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Effect of Surface Modified Nano-Calcium Carbonate on the Properties of Polyethylene/Nano-$CaCO_3$ Nanocomposites

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Abstract: In this study, nanocmposites of high-density polyethylene (HDPE)/linear low-density polyethylene (LLDPE) filled with unmodified nano-calcium carbonate (denoted as UnCC) and surface modified nano-calcium carbonate (indicated as MnCC) were manufactured by melt-mixing method then compression molding. The effect of isopropyl tri-(dioctylpyrophosphato) titanate (JN114) modified of nano-calcium carbonate (nCC) concentration on the mechanical, morphological and flow properties of the nanocomposites were studied. Three compositions of HDPE/LLDPE/nCC nanocomposites were prepared in a corotational twin-screw extruder with nCC content of 5, 10, 15, and 20 wt%. The results of scanning electron microscopy (SEM) showed that the JN114 treatment of nCC enhanced the interfacial adhesion between the filler and the matrix, indicating the improvement in the compatibility between HDPE/LLDPE and nCC. Measurements of mechanical properties showed that the tensile fracture strength (σ_b) of the modified nCC filled polyethylene composite was significantly higher than that of the unmodified composite, especially in the case of higher filler content. The tensile elastic modulus and impact strength was increased with increasing weight fraction (ϕ) of the fillers. Furthermore, the melt flow rate (MFR) of the nanocomposite materials was measured. It was found that the MFR decreased with the addition of ϕ.

Keywords: HDPE/LLDPE Blend, Nano-$CaCO_3$, Flow Property, and Mechanical Properties.

1. Introduction

Polyethylene nanocomposites have involved much attention from both industry applications and academic research fields due to their remarkable properties, like as mechanical, thermal, electrical, and rheological properties (Zaman & Beg, 2016). Polymer nanocomposites are a class of materials that are particle-filled, with at least one dimension in the nanometer range (Mishra, Seenivasan, Mukherjee & Chandrasekaran, 2019). These nanoparticles are dispersed in the polymer matrix at a relatively low wt% (often less than 10% by weight). In general, nanoparticles can significantly improve mechanical properties, thermal stability, gas barrier properties, and/or flame retardancy of the polymer matrix (Mohammed, Tcherbi-Narteh & Jeelani, 2020; Yas, Shahrani Korani & Zare Jouneghani, 2020).

Among the polymer nanocomposites, those based on polyethylene (PE) have attracted considerable interest because PE is one of the most important commercial polymers because of its low price and attractive combination of good processability, mechanical properties, and chemical resistance. It is well known that LLDPE has superiority in properties compared to conventional LDPE, for example, greater tensile strength, higher environmental stress crack resistance, etc (Mizushima, Kawamura, Takahashi & Nitta, 2012). Recently, LLDPE has been used as a blend with other polyethylenes, namely, HDPE/LLDPE (Oliveira, Freitas, Araújo, Cavalcanti, Câmara, Agrawal & Mélo, 2020) and LDPE/HDPE (Laria, Gaggino, Kreiker, Peisino, Positieri & Cappelletti, 2020), which have been investigated by various researchers. It is necessary for polymer processing and shaping of equipment design that the rheological properties of polymer melts are understood. Over the recent decade, the melt flow properties of HDPE/LLDPE blend melts have been conducted (Shaidullin, Salakhov, Borisenko, Tavtorkin & Nifant'ev, 2020).

