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The Effect of Oleic Acid-Grafted Linear Low-Density Polyethylene as Compatibilizer on the Properties of Linear Low-Density Polyethylene/Cyclic Natural Rubber Blends

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Abstract. In this paper, we report that the compatibility of polymer blends between linear low-density polyethylene (LLDPE) and cyclic natural rubber (CNR) can be increased by the addition of oleic acid-grafted linear low-density polyethylene. The research aims to investigate the effect of LLDPE-g-OA compatibilizer in LLDPE/CNR blends on improving the mechanical properties and characteristics of the blends. The LLDPE/CNR/LLDPE-g-OA blends (compatible blends) were prepared by blending methods in the molten phase using an internal mixer set at a temperature of 160 °C with a rotation speed of 100 rpm. The LLDPE pellets were inserted into it until melts and followed with an addition of CNR and LLDPE-g-OA. The tensile strength test results have shown an increase in mechanical properties after the addition of the compatibilizer with a maximum content of 4 wt%. The difference in FTIR spectra of compatible blends is indicated by the presence of C=C bonds from CNR, which appear at 1654.9 cm^{-1}, indicating the occurrence of physical bonds in the blends. The existence of the LLDPE-g-OA compatibilizer has increased the thermal stability of the polymer blends and changes in the melting point temperature of 124 °C. Compatible blends showed that the surface morphology was smoother, and there were significant microstructural changes compared to incompatible blends.

Keywords: Blends; Compatibilizer; Cyclic natural rubber; Linear low-density polyethylene; Oleic acid-grafted linear low-density polyethylene

1. Introduction

CNR is a natural rubber derivative obtained from the cis-1,4-polyisoprene intermolecular cyclization process involving acid catalysts such as sulfuric acid or Friedel-Craft. The advantages are resistance to chemicals, water, heat, and corrosion. CNR resins are widely applied commercially, including coatings, paint binders, printing inks, and good adhesives: on glass, metal, paper, and wood. CNR, also known as Resiprene-35 (CAS Registry no: 68441-13-4), is supplied as a granular solid. CNR is compatible with all mineral oils, maleic resins, alkyd resins, and bitumen (Siregar, Ardilla, and Nasution, 2021; Aritonang *et al.*, 2019). Meanwhile, industries, especially the plastics industry, have widely used and developed polyethylene. Linear low-density polyethylene (LLDPE) is one of

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several types of polyethylene that has been widely applied (Kusrini *et al.*, 2018). LLDPE has advantages: lightweight, heat resistant, water resistance, not easily broken, non-toxic, high flexibility, and resistant to chemicals, both acidic and alkaline compounds. Polyolefin materials also have disadvantages, including low adhesion, difficulty in the process of painting & coloring, and poor compatibility with other materials, such as the interaction between the surface of the LLDPE and CNR. The low adhesion interaction between LLDPE and CNR is caused by poor compatibility between the paint material and the polyolefin surface. Poor compatibility on the surface of LLDPE substrates is caused by LLDPE, which is classified as a semi-crystalline polymer with low surface energy, making it difficult for LLDPE to interact with other substrates such as CNR (Mahendra *et al.*, 2019; Kusrini *et al.*, 2018).

The development of polymer blends has become a particular interest among researchers, academics, and the industry. It can be the beginning of developing polymer blend products based on LLDPE/CNR and used as a reference in developing CNR as a paint binder on polyolefin surfaces, especially LLDPE. Polymer materials such as LLDPE and CNR can be blended to form miscible blends to produce homogeneous phase structures between LLDPE and CNR. It is necessary to have a compatibility process that can increase the interaction ability between the two polymer materials. The compatibility process is a technique to physically and chemically modify the interface and/or morphology (Aritonang *et al.*, 2020; Mahendra *et al.*, 2019).

The existence of a compatibilizer can increase the interaction between two polymers of different phases, where one part of the chemical composition of the compatibilizer has an affinity for one polymer. And the other part of the chemical component has an affinity for another polymer. This compatibilizer can also reduce the interface stress on the two polymer phases by increasing the interface adhesiveness of the two phases (Aritonang *et al.*, 2020). Mahendra *et al.* (2019) have used several types of compatibilizers from maleic anhydrides (MA), such as PP-g-MA and CNR-g-MA, to observe the compatibility between PP and PP CNR. LLDPE-g-MA copolymers have also been used as compatibilizers on LLDPE/PA-6 blends. The result has shown that the blends' compatibility increases after adding a compatibilizer to the polymer blends (Anjos *et al.*, 2019).

