

## Morphology and Mechanical Properties of PP/LLDPE Blends and Ternary PP/LLDPE/nano-CaCO<sub>3</sub> Composites

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## Морфологические и механические свойства смесей на основе ПП/ЛПЭНП и тройных композиционных материалов на основе ПП/ЛПЭНП/нано-CaCO<sub>3</sub>

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*Для получения полипропиленовых/линейно полиэтиленовых с низкой плотностью/наноразмерных CaCO<sub>3</sub> тройных композитов с соответствующими механическими и морфологическими свойствами используется двухшнековый экструдер. Исследовано влияние модифицированного линейного полиэтилена с низкой плотностью и объемного содержания нано-CaCO<sub>3</sub> на предел прочности при растяжении, модуль Юнга и поглощенную энергию удара композитов. Также исследовано влияние взаимодействия между частицами наполнителя и полимерной матрицей. Рассмотрено влияние количества микротрещин на механические свойства композитов при растяжении. С помощью сканирующего электронного микроскопа исследовано влияние нано-CaCO<sub>3</sub> с низкой плотностью на количество микротрещин и разрушенную при ударе поверхность полипропиленовых композитов. Для определения возможных механизмов улучшения сопротивления разрушению проведена сканирующая электронная микроскопия морфологии разрушенной при ударе поверхности. Результаты показали, что предел текучести смесей на основе полипропиленовых композитов с низкой плотностью и композитов на основе полипропиленовых композитов с низкой плотностью и нано-CaCO<sub>3</sub> с плотностью менее 10% постепенно уменьшается при незначительном увеличении модуля Юнга с повышением нагрузки. С ростом содержания CaCO<sub>3</sub> в обоих нанокompозитах значение ударной вязкости увеличилось.*

**Ключевые слова:** микротрещина, ударная вязкость, нанокompозиты, карбонат кальция.

**Introduction.** Polypropylene (PP), as one of the most important commodity polymers, is widely used in many applications [1]. However, its application is limited due to its high shrinkage rate and relatively poor impact resistance at room or low temperatures. Therefore, to improve impact toughness of PP, it has received extensive attention [2]. Recently, blending of various rubbers with PP for improving impact resistance has been studied by several researchers [3–6].

Bertin et al. studied a virgin and recycled linear low density polyethylene (LLDPE)/PP blends [1, 3]. Khare et al. performed thermal and dynamic analysis on

metallocene linear low density polyethylene (LLDPE)/PP blends to optimize impact properties [3, 7]. Also, other researchers have investigated mechanical properties, especially impact behavior of PP/polyethylene (PE) blend and found that, by adding PE to PP, impact resistance of PP increases [3, 8–10]. The incompatibility between LLDPE and PP has been already reported by various authors [11–13]. Low interfacial adhesion between the phases is responsible for the decrease in mechanical properties, especially related to its morphology, including impact strength, strain at break and ductile to brittle transition [11].

According to Shanks [11, 14], immiscibility between the phases makes the rule of mixtures ineffective in predicting some properties of interest. To overcome this difficulty, use of various coupling agents has been reported. Younesi et al. [3] showed that addition of high crystallizable, high density polyethylene (HDPE) improved tensile properties. Also, modification of PP/LLDPE interface increased impact absorbed energy dramatically for these composites with all contents of LLDPE [3]. Recently, three (and more) phase polymer composites have also attracted the attention of researchers and interest of the industry, which have been studied in an effort to design materials with novel properties or to improve the already existing ones [3].

The tremendous interfacial area in a polymer nanocomposite helps to affect the composites' properties to a great extent, even at low filler volume fractions [1, 15]. However, homogeneous dispersion of nanoparticles is very difficult because nanoparticles with their high surface energy are easy to agglomerate. Calcium carbonate ( $\text{CaCO}_3$ ) is one of the most commonly used inorganic filler in PP. Many researchers have studied mechanical properties of PP/ $\text{CaCO}_3$  nanocomposites [1]. Thio et al. [16, 17] used three types of  $\text{CaCO}_3$  nanoparticles with average diameters of 0.07, 0.7, and 3.5  $\mu\text{m}$  to toughen PP. It was reported that the 0.7  $\mu\text{m}$  diameter particles improved Izod impact strength up to four times with respect to the unfilled matrix. The major toughening mechanisms were interfacial debonding and plastic deformation of inter-particle and crack deflection.

