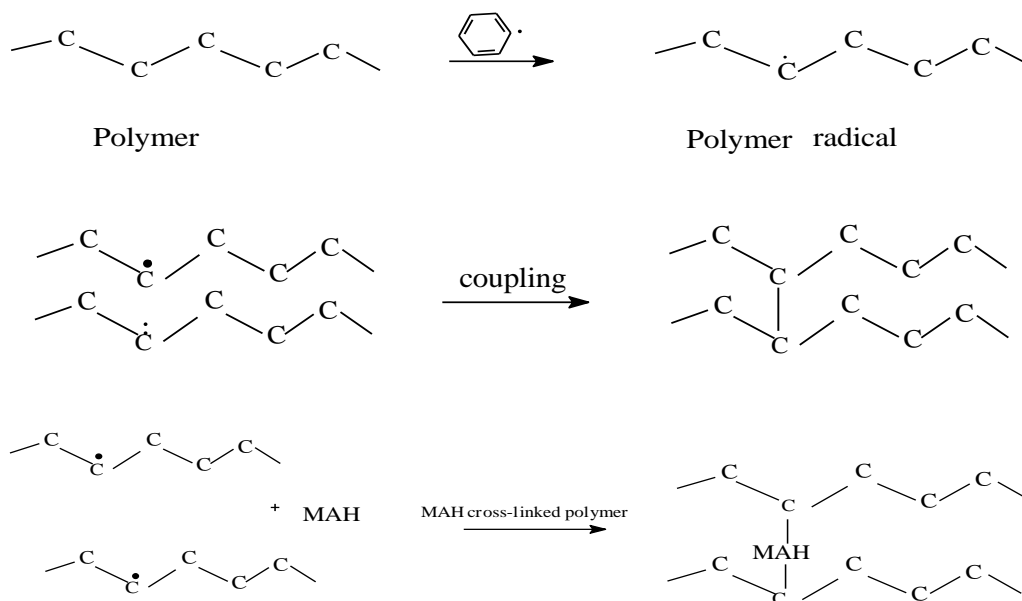
3. RESULTS AND DISCUSSION

The achievable method for the radically grafted copolymerization of linear low-density Polyethylene is given in schemes 1. The free radical made via initiators decompositions may eliminate atoms of the hydrogen from polymer backbones, results in forming of linear low-density Polyethylene macro radicals. The macro radicals face various possible paths, i.e. i) reacts with the fertilizers from expecting grafts products and or ii) undergoes radical combinations together to forming cross-linkage.
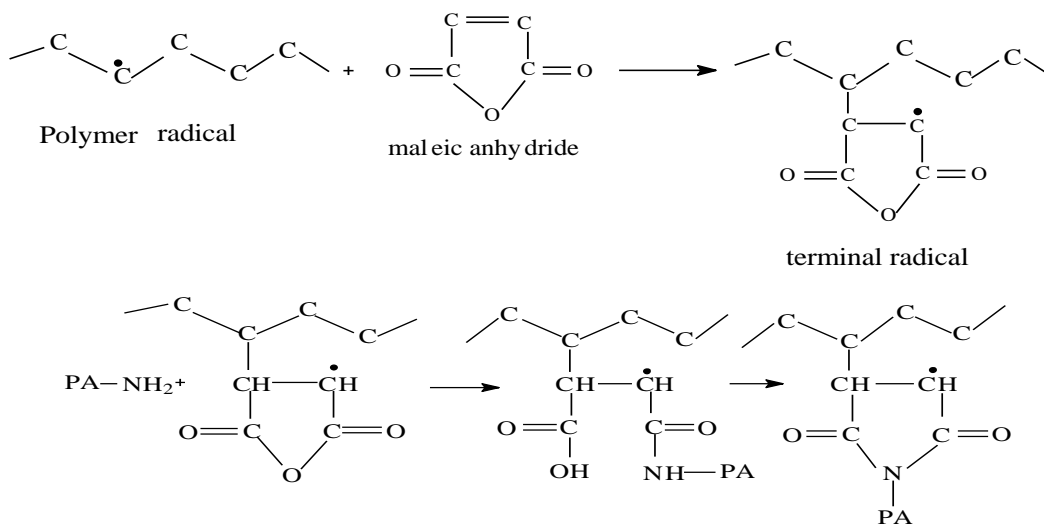
Peroxide decomposition:

