Mechanical and Thermal Properties of Calcium Carbonate-Filled PP/LLDPE Composite

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ABSTRACT: Polymer blends typically are the most economical means to develop new resins for specific applications with the best cost/performance balance. In this paper, the mechanical properties, melting, glass transition, and crystallization behavoir of 80 phr polypropylene (PP) with varying weights of linear low density polyethylene (LLDPE) at 10, 20/ 20 wt % CaCO$_3$, 30, 40, and 50 phr were studied. A variety of physical properties such as tensile strength, impact strength, and flexural strength of these blends were evaluated. The compatibility of these composite was examined by differential scanning calorimetry (DSC) to estimate T_m and T_c, and by dynamic mechanical analysis (DMA) to estimate T_g. The fractographic analysis of these blends was examined by scanning electron microscopy (SEM). It has been confirmed that increasing the LLDPE content trends to decreases the tensile strength and flexural strength. However, increasing the LLDPE content led to increases in the impact strength of PP/LLDPE blends. It was also found that up to 40 phr the corresponding melting point (T_m) was not effected with increasing LLDPE content. Each compound has more than one T_g, which was informed that there is a brittle-ductile transition in fracture nature of these blends, the amount of material plastically deformed on the failure surface seems to increase with the increasing the LLDPE content. And PP/LLDPE blends at temperature (23°C) showed a ductile fracture mode characterized by the co-existence of a shear yielding process; whereas at lower temperature (−20°C) the fractured surfaces of specimens appear completely brittle. The specimens broke into two pieces with no evidence of stress whitening, permanent macroscopic deformation or yielding. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 121: 2413–2421, 2011

Key words: thermal properties; random polypropylene; linear low density polyethylene; calcium carbonate composite

INTRODUCTION

Polymer blending can be used to obtain materials with a balanced combination of specific properties. Blends typically are the most economical means to develop new resins for specific applications with the best cost/performance balance. The components of the blends are chosen based on the properties, which they can contribute to a blend.[1,2] The preparation of binary PP/LLDPE blends has not been sufficiently studied due to the inherent problems of compatibility between PP and LLDPE. These include, for example, differences in melt viscosities, processing temperatures and thermodynamic solubilities, these differences are especially prominent with polypropylene (PP).[2–6] As our suggestion adding of a small amount of CaCO$_3$ to PP/LLDPE blends is expected to improve the mechanical and thermal properties of PP/LLDPE blends, because the T_g effect of PP by adding LLDPE would be excessive from the viewpoint of intended applications. CaCO$_3$ addition is also intended to reduce the difference between the processing temperature of PP and LLDPE. Blends of PP with linear low density polyethylene (LLDPE) were patented by Phillips Petroleum and by Esso Research and Engineering (1963). PP was blended without compatibilization with 20–40% LLDPE, to give materials with high impact resistance and low brittleness temperature. It is worth stressing that there is a great variety of LLDPE with different structures, chemical compositions, and molecular weight, some of these polymers show enough thermodynamic interaction with PP that compatibilization is not necessary.[6]

During the last few years, investigations of the melting and crystallization behavior of PP have become a subject of increasing interest. This is due to the development of some new technologies, where thermoplastic polymers are used as matrices for composite materials.[6] Filler such as calcium carbonate, talc, glass fiber reinforced to PP has found wide use as a light, stiff, and strong material, having a higher temperature resistance than the PP homopolymer. Thus, a system consisting of polymer

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matrix, elastomer, and filler could have been an attractive material for numerous engineering applications.[7]

In recent years, polymer/fillers composites have attracted great interest, both in industry and in academia, as they often exhibit remarkable improvement in materials properties when compared to virgin or conventional microcomposites. Today, more than 70 companies, government agencies, and academic institutions have been identified as having research and development activities.[8,9] These nanocomposites exhibit superior properties such as enhanced mechanical properties, reduced permeability, improved thermal stability, and flame retardancy.[10,11] One of the major limitations of PP nanocomposites is low impact resistance, particularly at high organoclay content. Several researchers have been reported that the impact strength and tensile elongation at break of PP decreased steadily as the inorganic material increased. Kaempfer et al. (2002) reported that by adding 20 wt % of the $CaCO_3$ and 20 wt % compatibilizer, the impact strength was reduced to one-third of the impact strength of the bulk PP.[11,12] The study of Reichert et al. (2000) showed that the PP nanocomposites exhibited brittle fracture and reduction of elongations at break compared to neat PP.[13] Thus, the incorporation of $CaCO_3$ especially at high loading resulted in a severe embrittlement manifested in a drop of the impact strength and tensile elongation at break. Esso Chemical Company was used PP/LLDPE blends and studied the mechanical and thermal proeperties of their products as shown in Table I.