Wang et al. [18] reported that mechanical properties of PP, especially ductility, were effectively improved by the incorporation of nano- $\text{CaCO}_3$  pretreated with stearic acid in an ultra-high-speed mixer (6000 rpm). Yong Lin et al. [19] aimed to improve nano- $\text{CaCO}_3$  dispersion in the polymer matrix and thus coated them with stearic acid to increase their compatibility with the PP matrix and to minimize the interactions among the particles. Ghasemi et al. [20] reported that increasing the calcium carbonate nanoparticles improved both impact strength and the Young modulus of PP; also, the more the PP-g-MA added to PP matrix, the more the increase of impact strength of the samples and the less the decrease in their Young's modulus. Ghasemi et al. [21] also studied stiffness and damping properties of PP/ $\text{CaCO}_3$  nanocomposites and showed that, by increasing the nanoparticles' weight percent, tensile strength of the nanocomposites decreased and their Young's modulus increased.

In this work, evaluation of tensile and impact properties of PP/LD blends and PP/LD/nano- $\text{CaCO}_3$  was carried out to investigate composition range for better mechanical performance. For compatibility of LLDPE with PP, HDPE was added to LLDPE in order to result in modified linear low density polyethylene (LD). By simultaneous change of composition of the matrix (PP/LD) and nano- $\text{CaCO}_3$

weight percentage, effects of composition on final properties of the composites were investigated. The main objective of this paper was to investigate effect of matrix composition (PP/LD) on the interaction between the particles and matrix. Furthermore, effect of microcracks on tensile properties and fracture toughness of the composites was studied.

### 1. Experimental Procedure.

1.1. **Materials.** PP (polypropylene, HP550j), LLDPE (linear low density polyethylene, LL0209AA) and HDPE (high crystallizable high density polyethylene, HD6070EA) of these ternary composites were obtained from Arak Petroleum Company, Iran, with melt flow index of 3, 0.9, and 7 g/10 min, respectively. CaCO<sub>3</sub> nanoparticles, with trade name of HAKUENKA CCR, were provided by Omya GmbH Company and the mean diameter of the particles was 80 nm.

1.2. **Surface Treatment of the Nanoparticles.** To improve their dispersion in the polymer matrix, the as-received CCR nanoparticles were further coated with 2.5 wt.% stearic acid to increase its compatibility with the PP matrix and to minimize the interactions among the particles. The coating method was as follows. First, 100 g of CCR was mixed with a 400 ml solution mixture of water and ethanol in the volume ratio of 3:1. The suspension was stirred by a magnetic stirrer for 2 h to completely wet the particles. Then, the suspension was heated to and maintained at 80°C. A fixed amount of stearic acid dissolved in the ethanol solvent was gradually added to the suspension drop by drop. After 2 h of reaction time, surface-treated calcium carbonate particles, which settled at the bottom of the beaker, were dried in an oven at 105°C overnight [19].

1.3. **Specimen Preparation.** LLDPE and HDPE interface modifiers were first melt-mixed in 1:4 ratio of HDPE to LLDPE; then, modified LLDPE and CCR were directly melt-blended with PP. Compounding of the materials was done using a Werner & Pfleiderer, Coperion EpcNTL (ZSK25) twin screw extruder. In the extrusion step, barrel temperatures were set at 170/175/185/190/185°C and screw speed of 300 rpm was used. L/D ratio of the screws was 40. After compounding, the blends were injection moulded into rectangular bars and dumbbell shaped specimens using an Emen machine injection moulding machine. The barrel had a flat temperature profile of 180°C with injection pressure of 105 bars. A single-edge V-shaped notch of 2 mm depth and tip radius of 0.25 mm was milled in the moulded specimens for the notched Izod impact experiments.

1.4. **Mechanical Properties.** Tensile tests were performed by a Zwick/Roell machine at crosshead speed of 50 mm/min. Tensile tests were done according to ASTM-D638. Notched Izod impact tests were conducted using a CEAST impact tester at room temperature using ASTM-D256. At least five samples were conducted for each property and mean values and standard deviations (SD) were calculated.

1.5. **Scanning Electron Microscopy (SEM).** Morphologies of both binary and ternary PP composites were observed by a Hitachi S-4160, SEM with accelerating voltage of 25 kV. To evaluate dispersion quality of the CaCO<sub>3</sub> nanoparticles and also extents of microcracks, 7 freeze-fractured surfaces of the nanocomposites obtained at liquid nitrogen temperature were examined; so were the impact-fractured surfaces in order to assess failure mechanisms. All the specimens were coated with gold before SEM observations.