Peroxide decomposition:

benzoyl peroxide   **benzoyl radical**

benzoyl radical   **phenyl radical**

Initiation

Polymer   Polymer radical

coupling

+ MAH   MAH cross-linked polymer

Propagation

Polymer radical   maleic anhydride   terminal radical

Scheme 1

FT-IR spectroscopy was employed to verify the presence of grafts reactions (Maleic Anhydride grafts on the chains of polyethylene) in the extrusions of blends. FTIR spectra of pure polyethylene (LLDPE), the purified product of Linear

Low-Density polyethylene-g-Maleic Anhydride, and Linear Low-Density polyethylene -g- Maleic Anhydride/ PA 6 specimens are depicted in Figure 1(a, b, c). Comparing of purifying Linear Low-Density polyethylene with graft

products (fig.1b), the peaks 1737.9 cm^{-1}, indicate the carbonyl bond (C=O) stretching vibration present in case of LLDPE -*g*-MAH has not been found in LLDPE(Fig.1a); thus confirming the grafting and was found to supports more observations of morphology. Band observes at 719 cm^{-1} are assigned to the rock movements of the –CH$_2$ bond in Linear Low-Density polyethylene. Furthermore the quantitative examination, it was observed that the number

of grafts Maleic Anhydride on the Linear Low-Density polyethylene chain was approximately 7.5 %. These results are in good agreement with related research and also higher characters of PA 6 absorptions were followed and shown in fig 1 (c).
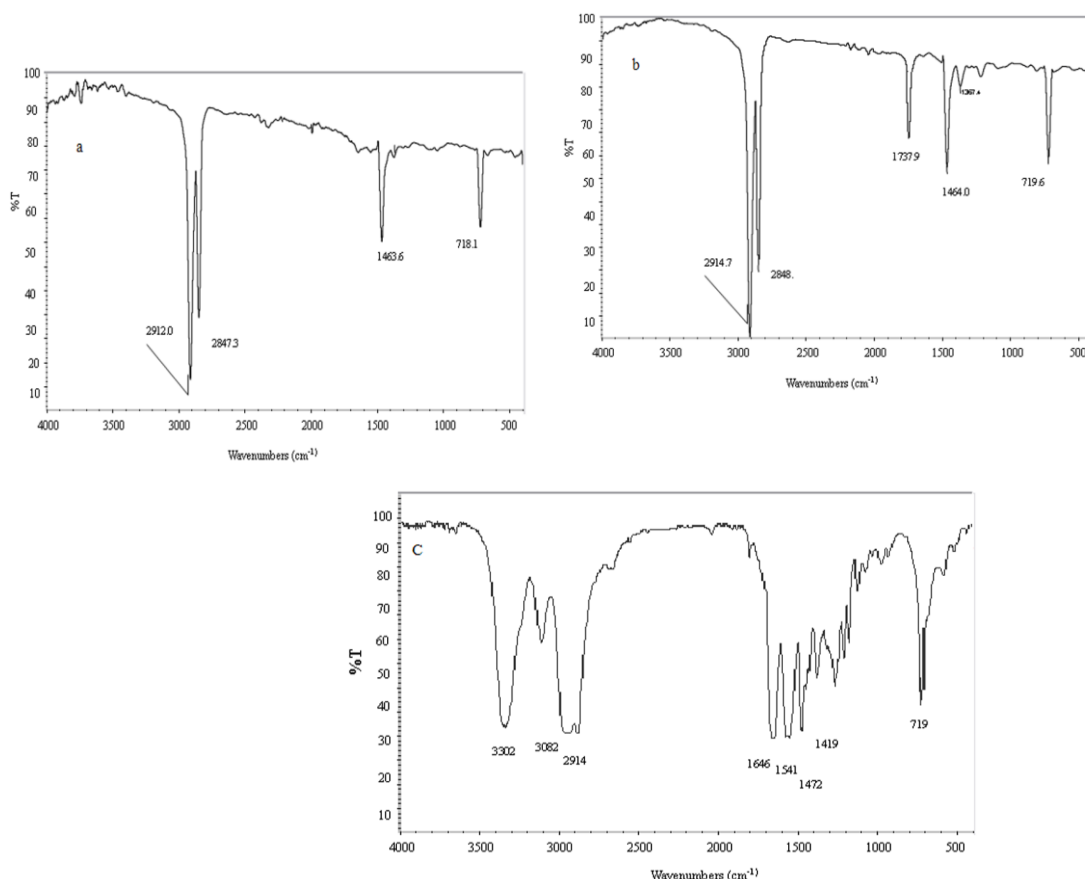
Fig 1 FT IR spectrum of (a) virgin LLDPE, (b) LLDPE-g MAH, and (c) LLDPE –g – Maleic Anhydride / PA 6