In this study, different polymer combinations were tried to produce new blends. PP used as a matrix to blended with LLDPE incorporate into $CaCO_3$, a small fixed amount of $CaCO_3$, and the LLDPE content is gradually increased. The product is tested for their mechanical and thermal properties. The aim was to study the effect of adding a small amount of $CaCO_3$ and increasing the weight percentage of LLDPE on the mechanical and thermal properties of PP/LLDPE blends, and compare the results of these blends with the most important performances of (Esso Chemical Company).[2]

EXPERIMENTAL

Materials

LLDPE is Raslanuf Co, LLF 181N type, Libya, MFI 1.0 g/min, in pellets form.

PP is PPR R200P type, Hyocung Co, Korea, MFI 0.25 g/min, in pellets form.

$CaCO_3$ of Al-jouf Company, Libya, Particle size in 2.61 μm. Basic properties of PP and LLDPE are presented in Table II.

Sample preparation

To achieve good results, the materials have to be properly dried. The materials were dried in circulating air oven at 80°C for a period of 3 h just prior to processing. 80 phr PP was melt blended with LLDPE at 10, 20/20 wt % $CaCO_3$, 30, 40, and 50 phr LLDPE using a twin screw extruder with a special mixing head. The above blends were coded, respectively, as I-1, I-2, I-3, I-4, and I-5, (the numerical value denotes percentage LLDPE). Extruded pellets of these blends were injection molded for study of the mechanical properties.

Sample chacaterization techniques

Tensile tests were measured using TIRA tests. Tests were conducted on injection molded dumb-bell-shaped specimens of dimensions $10 \times 0.8 \times 0.25$ cm³ at room temperature at a crosshead speed of 10 mm/min., according to ASTM standard D638. The samples were extended until break. The modulus was determined from the slope of the initial part of the stress-strain curve within the linear trend.

Notched Charpy impact strength was measured using a Charpy impact tester at different temperatures (−20, 0, 23, 40, and 60°C) according to ASTM D256. The samples were prepared from molded impact bars (MIB), all the specimens had the dimensions $5.5 \times 0.6 \times 0.4$ cm³ with a notch cutter, which provided a notch depth of 0.025 cm. Flexural tests were carried out using TIRA equipped with a three point flexural testing fixture. The standard bending bars of dimensions $5.5 \times 1.0 \times 0.4$ cm³ were used for this study. The fixture has a span length of 76.2 mm. Samples were tested at a speed of 10 mm/min. Strict ASTM standard D790-86 were followed.

TABLE I
Properties of Esso Chemical Company

Property	Esso 4010
Tensile strength/MPa	27.895
Flexural modulus/MPa	865
Notched Izod/kJ.m⁻²	4.349
Heat deflection temperature HDT °C	95
Melting temperature °C	158
Heat of fusion J/g	102

TABLE II
Basic Properties of PP, LLDPE

Properties	LLDPE	PP
Density/g.cm⁻³	0.918	0.923
Tensile yield strength/MPa	16	31.63
Tensile strength/MPa	25	27.00
Flexural strength/MPa	13	31.00
Flexural modulus/MPa	270	850

Dynamic mechanical thermal analysis (DMA) was performed by using NETZSCH DMA 309 (Germany) instrument of polymer laboratories. Bending storage modulus (E′), loss modulus (E″) and tanδ were measured in the temperature interval –100 to 150°C at a heating rate of 10°C min^{-1}. The frequency was 1 Hz. The values of glass transition temperature (T_g) were read off as the temperatures of the peak of the tanδ.

Differential scanning calorimetry (DSC) test was carried out with NETZSCH DSC 209 (Germany) instrument in the temperature range −50 to 500°C. The heating rate was 10°C min^{-1}. The T_g (if any) was determined as the temperature corresponding to the inflection point on the heating curve by means of a built-in program Graphware TA72. The melting temperature, T_m, corresponds to the location of the melting peak on the temperature scale. T_d was determined as the temperature corresponding to the degradation temperature on the heating curve.

Fracture surfaces were observed to reveal the macroscopic features of fracture initiation and propagation. Fracture surfaces were observed at low magnitude without etching to reveal the macroscopic behavior of the failure. The fracture surfaces for scanning electron microscopy (SEM) study were perpendicular to the mold filling direction of the injection-molded bars and were coated with a thin layer of gold under vacuum before being studied in an instrument 91-0431 microscope. Five specimens of each formulation were tested, and the average values were reported.