Inorganic fillers are often compounded into thermoplastic polymers not only to improve morphology and mechanical properties but also to reduce product cost. Calcium carbonate (calcite) plays an important role in the thermoplastic industry as a reinforcing agent for fillers. It has a low price, good availability, wide particle size, easy compounding, light color, easy dyeing, and various possibilities of surface treatment. $CaCO_3$ particles tend to strongly agglomerate because of their high surface energy and reactivity with atmospheric moisture, which forms hydroxyl groups on their surface. $CaCO_3$ particles are hydrophilic and highly polar, whereas many common polymers such as polyolefins are nonpolar and hydrophobic. Consequently, $CaCO_3$ particle surfaces are often modified for better compatibility and adhesion with fillers and the polymer matrix, characteristics that are necessary to obtain better mechanical properties. Because it reacts readily with fatty acids and their salts, $CaCO_3$ is usually surface-modified with stearic acid, which, among other advantages, is inexpensive and easy to process (Cao, Daly, Clémence, Geever, Major, Higginbotham & Devine, 2016). It is well recognized that in addition to such particles the viscosity of the composites is significantly increased. Several studies show that the viscosity of calcium carbonate-filled thermoplastic composites has increased (de Oliveira, Moreno, de Sousa, Escócio, Guimarães & da Silva, 2020; Z. Wu, Zhang & Mai, 2020), but in some cases, this refers to the increase in the value of a wide range of yields at a lower shear rate. Extensive flow observations for this composites have been reported in few studies (Rajamuneeswaran, Vairamuthu, Nagarajan, Stalin & Jayabal, 2020). Wang et al (Wang, Lu & Wang, 1997) used a modifier consisting of carboxylated PE and calcite grafted with acrylamide in high-density polyethylene (HDPE) to improve the mechanical properties of the composite. However, the studies on the physical properties of inorganic particulate-filled HDPE/LLDPE composites have been relatively few.

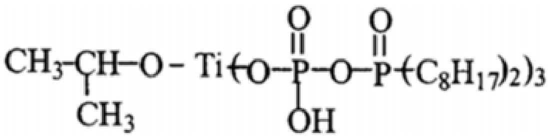
To our knowledge, no studies on HDPE/LLDPE nanocomposites filled with surface-treated nano-calcite particles have been reported. In our research, nano-calcium carbonate particles are used to fill HDPE/LLDPE blend, and the influences of nano-calcium carbonate particles content on the morphology, mechanical, as well as flow properties of the composites. We used the most common polymers and filler materials to process by the most common method in practice. This can significantly improve the property of polymer composites without increasing production costs and without changing processing technology and equipment, thus having wide application prospects.

2. Experimental Details

2.1 Materials

Polyolefins used in this study are HDPE and LLDPE supplied by SK Corporation, South Korea. The nano-calcium carbonate used in this study was kindly supplied by SK Corporation (South

Korea) with a density of 2.71 g/ml, and a particle diameter of 40 to 60 nm. It was coated with a titanate coupling agent for better dispersion. Titanate coupling agent, including isopropyl tri-(dioctylpyrophosphato) titanate (JN114), with its structure shown in Figure 1, was provided by Changzhou City Jinai Co., Ltd, Jiangsu Province, China, and other agents were commercial grades and used as received. An anti-oxidant, Irganox B225 (Ciba, Basel, Switzerland; 0.5 wt%) was added as the stabilizer (synergistic processing and long-term thermal stabilizer system) to the polyethylene during compounding. The information of the materials used in this study is listed in Table 1.



$$CH_3-\underset{\displaystyle CH_3}{\underset{|}{C}}H-O-Ti(O-\overset{\displaystyle O}{\overset{\|}{\underset{\underset{\displaystyle OH}{|}}{P}}}-O-\overset{\displaystyle O}{\overset{\|}{P}}(C_8H_{17})_2)_3$$

Figure1: Structure of the JN-114 titanate coupling agent

Table 1: Characteristics of polyethylene used in this study

Olefin (grade name)	Density (g/cm^3)	MI (g/10 min)	HDT (°C)	Code
HDPE (3300)	0.954	0.8	123	HDPE
LLDPE (FT810)	0.918	2.1	98	LLDPE

HDPE: High-density polyethylene; LLDPE: linear low-density polyethylene; MI: melt index; HDT: heat distortion temperature.