3.1. Morphology
Morphology of polymer blend, especially mechanical properties, plays a prominent role in the characteristics of the final products. The SEM micrographs of virgin LLDPE, modified LLDPE, LLDPE /PA6, and LLDPE -*g*- Maleic Anhydride/ PA 6 with the ratio of seventy thirty blends are displayed in Fig. 2(a-e). It can be seen from Fig. 2(a) in the absence of the functioned product the LLDPE particles with domain size are about 5µm and have the most complete smoothly surface. Furthermore Linear Low-Density polyethylene – *g*- Maleic Anhydride surface is too uneven and the shape of Maleic Anhydride pieces is irregular that indicates the Maleic Anhydride fertilizers were grafts on to Linear Low-Density polyethylene chains which shows in 10th fig. 2 (b). The interface between the PA6 domains and the LLDPE matrix is quite clear (Fig. 2 c). When 3phr BPO was added to the blend, in the study obtained the lower adhesions in the phase and greater dispersions of globalized domain sizes that show the lower

compatibilities in Linear Low-Density polyethylene and PA 6. This outcome implies the miscibility in the phase is improved to some extent at that BPO concentration, and the results are in good agreement with what has been reported for other LLDPE-g-MAH/PA6 [12]. Further, the addition of 0.46 phr BPO content into blends brings about a considerable improvement in compatibility (Fig. 2e), and we observed a co-continuous morphology. Those indicating the additions of 0.46 phr BPO reduce interfacial tensions that promoting fusions of various phases and there are possibilities to think the Linear Low-Density polyethylene-g- Maleic Anhydride locating himself at interfaces in PA6 and LLDPE. Therefore the blends exhibit a miscible structure.