RESULTS AND DISCUSSION

Strength properties

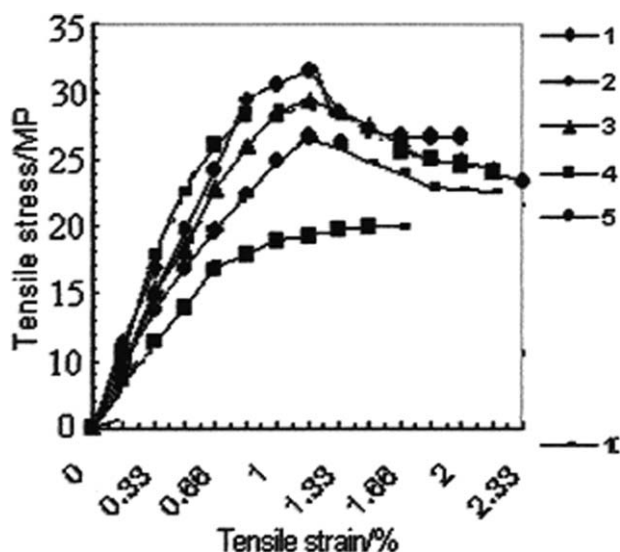Tensile stress-strain plots for PP/LLDPE blends are shown in Figure 1. It can be seen that PP exhibits the highest yield stress value. Addition of LLDPE to PP the yielding nature of PP. I-1 shows a similar trend but lower yield value with respect to the copolymer. For I-2, the yield stress as well as the stress continue to decrease. In addition, the shape of the yield peak is broadened. For I-3, the peak broadening still increases. The same trend is seen for I-4 and for I-5 the behavior begins to approach that of pure LLDPE, and the rupture becomes semiductile.

Tensile strength, tensile yield strength, tensile, fluxural strength, fluxural modulus, and impact strength of PP/LLDPE blends are presented in Table III. As can be seen in Figure 3 that tensile strength a decrease as the LLDPE content increase decrease as the LLDPE content is increased. This is due to the fact that LLDPE acts as an impact modifer for the PP.

It can also be seen that there is improvement in the tensile strength, the flexural modulus, and notched Izod impact strength in the case of PP/LLDPE filled to 20 wt % of CaCO₃ (I2) compared with Esso (see Table I). Better interfacial interaction in the case of PP/LLDPE blends with 20 wt % of CaCO₃ may be responsible.

Notched Charpy impact strength is one of the important objectives of the present work, and it is appropriate to present the results in graphical form (Figure 2). PP has notch and thickness sensitivity in unmodified commercial engineering plastics. These low critical thickness values tend to limit wall thickness of molded PP to a thickness below the critical thickness. PP exhibit notched Izod impact values that are dependent on the thickness of the PP. Thus, for example, while typical notched Izod impact values for a 3.2 mm thick PP test specimen are generally in the range of about 850 J/m. Typical notched Izod impact values for a 6.4 mm thick PP test
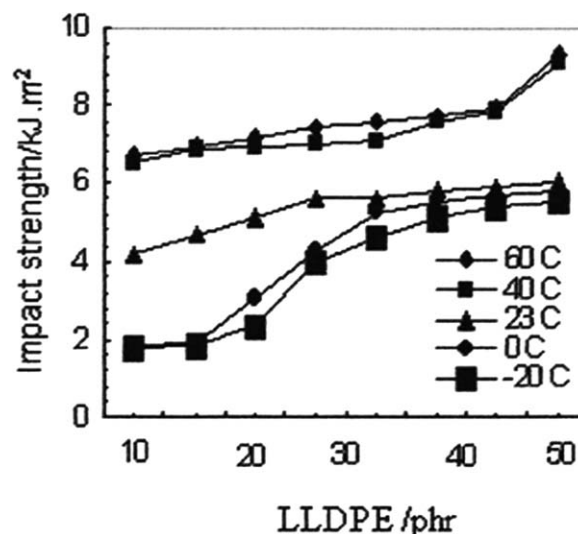

Figure 1 Tensile stress-strain plots for PPC/LLDPE blends: 1, PP; 2, I-1; 3, I-2; 4, I-3; 5, I-4; LLDPE.

Figure 2 Impact strength for PP/LLDPE blends.