## 2. Results and Discussion.

**2.1. Microcracks.** Frequently, microcracks are created in the manufacturing processes. It is crucial that microcracks reduce elastic modulus locally, resulting in the crack attraction by the particle. When the crack approaches the particle, the crack tip is partially unloaded because microcracks in their vicinity open as well. If microcracks are not restricted to the vicinity of the crack tip or the particles but are homogeneously distributed throughout the volume, they globally reduce Young's modulus. In a stress-controlled situation, the stored elastic strain energy increases. Considering that all the samples were made in the same conditions, it seems that the quantity of microcracks strongly depended on their composition. SEM micrographs in Fig. 1 show the cryo-fractured surfaces of the nanocomposite. By adding  $\text{CaCO}_3$  nanoparticles to the PP/LD blends, quantity of microcracks increased (Fig. 1). Observation of the figures also showed that, for the LD10 wt.% as a distinct behavior, nanocomposite exhibited the lowest quantity of microcracks in comparison to other blends. It seems that compatibility between the components was the reason for decreased quantity of microcracks in the nanocomposite.

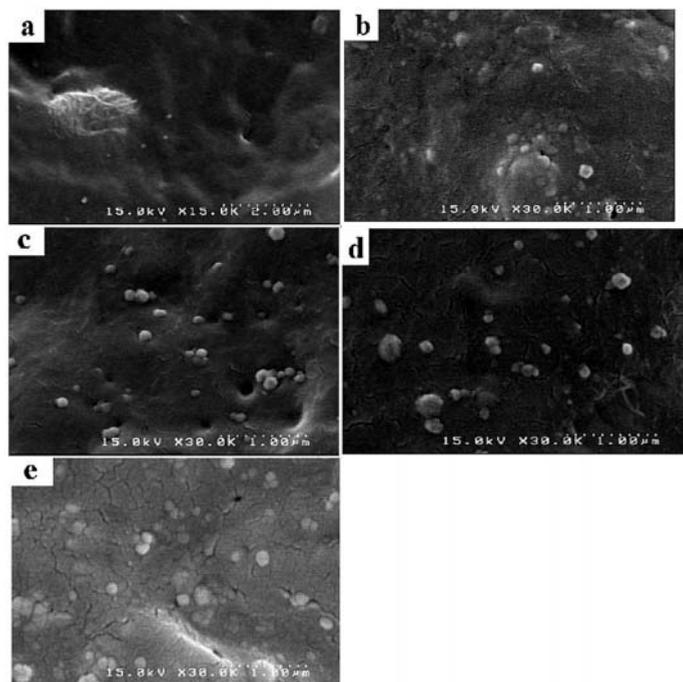


Fig. 1. Cryo-fractured surfaces of the PP/LD/CCR nanocomposites: (a) 90/10/0, (b) 90/5/5, (c) 85/10/5, (d) 75/20/5, (e) 65/20/15.

**2.2. Tensile Properties.** Effect of the interaction between filler particles and polymer matrix and quantity of microcracks on the tensile properties is discussed in this section. Figure 2 shows the relationship between elastic modulus and weight fraction of the fillers. The modulus is a low strain property. In this low strain regime, the adhesion between particle and polymer remains intact. The interaction strength between polymer and particle has little effect on the modulus [5]. The elastic modulus shows a maximum at 10 wt.% of LD content. If microcracks are

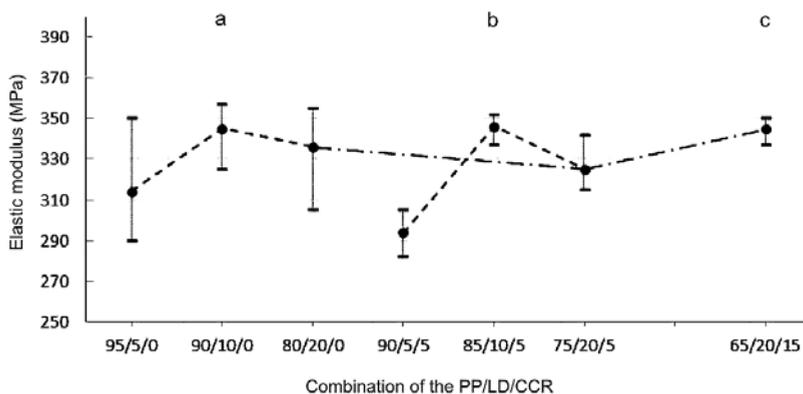


Fig. 2. Elastic modulus of PP composites: (a) CCR = 0, (b) CCR = 5%, and (c) CCR = 15%.