2.2 Surface Treatment of Nano-CaCO$_3$

Unmodified nano-calcium carbonate (nCC) was prepared by drying its water suspension in an oven at 100°C to constant weight. We added 100 g nano-CaCO$_3$ particles and 400 g ethanol to a high-speed dispersion machine filled with zirconium oxide beads and ground for 1–2 h at a speed of 400 rpm. Ground nanoparticles were filtered to remove zirconium oxide beads, titanate coupling agent (0.5 wt% for the filler) was introduced. The mixture was kept at 80°C for 2 h and was filtered to remove the solvent and then the mixture was dried at 120°C for 2 h to remove more residual solvent. After being ground by air current, treated nCC particles were prepared.

2.2.3 Preparation of Nanocomposites

Before melt mixing, HDPE, LLDPE, and surface-treated CaCO$_3$ nanoparticles were dried in a vacuum oven at 80°C for 6 h and then cooled down to room temperature. The materials were stored before processing in a desiccator. After the surface of nCC particles was treated with a titanate coupling agent, the filler particles with HDPE/LLDPE (50/50 wt%) blends were compounded at the resin melting state in a twin-screw extruder (Brabender plasticoorder, model: PLE-331). The mixing chamber capacity being 30 ml, Figure 2. The processing temperature, rotor speed, and blending time were set at 120-200°C, 60 rpm, and 10 min, respectively. The weight fractions (ϕ) of the filler were 0, 5, 10, 15, and 20 wt%, respectively. The sample weight of each mixture was controlled at 50 g. After 10 minutes the Brabender equipment was open in the mixing chamber and the resulting mixture was taken out. The resultant mixture was compression-molded in a hot press at 190°C for 5 min without any applied pressure. After this period, 10 MPa pressure was applied for 5 min, and then the press platelets, containing coils for fluids. Finally, the pressure was free and the mold was removed from the plate. This was followed by cooling to room temperature between two thick-metal blocks kept at room temperature. A template frame was used to ensure a constant film thickness (1 mm). Samples are cut to standard sizes and sized to test mechanical properties. There were two composites, HDPE/LLDPE filled with unmodified nCC particles, namely UnCC, and HDPE/LLDPE filled with surface-modified nCC particles, namely MnCC, in this experiment. The samples used for this study were prepared using twice-mixed composite materials for better blending. The specimens were then sealed in plastic bags as they waited for the processing and analysis.

2(a) 2(b)

Figure 2: (a) General view Brabender Plasticorder PLE 331 (b) Mixing chambers and rotors at the end of the mixing process

2.2.4 Characterization

2.2.4.1 Morphological Observations

Scanning Electron Microscopy (SEM; JEOL, Japan JSM- 6360LV) was employed to study fracture surfaces of all tested samples. The SEM samples were placed in liquid nitrogen for 30 min and then cut into two pieces. The fractured surfaces of the specimen were coated with a thin layer (10–20 nm) of gold-palladium.

2.2.4.2 Mechanical Properties

The effects of nCC on HDPE/LLDPE (50/50 wt%) mixtures were assessed by mechanical properties such as the tensile stress, elastic modulus, and Izod impact strength. Standard specimens were sampled from the compression molded sheet and then conditioned at the temperature of (25 ± 2)°C and the relative humidity of 50 ± 5% for 24 h. The tensile was tested in a screw-driven universal testing machine (Instron 4466) equipped with 10 kN electronic load cells and mechanical grips. The tests were conducted at a crosshead speed of 30 mm/min and data was acquired by a computer. All tests were carried out according to the ASTM standards, and five replicates were tested for each sample to get an average value. Izod impact tests of notched samples were carried out according to ASTM D256-93a standard, the instrument was Ceast pendulum impact tester (Model 6545/000). The dimension of the specimens (length × width × thickness) was 63.50 × 13 × 3.20 mm^3.

2.2.4.3 Flow Property Test

The melt-flow index (MFLs) of the nanocomposites were determined with a Zwick 4100 MFI test instrument according to the ISO 1133 (ISO, 2011) method. A load of 2.16 kg at 230°C was used in the measurement.