3.2. Effect of Maleic anhydride concentration
The effects of MAH concentrations on the grafting degree of MAH onto LLDPE were determined and the outcomes are presented in Fig.3.
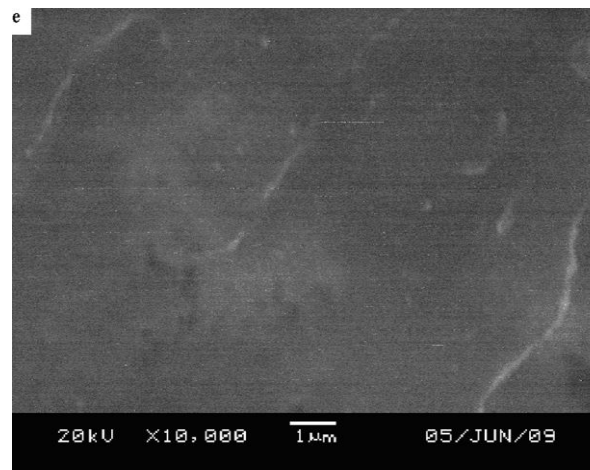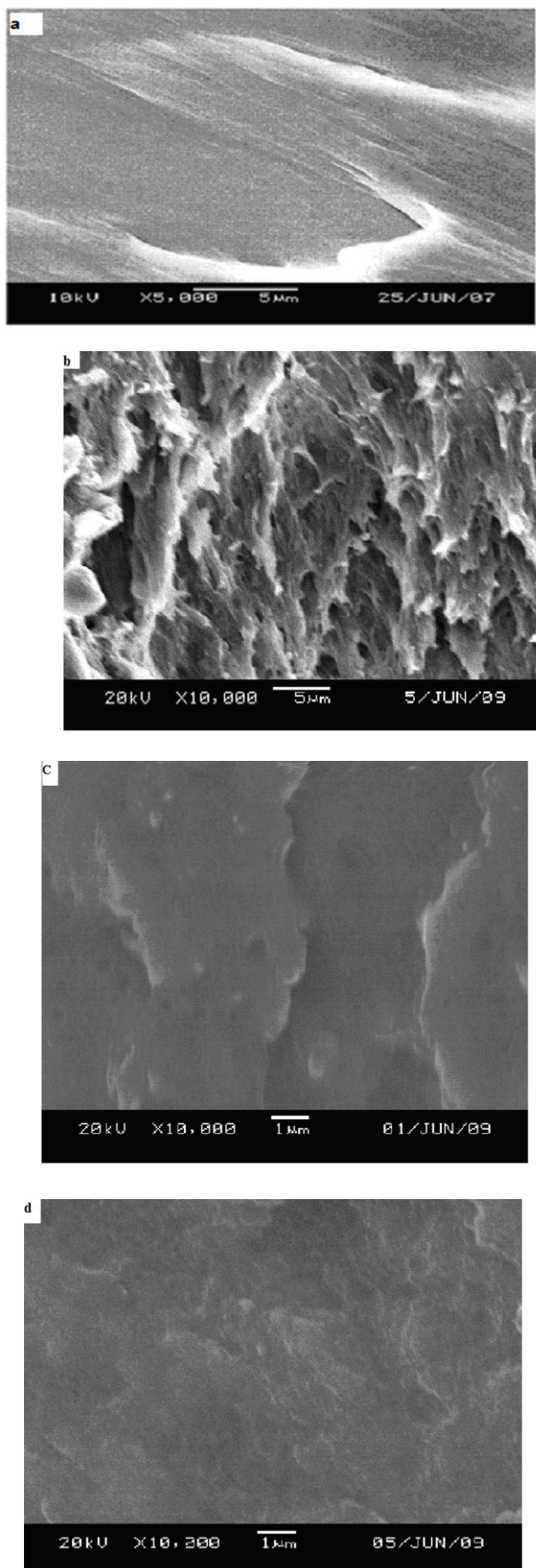
Fig. 2: Scanning Electron Microscopy snapshots of the Linear Low Density polyethylene/ PA6 /BPO /Maleic Anhydride blends: (a) 70/30/0/0, (b) 70/30/0.1/0.1, (c) 70/30/0.2/0.1, (d) 70/30/0.3/0.1, (e) 70/30/0.4/0.1