TABLE III
Mechanical Properties of PP/LLDPE Blends

Property		I-1	I-2	I-3	I-4	I-5
Tensile strength/MPa		31	28.6	27.5	26.7	24.7
Tensile yield strength/MPa		31.5	27	26	25	23.5
Flexural strength/MPa		33.6	31.6	28.5	24.7	24.1
Flexural modulus/MPa		1451	1266	1094	1012	1014
Impact strength /kJ.m^{-2}	−20	1.76	1.86	2.32	3.94	4.64
At different temperatures (°C)	0	1.81	1.95	3.02	4.26	5.24
	23	4.18	4.7	5.1	5.57	5.57
	40	6.49	6.87	6.96	7.01	7.09
	60	6.73	6.96	7.19	7.42	7.56

specimen are generally in the range of about 160 J/m. The high Izod values of the 3.2 mm thick PP test specimen are due to the fact that these specimens are thinner than the critical thickness of the polymer and, therefore, upon impact, a ductile break occurs. The low Izod impact values of the 6.4 mm thick PP test specimens are due to the fact that these specimens exceed the critical thickness of the polymer and, therefore, upon impact, a clean or brittle break occurs.[6] Blending of PP with LLDPE can improve this phenomenon. Figure 2 also shows the impact strength at different temperatures versus various LLDPE contents for PP/LLDPE blends. It can be seen that the impact strength increases as the temperature is increased, increasing LLDPE enhances the impact strength of PP/LLDPE blends, but the value of impact strength at room temperature is still lower than that of PP itself.

Thermal properties

Figure 4 shows the plot of tanδ versus temperature for PP/LLDPE blends and filled $CaCO_3$. From Figure 4, it can be seen that PP has one peak at about −10°C, this transition corresponds to the glass transition of the PP has one peak at the measured temperature range (−150 to 100°C). We estimated the T_g of PP, which at about −10°C. For I2/ 20 wt %, $CaCO_3$ has two peaks at about –60 and 80°C, the transition at

−75°C corresponds to the glass transition of the LLDPE phase, whereas that at −62°C corresponds to the glass transition of the LLDPE matrix phase blend of PP (there is a small peak at about −10°C which corresponds to the remained amout of PP). And it is clear that, for the blends the T_gs remain at about the same temperatures for PP but are much more lower for the LLDPE. The shift of LLDPE T_g (from −20°C for pure LLDPE to about −75°C for PP/LLDPE blends) suggested that probably a certain amount of low molecular weight PP molecules part of $CaCO_3$ may act as reienforcment for LLDPE. As the LLDPE T_g decreases, the compound becomes more resistance to impact.

Dynamic mechanical thermal analysis

The properties obtained from the dynamic mechanical thermal analysis are the storage modulus (E′) and loss modulus (E″) that is recorded as a function of temperature from −150 to 100°C and showed in Figure 5 1a showed the inset for temperature between −40 to 20°C. All the specimens showed a glass state that is followed by the rubbery state. In general, the storage modulus, E′ is

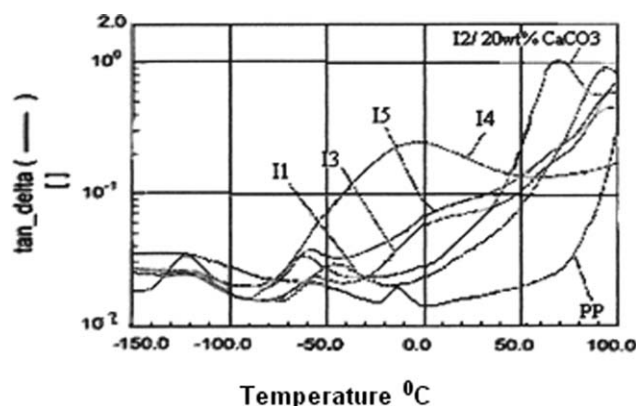**Figure 3** Tensile strength of PP as function of LLDPE content.

Figure 4 DMA curves LLDPE; PP; and PP/LLDPE/ filled $CaCO_3$ blends.
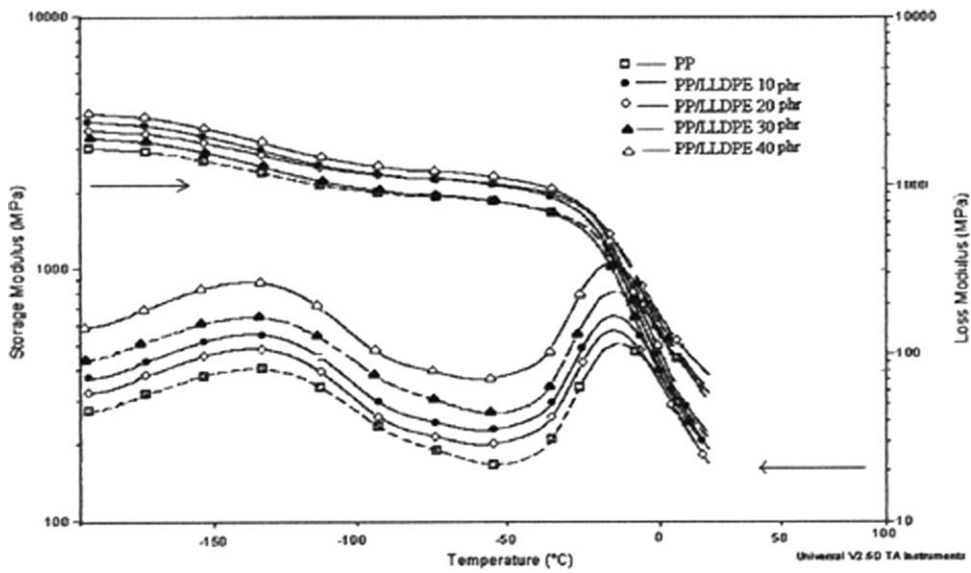
Figure 5 Variation of storage and loss modulus of PP/LLDPE blends versus temperature.