3. Results and discussion

3.1 Dispersion of Nano-$CaCO_3$ Particles in Polyethylene Matrix

It is known that the mechanical properties of composites are strongly related to the dispersion of the filler in the polymer matrix. As one kind of inorganic filler, $CaCO_3$ is incompatible with PE. When the nanoparticles are used the dispersion becomes a severe problem because the nanoparticles have a strong tendency to agglomerate. Figure 3 shows SEM micrographs of impact-fractured surfaces of the HDPE/LLDPE/10 wt% unmodified nano-$CaCO_3$ (UnCC) and HDPE/LLDPE/10 wt% modified nano-$CaCO_3$ (MnCC) composite specimens. The untreated nCC particles aggregated severely in the PE matrix with the size reaching up to 300 nm (Fig. 3a). Many cavities between the particles and the matrix were observed, which suggests that the interfacial adhesion between the two phases was poor. The untreated nCC particles aggregate in the PE matrix because of their high polar surface energy. The aggregated nCC particles have poor compatibility with the PE matrix because of their hydrophilic surfaces, which lead to cavities in the matrix and interface debonding. When the PE matrix was filed with the nCC particles treated with JN114, the nanoparticles were homogeneously dispersed in the PE matrix, and aggregates of the nanoparticles were hardly observed; the diameters of the particles were almost below 100 nm. The interfacial contact between the nanoparticles and PE matrix was good. The nCC particles treated with JN114 have a strong polar-polar interaction with the PE matrix through the pyrophosphate groups. As a result, good compatibility was obtained, thereby resulting in an excellent interfacial adhesion between the nCC particles and the matrix. The good compatibility also improved the dispersion and reduced the aggregates of nCC particles in the PE matrix.

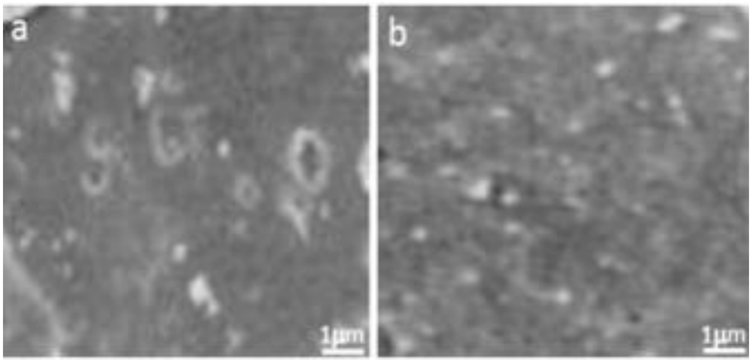


Figure 3: SEM micrographs of (a) HDPE/LLDPE/10 wt % unmodified-nCC composite and (b) HDPE/LLDPE/10 wt% modified-nCC composite specimens

3.2 Mechanical Properties of the Composites

3.2.1 Tensile Fracture Strength

Figure 4 shows the relationship between the tensile fracture strength (σ_b) and the content of nCC. For MnCC specimens, the tensile fracture strength (σ_b) decreases slightly with an addition of the filler content when the weight fraction of nCC (ϕ) is less than 15 wt%, while σ_b increases obviously when ϕ is more than 15%. Similarly, for UnCC specimens, σ_b decreases obviously with addition of ϕ when ϕ is less than 15%, and then increases somewhat. It appears that the minimum tensile fracture strength occurs at ϕ = 15% in the range of ϕ from o to 20%. It means that that there is no reinforced effect in these filled systems at a low concentration of the filler. With further increase in particles, the effect of stress concentration between the matrix and the interface and inclusions must be consistently significant, and the particles may absorb relevant tensile deformations or cracks to convince the yield first and crazes around the matrix, leading to improved tensile fracture toughness. In this case, the tensile fracture strength increases somewhat. It is known that the tensile strength of composites is influenced by the filler fraction and the interfacial adhesion between particles and the matrix (Pukánszky & Fekete, 1999). For spherical particles with no adhesion to the polymer matrix, which fail by random fracture, Liang and Li (Liang & Li, 1998) proposed a tensile strength equation as follows:

$$\sigma_c = \sigma_m (1- 1.21 \mathrm{Sin}^2\theta \ \phi^{2/3}) \qquad (1)$$

Where θ is the angle of interfacial adherence between the filler particle and the matrix, which is an angle of interfacial debonding of the particle from the pole. In other words, this is a parameter for a characteristic of interfacial bonding.

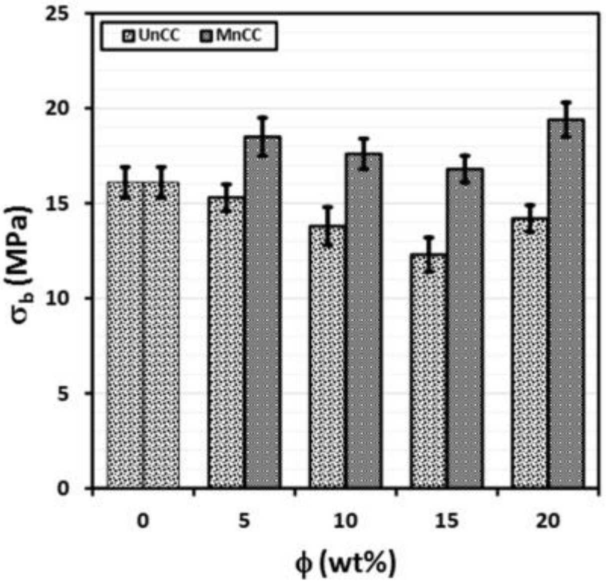


Figure 4: Tensile fracture strength vs. filler weight fraction

For a particulate filled polymer composite system with weak interfacial adhesion, its tensile strength will decrease obviously (Nicolais & Narkis, 1971) or will be slightly reduced for a fill system with relatively good interfacial adhesion with the addition of relatively inorganic particle concentration (Liang & Li, 1998). For instance, in a case of good interfacial adhesion, $\theta = 0$, and then $\sigma_c = \sigma_m$. Therefore, it might be concluded from the results shown in Figure 4 that the interfacial adhesion between the nCC particles and the matrix is good, especially for MnCC specimens, which is consistent with the SEM explanations.

3.2.2 Tensile Elastic Modulus

Figure 5 shows the variation of tensile elastic modulus (E_c) of the composites with nCC content. It can be seen that the tensile elastic modulus increase with increasing filler content. It is generally known that the addition of any rigid filler to a polymer matrix increases its modulus. Furthermore, for the same content of nCC, the modulus of composite increases with the amount of coupling agent (0.5 wt%) used in the surface treatment. We attributed this to the improved dispersion of nanoparticles and the strong interfacial adhesion between nCC particles and the polyethylene matrix, consistent with the conclusions based on the SEM photomicrographs. According to Goujon et al. (Goujon & Mutaftschiev, 1976), $CaCO_3$ contains a small amount of $Ca(OH)_2$ or other oxide and hydroxide impurities, which may react with the organofunctional groups of the coupling agent. Thus, the increased modulus may be attributed to the adhesion of titanate to the surface of $CaCO_3$, via the reaction with the reactive impurities present at the surface of the filler.

