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The Influence of Filler Component on Mechanical Properties and Thermal Analysis of PP-LDPE and PP-LDPE/DAP Ternary Composites

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1. Introduction

Composite material is a material system consisting of a mixture or combination of two or more micro-constituents mutually insoluble and differing in form and/or material composition. Particulate-filled thermoplastic composites have proved to be of significant commercial importance in recent years, as industrialists and technologists have sought to find new and cost-effective materials for specific applications (Shonaike & Advani, 2003; Ma et al., 2007). With addition of inorganic filler, various changes occur in the molecular and supermolecular structure of a thermoplastic resin. Composite properties depend on a variety of material-process variables (e.g., polymer matrix structure, filler content, chemical composition, surface activity, particle size and shape, compounding extruder design, mold design, and extruder-molding process conditions). Some changes may be latent or delayed (i.e., occurring later in the service life of the plastic part as a result of surrounding conditions). Reduction of molecular weight, crystal and spherulite size, and molecular mobility are among the most profound effects that solid filler has on the polymer matrix structure (Ayae & Takashi, 2004). The microstructure of the polymer-filler interphase is mirrored by the mechanical integrity of the molded part and long-term durability to extremes of surrounding temperatures and applied stresses. Calcium carbonate is very commonly used filler in the plastics industry. The incorporation of fillers such as calcium carbonate into thermoplastics is a common practice in the plastics industry, being used to reduce the production costs of molded products. Fillers are also used to modify the properties of plastics, such as the modulus and strength. High filler loadings, however, may adversely affect the processability, ductility, and strength of composites (Rai & Singh, 2003). Some polyolefins are prone to chain-scission reactions in the presence of free radicals. PP is degraded due to chain scission in β position to the macroradicals site, while PE is crosslinked, due to macroradical recombination (Braun et al., 1998). The use of organic

peroxides for controlled degradation of PP is the most important commercial application of the chain-scission or visbreaking of polyolefin chains and results in the so-called controlled rheology PP grades with enhanced melt flow behavior (Zweifel, 2001). Polypropylene (PP) filled with calcium carbonate is among the more recent development on the polyolefin market and in the last decade have shown impressive growth rates. In polyethylene sector, calcium carbonate fillers now play a role preferably in films and sheets. Low density polyethylene's (LDPE and LLDPE) are usually filled with very pure calcium carbonate grades.

Several studies dealing with the melt rheology, mechanical, deformation, impact behavior and thermal properties of various blend or composites were published during the last decade (Chen et al., 2004; El-Sabbagh et al., 2009; Kolarik & Jancar, 1992; Mishra et al., 1997; Sirin & Balcan, 2010, Zhang et al., 2002). Wang WY (Wang, 2007, 2008) was studied the preparation and characterization of calcium carbonate/Low-Density-Polyethylene and CaCO_3 /acrylonitrile-butadiene-styrene composites. Tang *et al.* studied rheological properties of nano- CaCO_3 /ABS composites such as shear viscosity, extension viscosity, and entry pressure dropped by capillary extrusion (Tang & Liang, 2003). Liang investigated the tensile, flow, and thermal properties of CaCO_3 -filled LDPE/LLDPE composites (Liang, 2007). Effects of coupling agents on mechanical and morphological behavior of the PP/HDPE blend with two different CaCO_3 were studied by Gonzalez *et. al* (Gonzalez et al., 2002). Also, several researches have reported different properties of ternary composites with calcium carbonate (Jancar & Dibenedetto, 1995; Kim et al, 1993; Premphet, 2000).

This study has focused on the investigation of the changes on thermal, mechanical and morphological properties of PP-LDPE/DAP (90/10 /0.06 wt. %) blend and PP-LDPE/DAP (90/10 /0.0 wt. %) blend when different ratios of 5-10-20 wt. % CaCO_3 are added. The blend (PP-LDPE/DAP (90/10/0.06 wt.%) used in this study was prepared in terms of heat sealing strength properties by the results based on our previous work (Sirin & Balcan, 2010) and is optimum as well (Şirin, 2008).