found to slight increase with increasing LLDPE content up to 40 wt % below the glass transition temperature. This indicates that incorporation of LLDPE has slightly improved stiffness of the blends and the dependence of E' on LLDPE content is more pronounced around the rubbery region. The drop in the storage modulus with temperature during the transition from the glassy to the rubbery state occurs around $-40°C$ for all specimens.[14–18]

In terms of the loss modulus (E″) spectra, two transitions can be clearly seen. The α transition, related to the glass-rubbery transition, is due to molecular motions associated with unrestricted amorphous PP.[17,18] The α transition is related to the relaxation of restricted PP amorphous chains in the crystalline phase. The presence of crystals is necessary for this transition to occur. The α transition, occurring at about -120 to $-130°C$ is associated with the glassy-rubbery transition of the amorphous chains of the polymer and the a transition is related to the crystallites, but the relaxation occurs due to the presence of "rigid" amorphous molecules present within the crystal.[10] These regions disrupt the purity of the
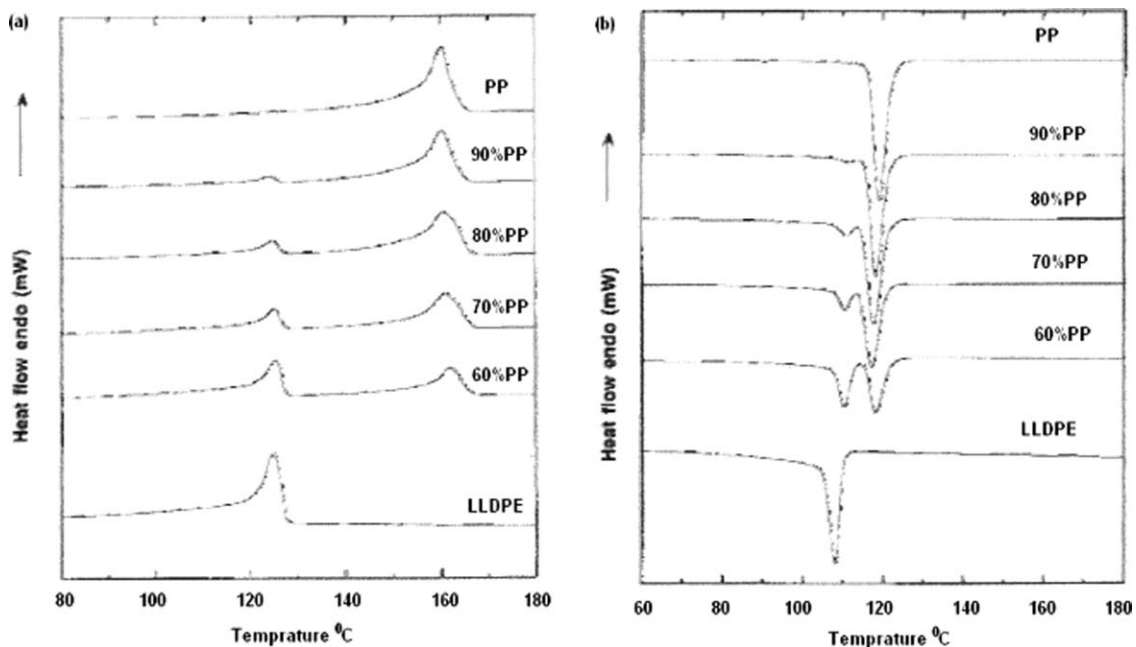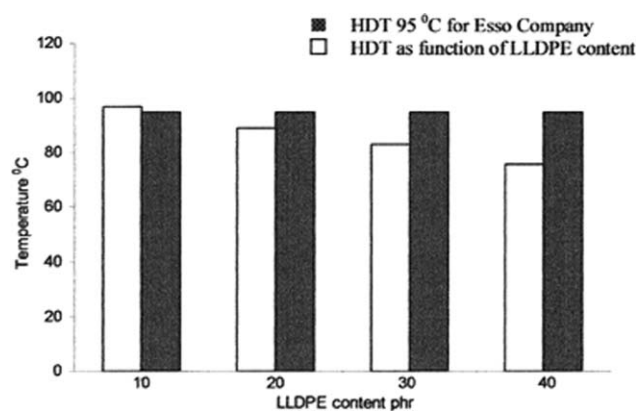

Figure 6 DSC curves of LLDPE, PP, and PP/LLDPE blends.