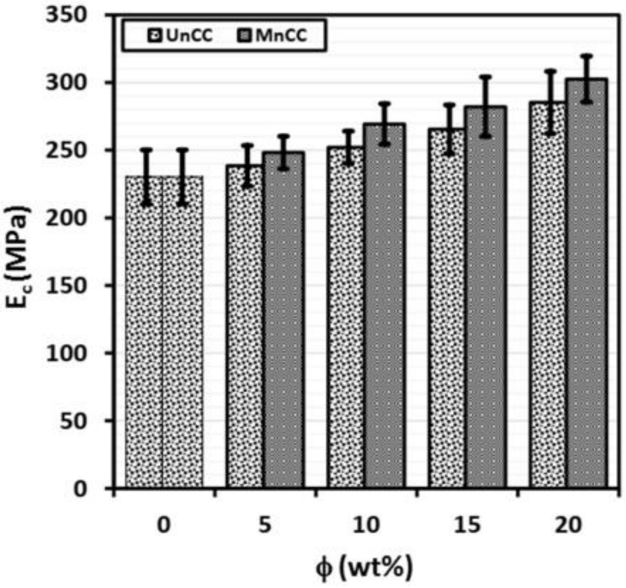


Figure 5: Tensile plastic modulus vs. filler weight fraction

3.2.3 Impact Strength

Figure 6 shows the variations in the Izod impact strength (S_{IC}) of the composites with nCC content. It can also observe from Figure 6 that when $\phi < 10$ wt%, S_{IC} increases with increasing ϕ and then decreases. There is a maximum value of S_{IC} at $\phi = 10\%$. Impact strength is an important characteristic for the impact toughness of materials. The results show that the impact toughness of polyethylene can be improved to some extent when polyethylene is filled with a suitable concentration of nCC. This peak phenomenon of the curve of S_{IC} versus ϕ can be explained as follows: the nCC fillers will act as inclusions that produce stress concentration will cause matrix yielding and plastic deformation which will absorb part of the impact energy and result in enhancing impact toughness correspondingly. With further increasing the content of nCC the distance between the neighboring particles (the thickness of matrix ligament) will be smaller than the critical value, L_c, which is giving by (S. Wu, 1985):

$$L_c = d\,[(\pi/6\phi)^{1/3}-1] \qquad (2)$$

In this case, the interfacial layer between the filler and matrix will change from the plane strain to plane stress and the ductile-brittle transition will occur, thus the impact strength of composites decreases accordingly. In Eq. 2, d is the diameter of the filler particle and ϕ is the volume fraction of the filler. When ϕ = 20%, the S_{IC} had already decreased to the level of polyethylene, and the maximum S_{IC} of UnCC was 3.4 kJ/m^2. By contrast, the maximum S_{IC} of MnCC was 4.8 kJ/m^2, about 167% of that of polyethylene. As expected, in contrast with nCC, the treated with the JN114 had a more obvious toughening effect on polyethylene. The JN114 that covered nCC, therefore, had a special modification ability to improve the impact strength of polyethylene.