2. Experimental

2.1 Materials

Isotactic polypropylene (PP-MH418) and Low-density polyethylene (LDPE-I 22-19 T) were supplied as pellets by Petkim Petrochemical Company (Aliaga, Izmir, Turkey). The number-average molecular weight (M_n), weight-average molecular weight (M_w) and polydispersity index (PDI) values of PP and LDPE homopolymer were 20300, 213600 $\text{g}\cdot\text{mol}^{-1}$ and 10.5, and 29600, 157000 $\text{g}\cdot\text{mol}^{-1}$ and 5.3, respectively. The specific gravity of the PP-MH418 is $0.905 \text{ g}\cdot\text{cm}^{-3}$ and that of the LDPE-I 22-19 T is $0.919\text{-}0.923 \text{ g}\cdot\text{cm}^{-3}$, with melt flow index of $4\text{-}6 \text{ g}\cdot 10 \text{ min}^{-1}$ (2.16 kg , $230 \pm 0.5 \text{ }^\circ\text{C}$) and $21\text{-}25 \text{ g}\cdot 10 \text{ min}^{-1}$ (2.16 kg , $190 \pm 0.5 \text{ }^\circ\text{C}$), respectively. (2, 5-dimethyl-2, 5-di (tert-butyl peroxy)-hexane, (Sinochem, Tinajin/Chine) was used as dialkly peroxide (DAP). Calcium carbonate filler (AS 0884 PEW) was provided by Tosaf Company (Israel).

2.2 Preparation of blends

In the preparation of the composites, two different procedures were used. In the first procedure, PP-LDPE/ CaCO_3 composites were prepared without addition of DAP. These composites are called as PC0, PC1, PC2 and PC3. In the second procedure, PP-LDPE/ CaCO_3

composites were prepared with (0.06 % wt.) addition of DAP. These composites are called as PC4, PC5, PC6 and PC7. All compounds were prepared by using single screw extruder (Collin E 30P). The blends were prepared by melting the mixed components in extruder which was set at the extruder diameter: 30 mm, length to diameter ratio: 20, pressure: 9-10 bar, temperature scale composites from filing part to head were 190-250 °C and screw operation speed: 30 rev.min⁻¹. The composites were produced as 70 µm thick and 10 cm wide films. These ratios and their codes are given in Table 1. All of these composites were prepared as samples weighing 1000 grams, while keeping the PP-LDPE (90/10) ratio constant.

Sample Code	Composition, wt %			
	PP	LDPE	CaCO ₃	DAP
PC0	90	10	-	-
PC1	90	10	5	-
PC2	90	10	10	-
PC3	90	10	20	-
PC4	90	10	-	0.06
PC5	90	10	5	0.06
PC6	90	10	10	0.06
PC7	90	10	20	0.06

Table 1. Nomenclature, components and composition of composites

2.3 Melt flow index (MFI) measurements

Melt flow index measurements of the composites were carried out on a MFI (MP-E) Microprocessor apparatus at 230 °C and under a 2.16 kg weight. The capillary die was 2.095 mm in diameter and 8 mm in length. About 5 g of composite was put into barrel and heated for 5 min to reach the predetermined temperature on the plunger to extrude the melt through the capillary die. After a steady flow state was reached, five samples were cut sequentially and their average weight value was obtained. Experiments were done according to ASTM D-1238.

2.4 Mechanical testing measurements

The tensile properties were determined using a Instron tensile tester (model 4411) following the ASTM D-638 procedure and using type 1 test specimen dimensions. The crosshead speed was set at 50 mm.min⁻¹ and 5 samples were tested for each composition. Tensile stress at yield, tensile strength at break and elongation at break were determined from the recorded force versus elongation curve.

2.5 Hardness test measurements

Shore D scale was used to determine the hardness values of all samples. The tests were carried out Zwick/Roell apparatus of out at the room temperature and 76 cm Hg pressure hardness

test (Shore D) was performed according to ASTM D 2240. Hardness test measurements were carried out the dimensions of 60x60x4 mm at 50 N, at the room temperature.

2.6 Heat seal tester

Heat sealing testers of samples were carried out with a TP701 (trade name, Tester Sangyo K.K.) apparatus at 1 sec timer and 2 kg/cm² pressure.