Figure 7 Effect of LLDPE on the heat deflection temperature compare to Esso company.

crystals and thus can be considered to be defects. The β-relaxation ($-10°C$) corresponds to the glass rubber relaxation (T_g) of constrained, noncrystalline chain segment of PP. Loss modulus increase upon increase LLDPE, as already discussed before. This increase can be due to the fact that the presence of LLDPE decreases mobility of the PP chains.[19–23]

Effect of addition LLDPE on crystallization and melting temperature

Figure 6. In the blends, the melting temperature (T_m) of PP increased slightly as shown in Figure 5(a). The increase in the T_m of PP could be caused by the dissolution of defective PP molecules into the LLDPE. The T_m of LLDPE also changed slightly in the blends Figure 6(a). However, the degree of change is smaller than that of PP, implying that PP may be more soluble in LLDPE than is LLDPE in PP. The crystallization endotherms (T_c) of both PP and LLDPE shifted toward each other in the blends Figure 6(b), indicating amutual interaction. The T_c of LLDPE increased 2–3°C in the blends, while the composition of PP did not affect the T_c of LLDPE significantly. On the contrary, the T_c of PP also decreased slightly with addition of LLDPE.

According to the results of DSC curve can be observed that the change in melting and crystallization temperatures of PP and LLDPE indicated that there was some degree of interaction between PP and LLDPE. The decrease of the PP in LLDPE the blends also suggested that PP was partially miscible with LLDPE. Also, this melting behavior is very dependent upon the chemical structure of the material along with the size and regularity of the crystallites found in the crystalline phase.[6] This result had been similar observations earlier Holden et al.[20] who confirmed that there is partial miscibility between PP and LLDPE. results are nearly alike with Jingbo et al.[24] and Premphet and Horanont[25] who investigated the effects of $CaCO_3$ of varying particle size (i.e., 1.9, 2.8, and 10.5 μm), content and type of surface modification on crystallization and melting behavior, mechanical properties, and processability of $CaCO_3$-filled syndiotactic PP (sPP). It was found that $CaCO_3$ was a good nucleating agent for sPP.

The results obtained for heat deflection temperature is given in Figure 7. The data reveal a reduction in deflection temperature on added of the LLDPE from 0 to 50 phr that may be because of high elastomeric character of the LLDPE.[6] Figure 7 shown that the suitable content of adding LLDPE to the PP at 10 phr was 110°C upon increase LLDPE contents trends to decrease HDT.

The crystallinity of PP decreased upon the addition of LLDPE (Table IV) decrease occurred when LLDPE retarded the crystallization of PP, and there was a certain degree of miscibility between PP and LLDPE, at sametime, the crystallinity of LLDPE was affected by the presence of PP.[20] Therefore, it is clear that the effect of LLDPE on PP was more significant than that of PP on LLDPE. The crystallization peak temperature of PP shift from 119 to 117°C, this indicated that during crystallization, some interaction between PP and LLDPE was present.[21,22] Compared to the nonnucleated blends, the incorporation of nucleating agents significantly increased the crystallization peak temperature of PP and LLDPE, the same trend was reported in the literature, and it was due to the nucleation effects of the nucleating agent.[16] As previously reported, all authors[17–22] agree that in PP-LLDPE blends a decrease in the crystallization of PP in the presence of molten PP