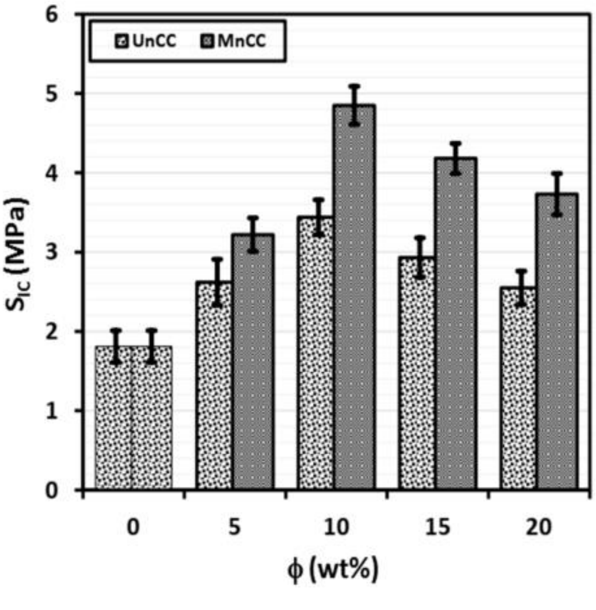


Figure 6: Izod impact strength versus filler weight fraction

3.2.4 Flow Properties

Melt flow index (MFI) is an important parameter for the characterization of the processing property of polymeric materials. Figure 7 displays the MFI with nCC weight fraction (ϕ) for the nanocomposites of HDPE/LLDPE/nCC untreated and treated with a coupling agent. Because the incorporation of fillers hinders plastic flow and increases the viscosity of a polymer melt, a reduction of MFI with the filler loading is expected. The MFI values decreased in direct proportion to the increase in the amount of nCC. With the 0% nCC point considered as the reference point, the MFI value decreased significantly with the addition of 5% nCC. A significant decrease was observed in the 5-15% range, and an insignificant decrease was observed in the 15-20% range. The chemical modification of the filler using the coupling agent causes an increase in the viscosity which leads to a decrease in the melt flow index. This decrease is a result of the interactions that developed between the filler and the coupling agent, reducing, therefore, the mobility of the chains around the filler. Increasing the amount of nCC in composite material increased the shear stress and viscosity. The polymer flow into the mold decreased according to the filler concentration. Therefore, the polymer processes were affected by the influence of particulate materials on the flow properties of the material. The flow properties could also be adversely affected by numerous phenomena related to the presence of the filler in the formulations (Abd El-Rahman, Ali, Khalil & Kandil, 2020).

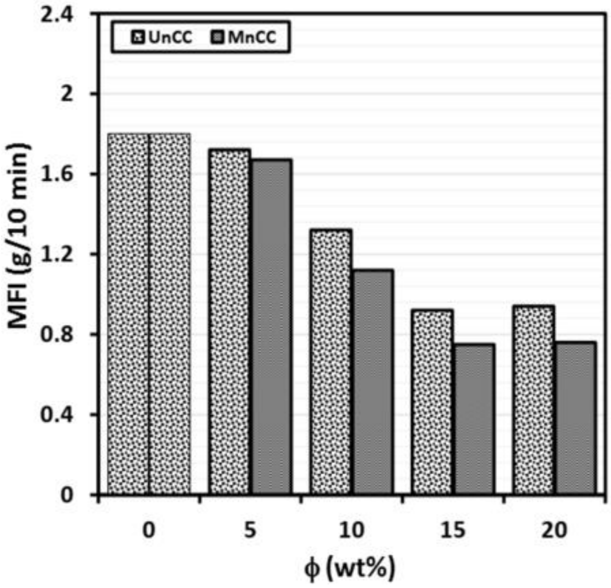


Figure 7: Effects of filler loading on MFI

4. Conclusions

High-density polyethylene/linear low-density polyethylene (HDPE/LLDPE) blends with 0.5 wt% Irganox B225 containing different amounts of nCC filler were prepared by melt-blending with a twin-screw extruder. In morphology, the effects of nCC material, tensile properties such as tensile fracture strength, elastic modulus, and Izod impact strength, and flow characteristics of the filled HDPE/LLDPE (50/50 wt%) mixture were investigated. The following results were obtained:

(1) SEM analysis indicated clearly that the titanate-treated nCC particles were homogeneously dispersed in the PE matrix on the nanoscale, and their interfacial adhesion with the matrix was superior to those of the untreated ones. These changes greatly modify the mechanical properties of the composites.

(2) With increasing the content of nCC the moduli of UnCC and MnCC composites increase, while the tensile strength decrease. When $\phi < 15$ wt%, the tensile fracture strength

decreases with the addition of ϕ and then increases obviously. It appears that the minimum tensile fracture strength occurs at ϕ = 15% in the range of ϕ from 0 to 20%.

(3) when ϕ < 10%, the Izod impact strength of the notched specimens enhances with increasing ϕ, then drops down gently. The maximum impact strength occurs at ϕ = 10% in the range of ϕ from 0 to 20%.

(4) The MFI reflects the flow property of thermoplastics. Under the standard test conditions, the MFI of the composites decreases with an addition of ϕ of the nCC particles.

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