2.7 Thermal measurements

Differential scanning calorimetric (DSC) analyses were performed in a Shimadzu DSC-50 thermal analyzer in nitrogen atmosphere. The samples were heated from 25 to 200 °C at 10 °C min⁻¹, cooled to 25 °C at the same rate, and re-heated and cooled under the same conditions. Melting (T_m) and crystallization (T_c) temperatures and enthalpies were determined from the second scan. T_m was considered to be the maximum of the endothermic melting peak from the heating scans and T_c that of the exothermic peak of the crystallization from the cooling scans. The heat of fusion (ΔH_f) and crystallization enthalpy (ΔH_c) were determined from the areas of melting peaks and crystallization peaks.

The crystallinity of composites were calculated with the total enthalpy method [see eq. (1)]; in all calculations, the heats of fusion at equilibrium melting temperature were 209 and 293 Jg⁻¹, for PP and LDPE crystals, respectively (Brandrup & Immergut, 2003)

$$(X_c) = \frac{\Delta H_f}{\Delta H_{crys}} \times 100 \quad (1)$$

ΔH_f = Heat of fusion (Jg⁻¹)

ΔH_{crys} = 100% crystal polymer crystallization energy (Jg⁻¹)

(X_c) = Crystallinity (%)

The various melting and crystallization parameters which were determined by means of heating and cooling scans for composites are given in Table 3. Thermogravimetric (TG-DTG-DTA) curves were performed on a Seteram Labsys TG-16 thermobalance, operating in dynamic mode, with the following conditions; sample weight ~5 mg, heating rate = 10 °C.min⁻¹, atmosphere of nitrogen (10 cm³.min⁻¹), sealed platinum pan.

2.8 Scanning electron microscopy (SEM)

A Philips XL-305 FEG e SEM model scanning electron microscopy (SEM) was used to examine the morphologies of the composites

3. Conclusion

3.1 Mechanical analysis

CaCO₃ has a high chemical purity, which eliminates a negative catalytic effect on the aging of polymers. In addition, it has high whiteness and low refractive index that can help to reduce consumption of expensive abrasive pigments such as titanium dioxide. On the other

hand CaCO_3 is very well suited for the manufacture of colorful products. Low abrasiveness, which contributes to low wear of machine parts such as extruder screws and cylinders, is another advantage. These properties and its low cost make CaCO_3 a very strong alternative to be considered as filler.

Melt flow index (MFI) analysis of composites are shown in Table 2. MFI values of composites without peroxide showed small differences by increasing amount of CaCO_3 . Contrariwise, MFI values of composites with peroxide are proportional to the increasing amount of CaCO_3 . MFI values of composites without DAP are between 9 and 10 g/10 min, however addition of 0.06 %wt. DAP to the composites resulted MFI values to vary between 22 and 26 g/10 min. This increase in MFI values is a result of degradation of PP by the DAP.

Sample Code	MFI / g.10 min ⁻¹ ± 0.1	Tensile strength at break / kg.cm ⁻² ±10	Tensile strength at yield/ kg.cm ⁻² ±10	Elongation at break/ (%)±5	Hardness (ShoreD) ±1	Heat sealing strength/ kg.cm ⁻² ± 0.1 at 145 °C	Heat sealing strength/ kg.cm ⁻² ±0.1 at 150 °C
PC0	9.20	270	322	240	62.10	2.15	3.27
PC1	9.40	171	263	190	62.60	1.52	3.10
PC2	9.60	125	240	160	63.10	1.33	2.50
PC3	9.70	98	210	141	63.20	1.11	2.04
PC4	22.00	290	360	310	63.00	2.53	9.80
PC5	23.80	180	298	287	63.50	1.62	4.74
PC6	24.50	230	336	302	63.90	1.96	5.22
PC7	25.20	146	218	161	63.40	1.44	4.22

Table 2. MFI, heat sealing strength and mechanical analysis values of composites

Mechanical analysis of the composites such as tensile strength at break, tensile strength at yield and elongation at break are shown in Table 2. As shown in Table 2, samples with/without peroxides showed increase and decrease in their mechanical properties of composites in terms of increasing amount of CaCO_3 . The highest values in mechanical properties were observed in PC6. Even though PP, LDPE and CaCO_3 amounts were same in PC6 and PC2, there was only change in peroxide amounts. In other words, when PC6 and PC2 were compared it was observed that tensile strength at break, at yield, elongation at break values and heat sealing strength values of the composite PC6 showed high peaks. Table 2 summarizes tensile strength at break values for composites which do not contain peroxide displayed decreasing. However, by adding peroxide and increasing the amount of CaCO_3 , these values showed increasing due to crosslinking of LDPE with the effect of peroxide. Furthermore, the tensile strength of the composites decreased with increasing amount of CaCO_3 due to the weak interfacial adhesion and dispersion of the CaCO_3 filler