Oleic acid (OA) is a reactive monomer developed in recent years in polymer material modification. The oleic acid monomer in the grafting process can improve solvent-based paint's emulsion and adhesion properties because oleic acid is an unsaturated fatty acid with double bonds. The acid composition also has similarities with MA, which can react with hydroxyl groups (Irawan *et al.*, 2017; Liu *et al.*, 2003). OA monomer grafted onto LDPE has been documented by Liu *et al.* (2003). They obtained LDPE-g-OA copolymer through a molten phase in an internal mixer using dicumyl peroxide as the initiator. Considering the similarity of the main chain structure of LDPE with LLDPE, it is certain that LLDPE-g-OA can also be obtained from grafting OA monomer onto LLDPE through the molten phase in an internal mixer with the resulting side chain in the form of a hydroxyl group (OH).

The novelty of this research is the preparation of LLDPE-g-OA and its application as a compatibilizer in LLDPE/CNR blends. LLDPE-g-OA is used as a compatibilizer in the LLDPE/CNR blends based on the chemical structure of CNR, which has a double bond (C=C) 10-20%. The chemical structure is thought to be able to interact with the C=O carbonyl group of the OA molecule on LLDPE-g-OA material during the reactive process in the internal mixer. In addition, LLDPE is a matrix in LLDPE/CNR blends, so it is more suitable if LLDPE-g-OA is used as a compatibilizer in polymer blends (Mahendra *et al.*, 2019).

In this research, the compatibilizer used is LLDPE-g-OA obtained from the modification of LLDPE with OA through the grafting method using the BPO initiator. The LLDPE-g-OA copolymer can be used as a compatibilizer in the polymer blends increasing compatibility between the two polymer materials, both LLDPE and CNR. This study evaluated the use of LLDPE-g-OA as a compatibilizer onto LLDPE/CNR blends in the molten phase on its mechanical, thermal, and morphological properties.

2. Methods

2.1. Materials

Linear low-density polyethylene, LLDPE (Asrene UF1810) with a density of 0.919 g/cm^3 and melt flow index (MFI) of 1.0 g/10 min (190 ^{o}C/2.16 kg) was purchased from *PT. Chandra Asri Petrochemical Tbk,* Indonesia. Cyclic Natural Rubber, CNR (Resiprene-35) with a density of 0.91 g/mL and melt flow index (MFI) of 36.37 g/10 min (190 ^{o}C/2.16 kg) was supplied by *PT. Industri Karet Nusantara*, Indonesia. Subur Kimia Jaya Chemical Company, Indonesia, provided oleic acid with a density of 0.895 g/mL. Benzoyl peroxide (BPO), xylene, acetone, methanol, acetic acid, KOH, and phenolphthalein were all chemical reagents obtained from Merck.

2.2. Preparation of LLDPE-g-OA

Oleic acid (OA) grafting onto the LLDPE by free radical reaction at the molten phase was prepared in the internal mixer Thermo HAAKE Polydrive with a maximum loading capacity of 35 g. The processing conditions on the internal mixer were set at a temperature of 160 ^{0}C with a rotation speed of 100 rpm. LLDPE pellets were slowly inserted into the mixing chamber in the internal mixer for 5 min. After 5 min, the LLDPE has melted, and the torque value will be constant at this time. The OA of 15 wt% was added to the chamber containing LLDPE, followed by the addition of a BPO initiator of 5 wt%, and the free radical grafting process was left for 10 min, then the process was stopped. The results from the grafting process were removed from the chamber, cooled at room temperature, and cut into small pieces. The result obtained was an unpurified LLDPE-g-OA copolymer in the form of granules. The LLDPE-g-OA unpurified copolymer was weighed and put into a bottom flask containing xylene 200 mL of and a magnetic stirrer. The reflux device is connected to a condenser and heated on a hotplate at 140 ^{o}C while stirring at 60 rpm until LLDPE-g-OA dissolves completely. The LLDPE-g-OA solution was added with 80 mL of acetone to separate the OA homopolymer and the unreacted OA. The precipitate formed was filtered and washed with 150 mL of methanol (2 times). After that, LLDPE-g-OA was dried in an oven at 85 ^{o}C for 12 hours and stored in a desiccator for 24 hours to obtain LLDPE-g-AO purified (Figure 1a) (Aritonang *et al.*, 2019; Liu *et al.*, 2003).