homogeneously distributed throughout the volume, they globally reduce Young's modulus [22]. As seen in Fig. 1a and c, the lowest quantity of microcracks was observed in the composites with LD content of 10 wt.%; so, the increasing Young's modulus can be predicted.

Effect of adding  $\text{CaCO}_3$  nanoparticles to Young's modulus of the PP/LD blends can be studied from two viewpoints. First, by adding the solid particles such as calcium carbonate, the Young's modulus improves slightly. It has been reported by other investigators [1, 16, 22], on the other hand, that addition of  $\text{CaCO}_3$  nanoparticles increases quantity of microcracks of PP/LD blends, which results in decreased Young's modulus. The results of tensile tests show that addition of 5 wt.%  $\text{CaCO}_3$  nanoparticles to PP/LD decreases Young's modulus. This result is caused by growth of microcracks. It can be also observed that, for the LD10, a distinct behavior, insignificant quantity of microcracks explains this behavior. Moreover, the modulus of the nanocomposites slightly improves by further increase of the filler content. The yield stress is measured at considerable deformations, which leads to complete different dependency of properties on blend composition, quantity of microcracks and particle-polymer interaction [22].

At this strain level, the particles completely or partially debond from the polymer matrix, which leads to lowering of the yield stress through the formation of voids, and do not contribute to the stress level. The tensile yield stress is raised if the particles do not debond from the polymer surface, as would be the case when the adhesion is very high [5].

Effect of increasing the LD content on yield strength of the PP/LD/CCR nanocomposites can be studied from a different point of view:

Occurrence 1: Yield strength of PP/LD blends decreases by increasing the LD content according to the rule of mixtures [2].

Occurrence 2: Microcracks reduce yield strength. Therefore, when LD content is lower than 10 wt.%, microcracks decrease by increasing LD content. When LD content is higher than 10 wt.%, microcracks increase again. Therefore, yield strength first increases until LD 10%; then, it decreases.

Occurrence 3: The polymer-particle adhesion increases by enhancement of the LD content. When the adhesion is low, early debonding can occur and, as a result, the yield strength decreases.

Figure 3 shows the relationship between yield strength and weight fraction of components for these two composite systems. In Fig. 3a, it can be seen that the yield strength of PP/LD blends decreased by increasing LD content since, in the first case (Fig. 3a) when the weight percentage of nanoparticles was zero, only the strength originated from occurrences 1 and 2 dominated. When LD content was up to 10 wt.%, occurrence 1 was more effective than occurrence 2, which resulted in diminishing of the yield strength. When LD was greater than 10 wt.%, by increasing LD content, both occurrences 1 and 2 reduced yield strength; so, loss of strength occurred more quickly.

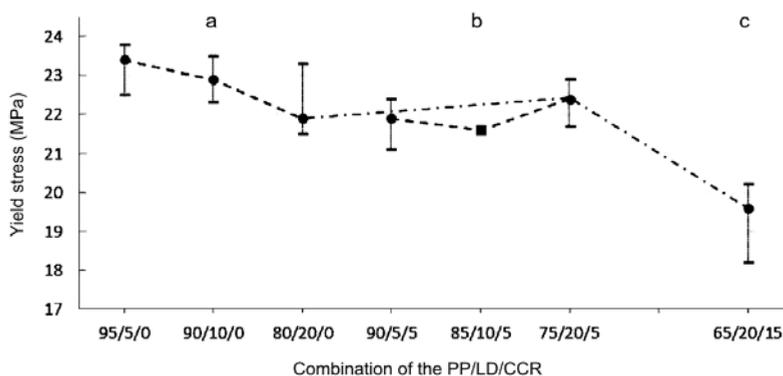


Fig. 3. Yield strength of PP composites: (a) CCR = 0, (b) CCR = 5%, and (c) CCR = 15%.