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LLDPE-g- Maleic Anhydride raises mostly 0.2phr and the rise is reduced   linear with rising Maleic Anhydride concentrations. The results are consistent with yang *et al* [37]. In this experimental work, it was observed that the degree grafts are raised significantly with raising the contents of Maleic Anhydride. Importantly the grafts degrees in the laboratories modified LLDPE are greater than the works done by others [23, 38]. Moreover, the weight of molecules (Mn) of graft LLDPE increases remarkably with a rise in Maleic Anhydride contents limits to 0.2phr and the %age of grafts Maleic Anhydride percent of grafts maleic anhydride levels off or decreases and further, contributing to rising the melting material viscosities [9].
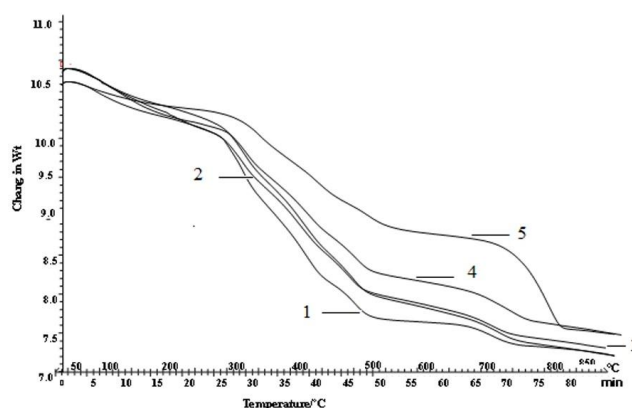

Fig.3: Effect of the MAH Concentration on Grafts Degree

The effect of Maleic Anhydride concentrations on the rheological behavior of functionalized LLDPE –g- Maleic Anhydride is shown in Fig. 4; with rising of Maleic Anhydride contents in the reaction mixtures when the melt viscosity of MAH grafted LLDPE is raised. As the MAH content increases the amount of monomer available for grafting/cross linking thereby raising the degree of graft and cross linking. Furthermore, according to Gaylord [30] the cross-linking reactions resulting that the generations of radicals site on the polymers backbones follows by couples. The presence of Maleic Anhydride raises the numbers of site and also increase the extents of cross linkage. In this experimental work the crossed links of resulting LLDPE -g – Maleic Anhydride distributes in two categories like coupling through one or more Maleic Anhydride fertilizers PE (MAH) n – PE and PE chain coupling directly (PE and PE) [38].
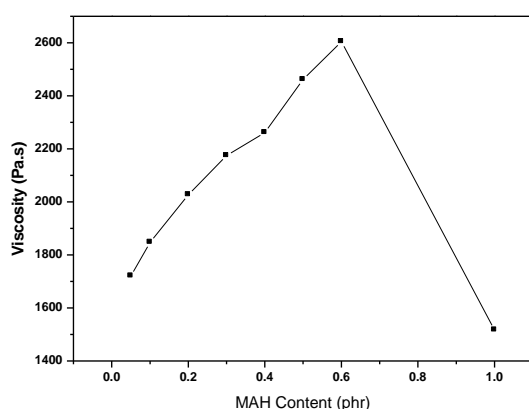

Fig. 4: Maleic Anhydride concentrations effects on the melting viscosity of Maleated LDPE
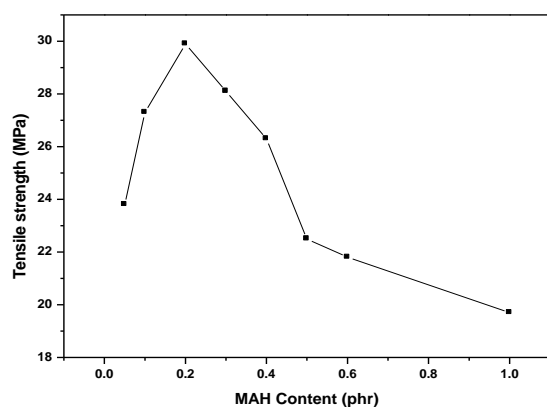

Fig.5 Effects of the MAH concentration on the tensile strength of LDPE – g – MAH / PA 6

The study results shown in fig. 5, which explains the MAH, have distinct effects on tensile strength of LLDPE – g – Maleic Anhydride / PA 6 weight proportions of seventy and thirty blends. It is observed from the results that, marked improvement was achieved in tensile strength limit of 0.2phr and then reduction with raising Maleic Anhydride contents. According to these results it is cleare that the adding of Maleic Anhydride gives the strength to the PA 6 and LLDPE inter facial adhesion.