TABLE IV
DSC Analysis Data for PP in Neat and Blends Samples PP/LLDPE

Sample	Heat deflection temperature (HDT)°C	Crystallization temperature for PP (°C)	Melting temperature for PP (°C)	Crystallinity content of PP (%)	Heat of fusion ΔH (J/g)
PP	110 ± 0.2	119.0	160.0	49.2	102.8
PP/LLDPE 10 phr	97 ± 0.1	118.5	160.2	47.3	98.8
PP/LLDPE 20 phr	89 ± 0.3	118.1	160.4	46.6	97.3
PP/LLDPE 30 phr	83 ± 0.4	118.0	161.0	45.8	95.7
PP/LLDPE 40 phr	76 ± 0.2	117.0	161.5	43.1	90.0
LLDPE	–	114.2	122.0	–	–
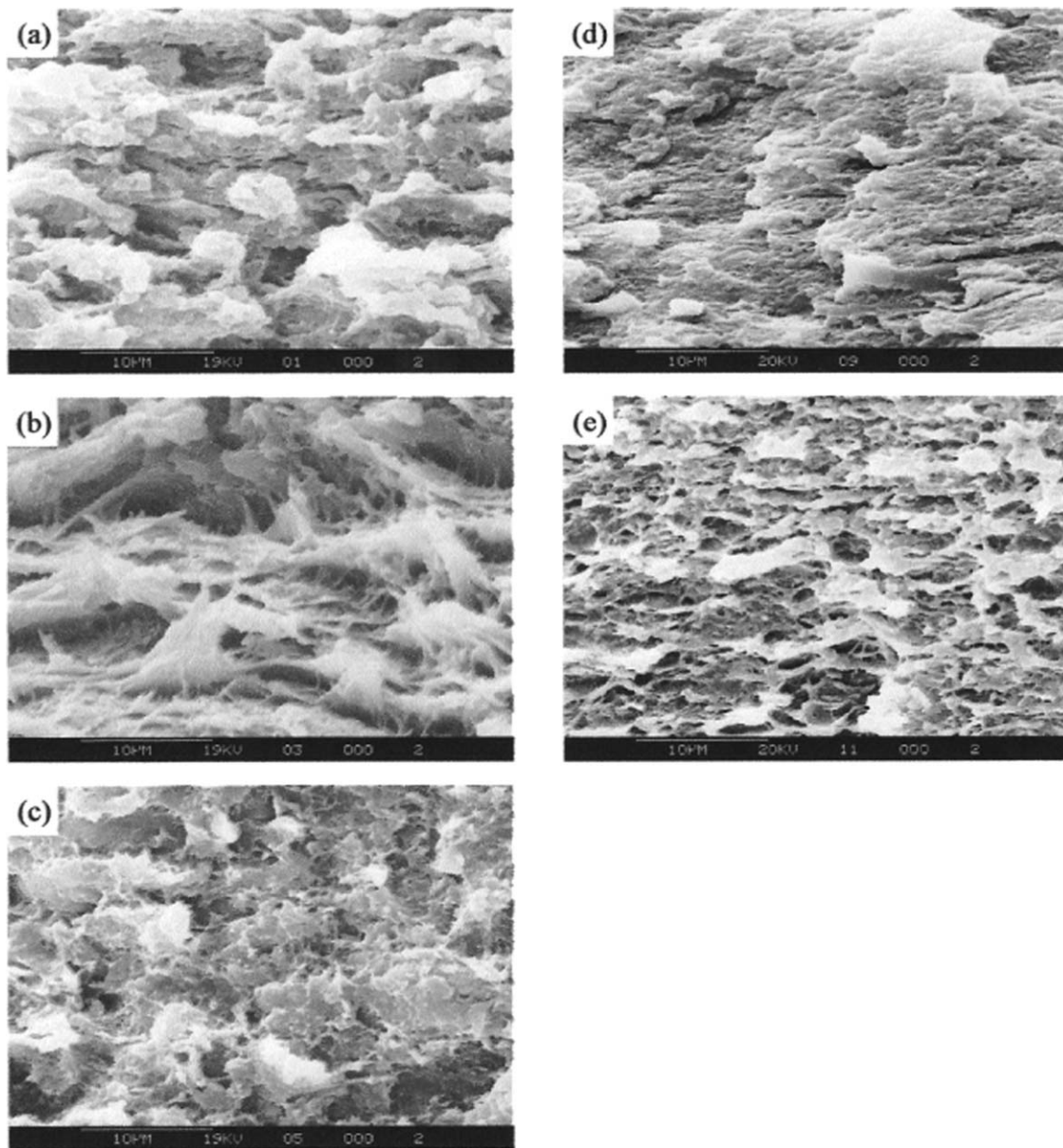
Figure 8 SEM micrographs of fracture surfaces: (a) I-1:PP/LLDPE/CaCO$_3$ = 80/10/20 (at 40°C); (b) I-2:PP/LLDPE/CaCO$_3$ = 80/40/20 (at 40°C); (c) I-3:PP/LLDPE/CaCO$_3$ = 80/80/20 (at 40°C); (d) I-5:PP/LLDPE/CaCO$_3$ = 80/50/20 (at 23°C); (e) I-5:PP/LLDPE/CaCO$_3$ = 80/50/20 (at −20°C).

is attributable only to a reduction in the nuclei density, which take place from immiscible melts.[23] It can be observed that melting temperature T_m for pure PP was 160°C and with the addition of LLDPE reached at high temperature 161.5°C at LLDPE 40 phr was increase slightly could be caused by the dissolution of defective PP molecules into the LLDPE. It was proposed by Greco et al.[16] The degree of change is smaller than that of PP, implying that PP may be more soluble in LLDPE than is LLDPE in PP.[15]

It can be concluded that an addition of rigid particles CaCO$_3$ to ductile polymer enhances the modulus and the heat deflection temperature. Also,

it has been reported that the incorporation of fillers into highly crystalline polymers generally contributes to an increase in HDT. It has been reported by (Gupta et al 6 ; Hatakyama and Quinn 19) that incorperation of filler into high crystalline polymer, generally contributes to an increase HDT Gupta et al.[6] The thermal properties was also compared with Saroop and Mathur[26] reveals that there is a decrease observed in the crystallite size on addition of 10 wt % LLDPE copolymer that increases afterward in the composition range of 10–40 wt %. Again, incorporation of glass fiber results in the reduction in the size, which is consistent with Table IV.