to PP-LDPE surface. The decrease of yield stress is likely due to the depending between inorganic fillers and the PP matrix at large deformations. In particular, a higher drop in tensile strength at yield is observed for PP-LDPE/CaCO₃, possibly due to the splitting of aggregated particles as well as depending between CaCO₃ particles and the PP matrix. In Table 2, with/without peroxide it is clearly shown that elongation at break values of the composites decreases with increasing ratio of CaCO₃. On the other hand, while comparing samples with CaCO₃ to each other the highest mechanical properties were observed in PC6. The reason for this is that adding DAP effect LDPE by crosslinking and PP by degradation. Shore D values of the composites with/without DAP increase in some degree with increasing amount of CaCO₃. As mentioned above, the reasons for increase in shore d values of the composites are addition of DAP effect to LDPE by crosslinking, PP by degradation, and dispersion of CaCO₃ in PP and LDPE. Table 2 shows that, increasing amounts of CaCO₃ resulted a decrease on heat sealing strength values at 145 °C and 150 °C independent of DAP. Therefore, the decrease of heat sealing strength is owing to the depending between in organic filler and the blends matrix of deformations. PP-LDPE/CaCO₃ composites still gave better heat sealing strength results than homopolymer PP and LDPE. In previous studies, we have examined heat sealing strength of homopolymer PP and LDPE (Sirin & Balcan, 2010; Şirin, 2008) Same as in mechanical properties values, heat sealing strength values showed similar trend in composite PC6 for best results.

3.2 Morphology observation

Figure 1 (a-g) shows the scanning electron microscopy (SEM) micrographs of composites reinforced with different amounts of CaCO₃ (0, 5, 10, and 20 %wt.) and DAP (0, 0.06 %wt.). From these micrographs, it is clear that CaCO₃ fillers were dispersed well in PC4, PC6 and PC7 matrices with DAP of composites. In these matrices, a homogeneous dispersion of reinforcing particles can also be observed. Moreover, the fillers remained intact within the matrix. This indicates that good bonding existed between the CaCO₃ particles and matrix. In contrast, the agglomeration of fillers can be observed for the composites containing CaCO₃ particles and dialkylperoxide (DAP). In homogeneous dispersion of fillers can cause a loss in the mechanical strength of the composites considerably. At higher loading levels however, CaCO₃ will agglomerate and remain confined in the polymer matrix.

3.3 Thermal analysis of composites

The results of the thermal analysis that was carried out by means of differential scanning calorimetry (DSC) are presented in Table 3. Overall, there were increased effect of both the content of the CaCO₃ filler and its healing on the melting temperature (T_m) and the crystallization temperature (T_c). However, a decrease in the energy required for the fusion of the crystalline parts was noted when the content of the filler in the composite increased. The melting enthalpy and crystallinity % (X_c) of the CaCO₃ containing composites with DAP (0.06 %wt.) were a little higher than the composites without DAP. Yet, the values of heat of fusion remain lower than that of homopolymer PP or LDPE. It can therefore be noted that the filler alters the crystalline phase of the polymer.