3.3. Effect of benzoyl peroxide concentration

It is reported that the amount of BPO is an important factor affecting the rheological behavior of the MAH grafted LLDPE. Fig. 6 depicts the effect of BPO concentrations on rheological properties of Maleic Anhydride grafts LLDPE at regular Maleic Anhydride contents (0.2phr). The results showed that in the nonappearance of BPO, the grafts are not present as expected limit, the quantity of Maleic anhydride rise when the initiation concentrations were raised at the limits of 0. 46 phr; further increasing greater than the limits didn't leads to improve in the quantity of grafts maleic anhydride and cross linking side reactions increases with increase in BPA contents in the maleic anhydride grafts LLDPE to the higher extents that was reflected by the increasing in the melting viscosity [39].
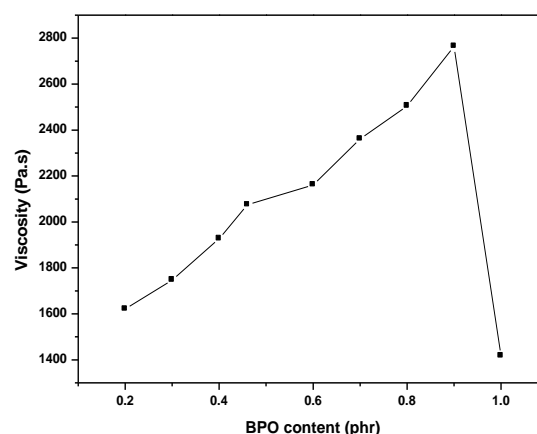

Fig. 6: Concentrations effects of BPO on the melt viscosity of maleated LDPE

Fig. 7 represents the influence of BPO concentrations on degrees of grafts. It is found that the degree of graft showed an increased tendency while increasing initiation concentrations at the regular maleic anhydride contents of 0.2phr resulting the rise in the grafts degrees with limits of 0.46phr, then decreased with raising the concentrations of BPO. The results show that the rising of contents of BPO have the greater free radicals generating by decomposition of Peroxides that enhance the formations of reaction sites on the polymer chains.
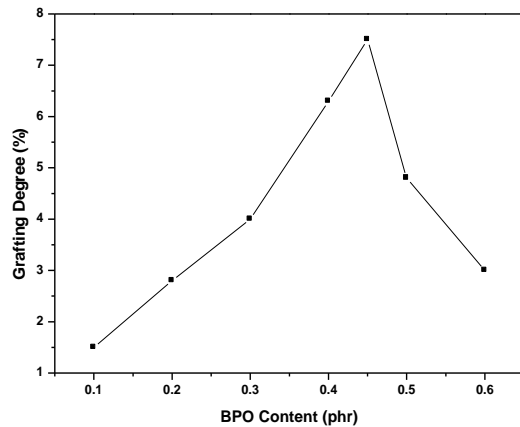
Fig. 7: BPO concentration effects on the grafts degrees

In addition, the weighs of the molecules of grafts LLDPE increased markedly with rising concentrations of BPO and after contributing to the remaining enhancements in the viscosity melting of the materials. It reveals that the reactions of chemicals for the grafts are mostly completes at 0.46phr when the degradation of thermals are rejected.

This section discusses the way in which the degrees of grafts of LLDPE–*g*–Maleic Anhydride affect the tensile strength linear low density polyethylene–g-Maleic Anhydride/ PA 6 blends. The results are depicted in Fig. 8 which was found from tensile strength data of the LLDPE -*g*-MAH/PA6 blend rise to 0.46phr, and drops constantly with rising concentrations of BPO. The rising trends of tensile strengths confirms inter facial adhesions were enhance by the reactions of the Polyamide with Maleic Anhydrides values.
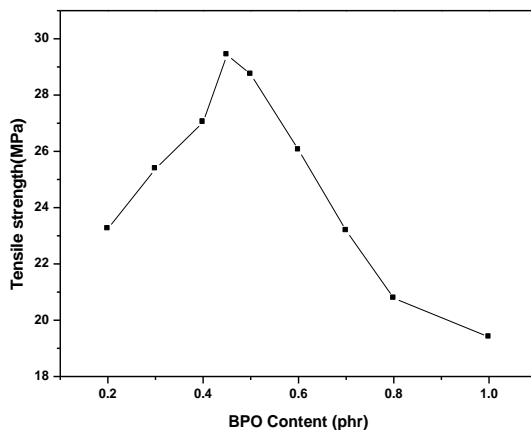