To clarify the influence of blends and temperature on the fracture mode of PP/LLDPE blends, a fractographic investigation was performed on the Charpy impact fractural specimens. Figure 8 shows scanning electron micrographs of failure surfaces of Charpy impact specimens of PP/LLDPE blends at different blends and temperatures. It is expect that, adding LLDPE and 20 wt % $CaCO_3$ (or I-1) in PP formed triple phase morphology, PP continuous phase, and LLDPE in PP and $CaCO_3$ particles in PP. It cannot distinguish between PP and $CaCO_3$ particles on the micrographs. As the miscibility of both PP-LLDPE and PP-$CaCO_3$ was acceptable, the interface between LLDPE and $CaCO_3$ particles induced some weak points, which were unable to withstand the impact loading.[10] The brittle fracture surface with LLDPE and/or $CaCO_3$ particles pulled out is seen in Figure 7a. As LLDPE content is increased to 40 phr (I-2), the trend is not brittle, also the surface appears to be rougher than of 10 phr LLDPE (I-1). But deformed zones are clearly visible on the surface (Figure 8b). These regions of the sample were prestrained during the blends before fracture initiation and underwent plastic flow during the crack opening. When LLDPE was 80 phr (I-3) in the blend, the fractural surface showed strong plastic deformation, accompanied by high shear deformation of LLDPE, and the impact strength increased gradually (Figure 8c). Temperature also seems to influence the fracture, all the specimens are broken at the temperature which they are tested. But can be concluded that PP/LLDPE blends at temperature (23°C) for I-5 blend show a ductile fracture mode characterized by the coexistence of a shear yielding process (Figure 8d).[20–22] At lower temperature (−20°C), the fractured surfaces of specimens for I-5 blend appear brittle. The some specimens broke into two pieces with no evidence of stress-whitening, permanent macroscopic deformation or yielding (Figure 8e). Considering the fact that LLDPE is an elastomer and PP is a semicrystalline matrix, it is expected that cavitations form on the dispersed LLDPE phase. But, the fractographic investigation shows that there are no clearly visible cavitations.[22] Similar study was confirmed by Jingbo et al.[24] The addition of small amounts of the nucleating agents had little effects on the viscosity ratio; therefore, they had slight influence on the phase morphology of the blends.

As reported by Starke et al.[27] suggested that the cavitation of the elastomer is the first step of deformation and that the formation of cavitation bands can accelerate the shear yielding of the matrix; this results in high impact energy absorption. It was found that in PP/elastomer/filler composites, the ligament thickness between neighboring elastomer particles was the key parameter determining whether the material would be brittle or tough, similar result of Silva et al.[28] The interaction between the elastomer and filler, led to a decrease in the impact energy. In addition to, two other requirements were important: (1) the elastomer and filler particles had to be dispersed homogeneously and separately in the matrix, and large aggregation had to be avoided, (2) particle debonding had to occur to allow unhindered plastic deformation around the particles.

The concluded of our results indicate that there exhibits a limit elastomer concentration that is required to obtain a brittle-to-ductile transition for polymer/elastomer/filler composites. However, whether the main factor determining the impact resistance was between elastomer, particles, which was affected by the concentrations of the elastomer and filler. At different weight ratio of PP/LLDPE and $CaCO_3$.[17]

CONCLUSIONS

From this study, the following conclusions could be drawn:

1. The tensile strength, the flexural strength, and the flexural modulus decrease as the content of LLDPE is increased.
2. The impact strength for the products is quite high. Both increasing temperature and LLDPE content increase the impact strength.
3. According to DSC test results, For PP/LLDPE blends melting point (T_m) indicated that there was some degree of interaction between PP and LLDPE, also the (T_c) of PP trends to decreased slightly with addition of LLDPE.
4. An alternation of mechanisms between brittle and ductile failure was evidenced by Charpy impact tests and confirmed by a fractographic analysis.
5. Comparing these results with the most important mechanical properties of Esso chemistry Company, it has been observed that there is a development in the mechanical properties of the produced blends, and balance properties of thermal properties than the Esso Company results.

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