Figure 3b shows that, by increasing LD content, yield strength decreases slightly as LD content is less than 10 wt.% while increasing if LD content is more than 10 wt.% [22]. By adding the  $\text{CaCO}_3$  nanoparticles to the composition of PP/LD, effect of occurrence 3 should be considered along with that of occurrences 1 and 2 by the variation of yield strength.

In the nanocomposite with LD content of 10 wt.% and less than 10 wt.%, modulus of the nanocomposite increased while yield stress was reduced by LD content (Fig. 3b). This lowering of yield stress was connected to the early debonding of the filler particles from the PP/LD polymer matrix because the debonded particles did not contribute to the yield stress [22]. When the LD percentage was less than 10 wt.%, it was not enough for preventing debonding of the nanoparticles from the matrix; therefore, occurrence 3 had little effect in this stage. Therefore, decreased yield strength can be explained by occurrences 1 and 2.

In the nanocomposites with more than 10 wt.% of LD content, it is believed that the adhesion between the filler particles and PP/LD matrix was strong enough; so, nanoparticles could bear the same fraction of the external load and the particles did not early deboned from the matrix. Thus, the yield strength of the composite increased [1]. In other words, in the nanocomposite with LD content of greater than 10 wt.%, strength of occurrence 3 was dominated in comparison with occurrences 1 and 2.

Figure 3c shows yield strength of the nanocomposites for the high CCR contents. The yield strength decreased by increasing the CCR contents. It is supposed that increasing of the nanoparticles, aggregate and microcracks led to this reduction.

The dispersion of CCR was very difficult because nanoparticles with high surface energy were easy to agglomerate. The agglomerates were easy to debond from the matrix and the debonded nanoparticles did not bear any fraction of the external load; therefore, the yield strength decreased [1]. Effect of LD and CCR on other tensile properties is shown in Table 1. By increasing LD content, a slight decrease happened in fracture strength and elongation at break [1]. In combination of the PP/LD/CCR (75/20/5), adhesion and compatibility between the particles and matrix prevented early debonding of particles and led to enhancement of break strength. Furthermore, increase of the CCR content resulted in slight decrease of the fracture strength [3, 8].

T a b l e 1

**Mechanical Properties of PP Nanocomposites**

Composition (weight ratio)			Elongation at break (%)		Break strength (MPa)	
PP	LD	CC-R	Mean	SD	Mean	SD
95	5	0	15.4	0.6	21.4	0.9
90	10	0	15.6	1.3	19.2	0.9
80	20	0	16.7	1.6	19.0	0.9
90	5	5	17.8	1.7	19.1	1.3
85	10	5	14.2	1.6	18.2	1.2
75	20	5	14.5	0.8	20.3	0.6
65	20	15	9.9	1.0	18.8	0.6

**2.3. Impact Properties.** Formation of microcracks during crack propagation can increase fracture toughness owing to additional dissipated energy. Depending on distribution of microcracks, they may either increase or decrease fracture toughness [23]. Figure 4a illustrates the impact absorbed energy for PP/LD blends versus LD content wt.%. As expected, the impact absorbed energy increased by increasing the LD content [3, 4, 6, 7 and 24]. Beside the effect of LD content, quantity of microcracks can also affect impact strength [23]. Since the PP/LD10 blends have the least quantity of microcracks, PP/LD10 blends had the minimum of impact strength. This condition can be concluded from Fig. 4a too.

Moreover, LD increase up to 20 wt.% led to improvement of the impact strength which originated from synergistic toughening effect of both LD and microcracks. Figure 4b illustrates the impact absorbed energy of PP/LD/CCR nanocomposites versus LD content (wt.%). Impact strength of nanocomposites increased slowly by increasing LD content [3, 4, 6, 7, 24]. In addition, changes in interfacial interactions between the fillers and polymer matrix can modify the deboning mechanism, failure behavior and thus overall performance of the composites [22].