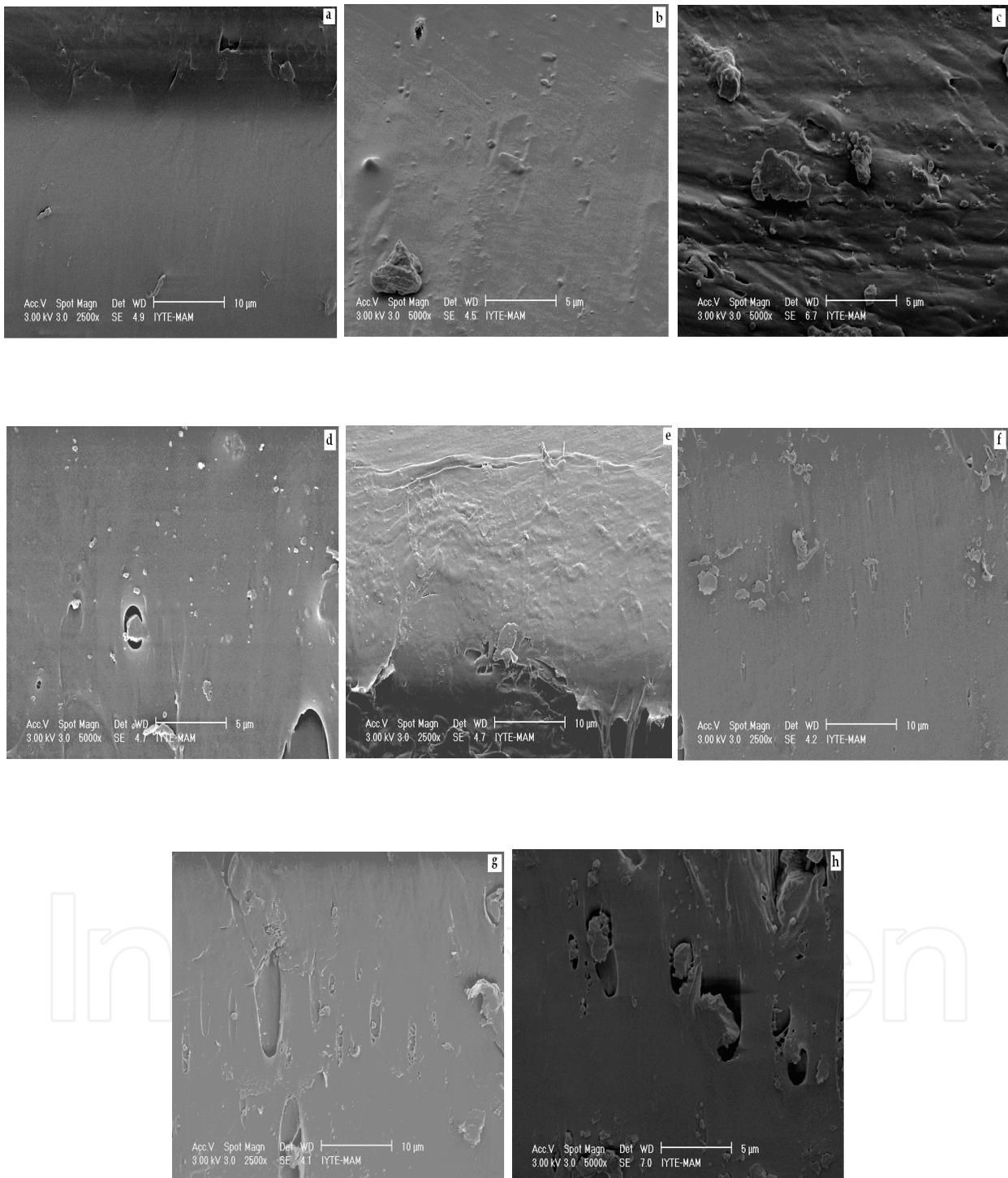


Fig. 1. SEM micrograph of blend and composites (a) PC0 (b) PC1 (c) PC2 (d) PC3 (e) PC4 (f) PC5 (g) PC6 (h) PC7

Sample Code	Melting (from second heating scans)				Crystallization (from second cooling scans)					
	LDPE		PP		LDPE		PP		LDPE	PP
	$T_m/$ °C	$\Delta H_f/$ J.g ⁻¹	$T_m/$ °C	$\Delta H_f/$ J.g ⁻¹	$T_c/$ °C	$\Delta H_c/$ J.g ⁻¹	$T_c/$ °C	$\Delta H_c/$ J.g ⁻¹	$X_c/$ %	$X_c/$ %
PC0	102.00	2.90	162.10	91.00	95.00	3.00	118.40	88.90	1.00	43.50
PC1	102.60	2.90	162.90	83.10	95.50	1.40	118.90	90.40	1.00	39.80
PC2	102.80	2.30	162.30	73.20	96.80	1.20	119.30	76.00	0.80	35.00
PC3	103.40	2.20	162.60	70.30	99.40	1.10	120.10	66.30	0.70	33.60
PC4	102.50	3.10	162.30	85.50	94.20	2.90	118.50	87.00	1.10	40.90
PC5	103.20	2.40	164.90	81.70	95.30	2.00	119.00	80.40	0.80	34.20
PC6	106.30	2.80	165.10	86.40	99.20	2.80	120.70	86.90	0.70	39.10
PC7	104.70	1.10	164.60	62.00	97.40	1.30	120.40	76.90	0.40	29.70