Fig.8 Concentration effects of PBO concentration on the tensile strengths of LDPE – g – MAH / PA 6

3.4 Temperature Effects

The influencing of temperature reactions was believed significant factor affects the microstructural change of LLDPE -*g*-MAH/PA6 blend. Obviously, by increasing the temperature, the proportion of the formation of free radical's specie was prepared, decomposing of the initiations. It can be seen that when LLDPE /BPO/MAH, 100phr/0.5phr/0.2phr ratios was fixed. The results are summarized in Fig 9. It is clear from the above discussion that, above 170℃ the reduction in levels of grafts are because of chain scissions of LLDPE at greater temperature reactions.
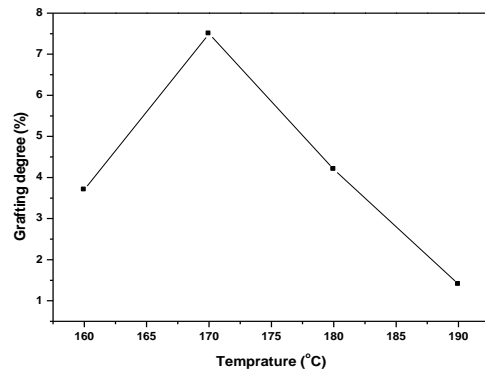

Fig. 9: Temperature effects on the graft degree

Fig.10 depicts the outcomes of temperatures depending on the rheological behavior of LLDPE-g-MAH. Melt viscosity of the grafted LLDPE samples were found rising with rising temperature and after decreasing sharply after 170℃ of temperature. The reduction in melting viscosity is due to generation of free radical species prepared quicker owns to the quicker decomposing of BPO that leading to the chain scission of LLDPE at higher temperature.
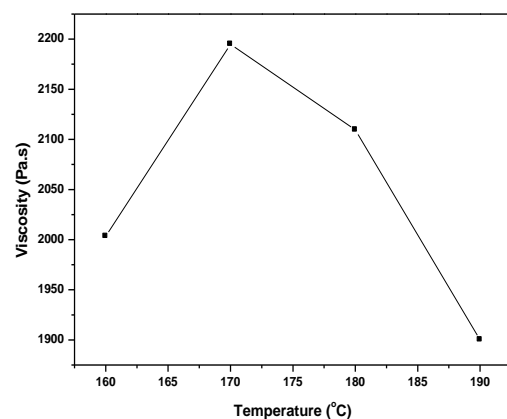