In PP/LD/CCR nanocomposites, increased weight percentage of LD blends can cause changes in interaction between the matrix and nanoparticles. The polymerparticle adhesion is lowered when the LD content decreases. When the adhesion is low, deboning can occur, which is consistent with the cavitation

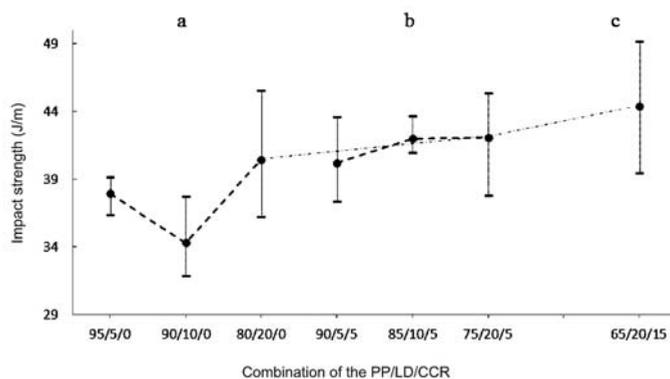


Fig. 4. Impact strength of PP ternary composites as a function of LD content: (a) CCR = 0, (b) CCR = 5%, and (c) CCR = 15%.

mechanism of micro-sized rigid particles as summarized by Zuiderduin et al. [22], consisting of three stages: stress concentration, debonding and shear yielding.

In composites with LD content of 10 wt.% and less than 10 wt.%, due to weak adhesion between the particles and matrix, the filler particles debond from the PP/LD matrix. As a result, in PP/LD/CCR (85/10/5) nanocomposites, because of early debonding of the  $\text{CaCO}_3$  nanoparticles from the PP/LD matrix, fracture energy of the composites stems largely from the shear yielding of the matrix. Strong adhesion and interaction between particles and matrix are not always conducive to toughening [25]. Vollengberg and Heikens [26, 27] reported decrease of impact strength in chalk-filled PP composites because of strong interfacial bonding.

Also, microcracks do not consume large quantities of energy because quantity of the initial microcracks in PP/LD/CCR (85/10/5) nanocomposites is very low. Furthermore, in ternary composites (PP/LD/CCR), incorporation of nanoparticles mildly decreases impact strength of composites (Fig. 4c).

Debonding of the particles creates free volume at the particle size level; therefore, stress state is altered in the vicinity of the particles. This mechanism is similar to cavitation in rubber toughened blends [22]. In addition to the toughening effect of nanoparticles, toughening effect of microcracks should be investigated since adding  $\text{CaCO}_3$  nanoparticles to PP /LD matrix can cause dramatic increase in microcracks.

Figure 5 shows SEM micrographs of the V-notched impact fracture surface morphology of the composites. Two rough surfaces are demonstrated in Fig. 5a and c; in other words, these two blends exhibit ductile fracture. Also in Fig. 5a and c, fracture surface is composed of angular lines which are the path of crack propagation. Having large quantity of microcracks in PP/LD20 blend, the aggregation of the angular lines in fracture surfaces of PP/LD20 was further than PP/LD5 blends. Thus, path length of crack propagation and 16 energy for fracture of composite increased and, consequently, impact strength of the composites increased as well (Fig. 4b) [3].

Figure 5b shows that PP/LD10 exhibited brittle fracture; it may be observed that the fracture surface looked like waves [2]. It means that the PP/LD10 specimen broke relatively fast. As seen in Fig. 1d, there was a large quantity of

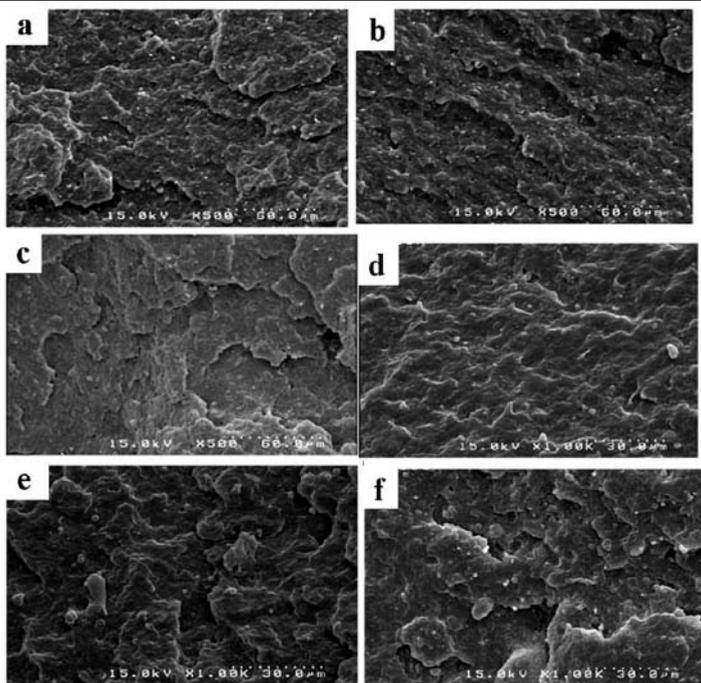


Fig. 5. SEM micrographs of impact-fractured surface of the PP composites: (a) 95/5, (b) 90/10, (c) 80/20, (d) 90/5/5, (e) 85/10/5, and (f) 75/20/5.