Table 3. Thermal properties of PP-LDPE/CaCO₃ composites

Thermal behavior of the composites was studied with a thermogravimetric analyzer under a protective nitrogen atmosphere. The temperature was scanned from 30 °C to 700 °C at a heating rate of 10 °C.min⁻¹. Figure 2 and 3 shows the typical thermogravimetric curves for composites with/without DAP and different filler contents are presented. 5 % and 20 % weight loss temperatures ($T_{-5\%}$ and $T_{-20\%}$) and maximum weight loss temperature (${}^bW_{max.T}$), derived from the derivative weight loss and differential thermal analysis curves are tabulated in Table 4.

Compounds	TG					DTA
	${}^aT_{on}$	${}^bW_{max.T}$	weight loss/ (5 %)	weight loss/ (20 %)	residual	Endo
PC0	403	454	406	438	0.5	105.2, 162.2, 457
PC1	402	453	406	438	3	105.8, 162.6, 458
PC2	405	454	407	440	9	106.6, 163.2, 457
PC3	405	455	407	440	18	106.5, 163.5, 459
PC4	407	456	409	443	6	107.2, 164.4, 459
PC5	409	457	413	444	11	107.9, 165.7, 459
PC6	414	461	415	444	11	108.1, 167.3, 462
PC7	414	459	409	444	26	108.7, 164.5, 460

Table 4. Thermal decomposition values of all the compounds (^aThe onset temperature, ^bMaximum Weight Temperature)

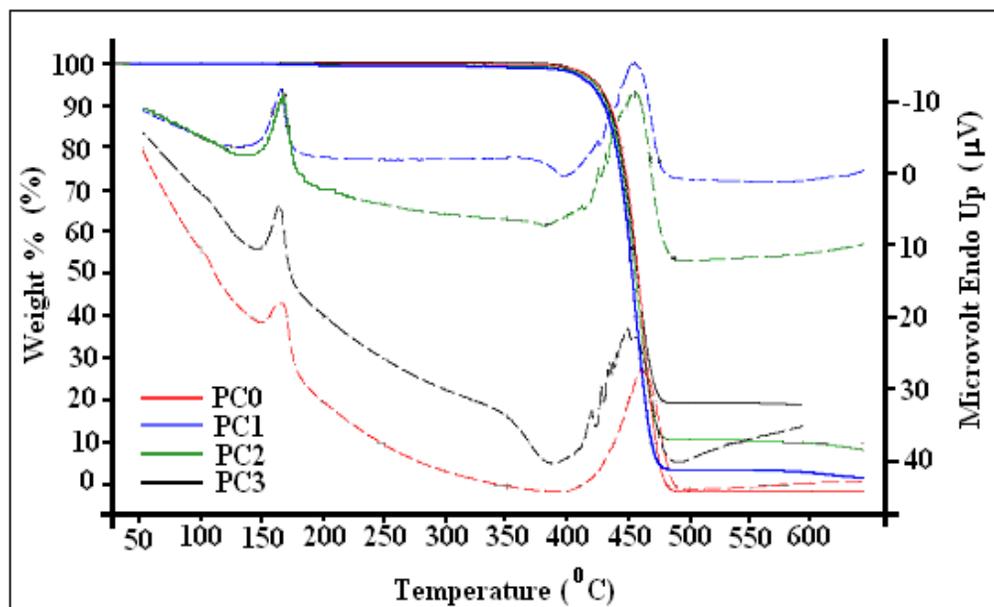


Fig. 2. TG and DTA curves of composites containing the different amounts of CaCO₃ (PC0, PC1, PC2, and PC3)

TG curves for all the compounds exhibits one stage decomposition and a similar characterizations. The thermal decomposition of composites occurs between 403 and 500 °C. The thermal stabilities of composites increased usually with increasing CaCO₃ content. PC6 has the highest thermal stability among the polymer blends. It is also shown that the rate curve related to compound shifts to a higher temperature. Also, three endothermic thermal effects at different temperature in DTA profiles correspond to the melting and the decomposition of composites. In DTA curves, the first two peaks are two melting peaks.