Fig. 10: Effect of temperature on the meltviscosity

3.5. Mechanical properties

The ability to resist breaking under load has been the most significant and widely measured characteristics of material used in the structural application. Fig. 11 shows the characters of tensile of LLDPE–g –Maleic Anhydride / PA 6 (weight percent of seventy to thirty) blends. It is calculated that the tensile properties Low LDPE – g – Maleic Anhydride / PA 6 blends were found to rise with rising temperature to 170℃; then decreased appreciably with increasing reaction temperatures. This phenomenon could be explained the strength tensile slowly rises as the grafts degrees rises to 170℃ and after reduces. After 170℃ the blends LLDPE -g-MAH/PA6 tensile strength was found to be decreased swiftly. It could be due to the degradation of LLDPE at high temperature. The outcomes demonstrate the best relation in the tensile strength and grafting degrees.
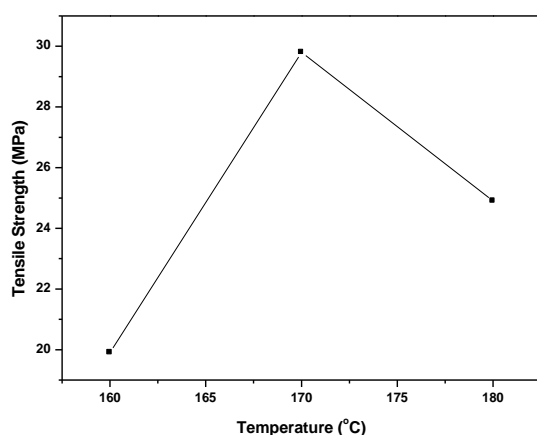
Fig.11: Tingture effects on the tensile strength of LDPE − g −

MAH / PA 63.6. Thermal analysis

To determine the crystallinity and melting behavior of the samples, DSC experiments were conducted. The DSC endothermic of pure linear low density polyethylene and linear low density polyethylene -*g*-MAH are revealed in Fig. 12 and it may be seen that the inclusion of polar functionality such as carbonyl group into nonpolar polymer changes its structure in comparison with pure LLDPE When increasing the grafting degree, the temperature of melts change in low temperatures and reveals that higher degree of grafts can induced various micro structures than of the specimen with lesser grafts degree. The results are obtained are similar to that of the results of Collar et al. Furthermore, crystallization and melting temperature of every graft specimen is lesser than the untainted HDPE. It means that graft the maleic anhydride against polyethylene's variations in structures, in comparing with basic LLDPE. DSC studies on maleated LLDPE with different MAH contents showing melting endothermic peak between 120- 125℃, indicates lesser crystalline degrees for the functions LLDPE. In the light of the above discussion, it is clear that grafts didn't stifle the capability to crystallization while variations the crystalline degrees.
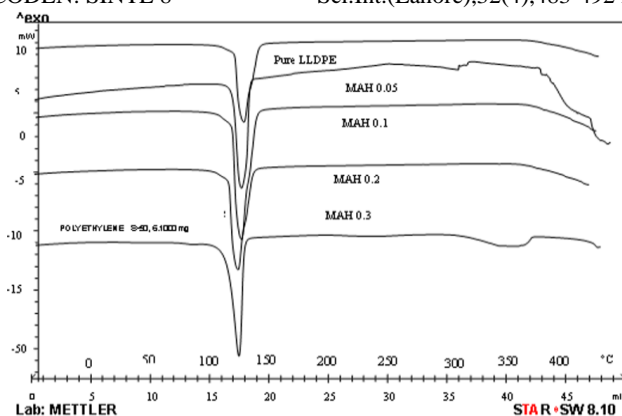
Fig. 12: Temperature effects on the melting temperature

4. CONCLUSIONS

It is evident from the investigation that the degrees of grafts and tensile characters showed an increasing tendency towards greater value, then decreasing with rising BPO content. However the outcomes indicated that the weight of molecules (Mn) of modified LLDPE rise with raising the contents of BPO and further, contributes to remaining enhancements in the melting viscosities of the materials. In addition the outcomes observed that the tensile characteristics and graft degrees rises greatly with rising maleic anhydride concentrations until regarding 0.2phr, then dropped markedly with more rising in the contents of maleic anhydride. The improved interfacial adhesion between LLDPE -g-MAH and PA6 has the significant impacts on tensile strength, consequently, reduce the chances of inter facial deboned and leads to improving tensile characteristics. It was found that the introduction of 0.2phr MAH, 0.46phr BPO is sufficient in the blend to determine the grafts degrees greater than 7.5 %.

DSC curves observed that the graft degree increases the moving ability of chains of LLDPE and further decrease the crystallization melts temperatures of the LLDPE. Hence this experimental work tells that the LLDPE maleation productively prepared by twin-screw extruder.

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