initial microcracks in the PP/LD/CCR (75/20/5) samples; so, microcracks were expanded upon loading. Increasing the path length of cracks led to energy dissipation and thereby increased impact strength of the nanocomposite. Furthermore, result of tensile tests indicated that compatibility and high adhesion between the particles and matrix prevented debonding of the particles from matrix in the specific compositions. Therefore, it seems that a slight improvement in the impact strength in the PP/LD/CCR (75/20/5) samples was originated largely from the expansion of microcracks and small fraction of the energy absorption by the shear yielding of the matrix. Figure 5d is the SEM picture of the morphology of V-notched impact fracture surface of the PP/LD/CCR (90/5/5) nanocomposites. The impact-fractured surface was relatively smooth and homogeneous, indicating that the fracture occurred at relatively high speed. In other words, the samples exhibited brittle fracture, which agreed with the minimum impact strength as observed in Fig. 4b. The fracture surface of the PP/LD/CCR (85/10/5) nanocomposites was rougher than that of the PP/LD/CCR (90/5/5) nanocomposites. Two rough surfaces shown in Figs. 5e and 5f imply more ductile failure than the one in Fig. 5d. This means that these two composites exhibited ductile fracture.

**Conclusions.** In this paper, to assess effects of matrix composition on the mechanical properties of composites, a comprehensive experimental study was conducted. Novelties of the present research can be summarized as follows:

1. During the manufacturing process, in all the matrices, microcracks increased, in which the volume was influenced by the composition of composite.
2. SEM images of the samples showed that quantity of microcracks in the PP/LD blends increased by adding the  $\text{CaCO}_3$  nanoparticles.

3. In the components with LD content of 10 wt.%, the lowest microcracks were observed.
4. Increases in the amount of microcracks in material resulted in the reduction of elastic modulus and yield strength of the composite.
5. Elastic modulus of the matrix decreased and increased with respect to adding 5 and 15 wt.% CaCO<sub>3</sub> nanoparticles, respectively.
6. Combinations with LD = 10% had the highest modulus due to the lowest presence of microcracks in this composition.
7. Adding CaCO<sub>3</sub> nanoparticles to PP/LD diminished yield strength.
8. Enhancement of LD content prevented early debonding.
9. The V-notched impact fracture strength of the blends increased by increasing the nano-CaCO<sub>3</sub>.
10. Fracture surface of LD = 10% was smoother than others, indicating low impact fracture strength.
11. Impact strength of composites increased by adding CaCO<sub>3</sub> nanoparticles.

## Резюме

Для отримання поліпропіленових/лінійно поліетиленових із низькою густиною/нанорозмірних CaCO<sub>3</sub> потрійних композитів, що мають відповідні механічні і морфологічні властивості, використовується двошнековий екструдер. Досліджено вплив модифікованого лінійного поліетилену з низькою густиною й об'ємного вмісту нано-CaCO<sub>3</sub> на границю міцності при розтязі, модуль Юнга і поглинену енергію удару композитів. Також досліджено вплив взаємодії між частинками наповнювача і полімерною матрицею. Розглянуто вплив кількості мікротріщин на механічні властивості композитів при розтязі. За допомогою скануючого електронного мікроскопа досліджено вплив нано-CaCO<sub>3</sub> з низькою густиною на кількість мікротріщин і зруйновану при ударі поверхню поліпропіленових композитів. Для визначення можливих механізмів покращання опору руйнуванню проведено скануючу електронну мікроскопію морфології зруйнованої при ударі поверхні. Результати показали, що границя текучості сумішей на основі поліпропіленових композитів із низькою густиною і композитів на основі поліпропіленових композитів із низькою густиною і нано-CaCO<sub>3</sub> із густиною менше ніж 10% поступово зменшується при незначному збільшенні модуля Юнга зі зростанням навантаження. Зі збільшенням вмісту CaCO<sub>3</sub> в обох нанокompозитах значення ударної в'язкості зросло.

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