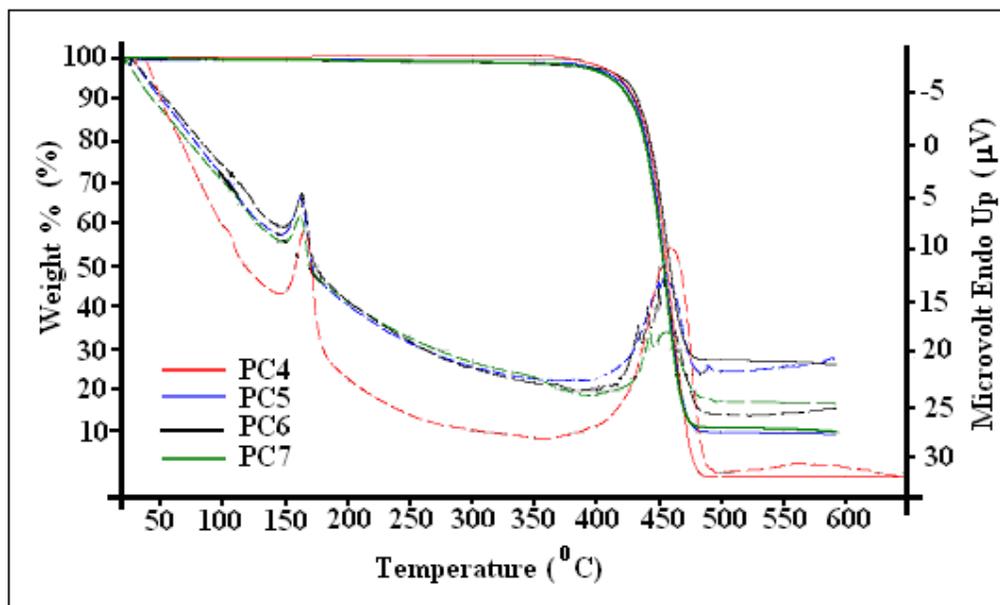


Fig. 3. TG and DTA curves of composites containing the different amounts of CaCO₃ (PC4, PC5, PC6 and PC7)

This is another proof showing that the components are incompatible. But CaCO₃ dispersed completely in the blends and it didn't result another peak. As observed, melting peaks were between 105-108 °C (LDPE) and 162-167 °C (PP), decomposition temperatures related to the maximum weight loss ($W_{max.T}$) were between 454-459 °C. In the case of composites, although PP and LDPE decomposition completely at 500 °C, according to the amount of CaCO₃ 5, 10, or 20 % amount of mass, remained due to the reason that CaCO₃'s decomposition temperature is between 850-900°C.

In conclusion, polypropylene-Low Density Polyethylene blends with/without DAP and containing different amount CaCO₃ filler component was prepared by melting-blend with a single-screw extruder. The effects of CaCO₃ filler component on mechanical and thermal properties of prepared composites were investigated. Addition of CaCO₃ particles to the polymer matrix with DAP significantly increased MFI values. It was observed that mechanical properties (tensile strength at break, at yield, elongation at break values and heat sealing strength values) of the composite PC6 showed high peaks. With addition of CaCO₃, while mechanical properties of the composites were decreasing shore D values showed increasing. Heat sealing strength at 150 °C increased by increasing amount of CaCO₃ particles in the polymer matrix with DAP. SEM images showed that CaCO₃ particles were well-dispersed in the polymer matrix with DAP. The observation of TG/DTG/DTA curves revealed that the thermal stabilities of composites increased usually by increasing amounts of CaCO₃ and the blends are incompatible.

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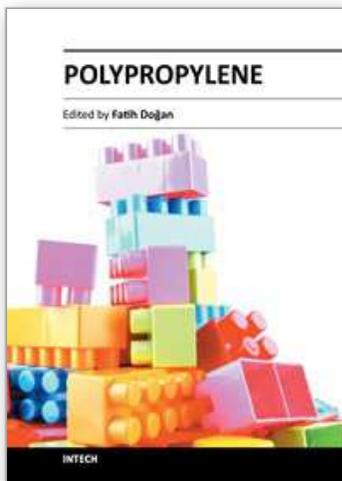
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