The grafting degree (GD) percentage of LLDPE-g-OA was measured carried out as follows: The LLDPE-g-AO sample of 0.4 g was put into an Erlenmeyer flask containing xylene of 60 mL, heated until it dissolved completely, cooled, added 2 mL of acetic acid 0.1 M in xylene, heated for two h, cooled, added three drops of phenolphthalein indicator, then titrated with KOH 0.1 M in ethanol. The titration was stopped after showing a color change, the volume of KOH used was recorded, and the GD was calculated using the following equation (Liu *et al.*, 2003) :

$$\boldsymbol{GD\ (wt\ \%) = \frac{V.N.M}{W}\ x\ 100\ \%} \tag{1}$$

Where V is the volume of KOH/ethanol solution, N is the concentration of KOH/ethanol solution, M is the molecular weight of OA, and W is the weight of LLDPE-g-OA.

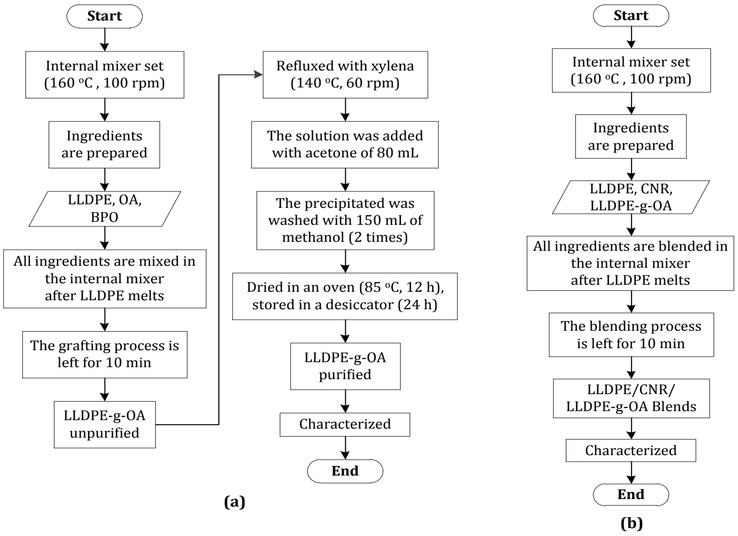

Figure 1 Preparation of (a) LLDPE-g-OA compatibilizer; (b) LC blends and LCC blends

2.3. Preparation of LLDPE/CNR (LC) Blends and LLDPE/CNR/LLDPE-g-OA (LCC) Blends

LLDPE pellets were slowly inserted into the mixing chamber in the internal mixer Thermo HAAKE Polydrive for 5 min. The processing conditions were set at a temperature of 160 °C with a rotation speed of 100 rpm. After 5 min, the LLDPE has melted, and the torque value will be constant at this time. CNR was added to the chamber containing LLDPE, followed by adding the LLDPE-g-OA copolymer as a compatibilizer to the LLDPE/CNR blends, with composition as shown in Table 1. The blends were left for 10 min. The blends were removed from the internal mixer and left at room temperature (Mahendra *et al.*, 2019). The result obtained was (1) LLDPE/CNR blends or incompatible blends after this referred to as the LC blends, (2) LLDPE/CNR/ LLDPE-g-AO blends after this referred to as the LCC blends (Figure 1b).

Table 1 Composition of LC blends and LCC blends

Sample Code	LLDPE (wt%)	CNR (wt%)	LLDPE-g-OA (wt%)
LLDPE	100.0	-	-
LC1	90.0	10.0	-
LC2	80.0	20.0	-
LC3	70.0	30.0	-
LC4	60.0	40.0	-
LC5	50.0	50.0	-
LCC1	69.5	29.5	1.0
LCC2	69.0	29.0	2.0
LCC3	68.5	28.5	3.0
LCC4	68.0	28.0	4.0
LCC5	67.5	27.5	5.0

2.4. Characterization

2.4.1. Mechanical Properties

The test specimens for mechanical testing were prepared according to ASTM D638-14 type I with compression molding using a hot press machine at the temperature of 160 °C for 15 min. The test specimens were placed in the grip of the testing machine. The ultimate tensile strength, elongation at break, and Young's modulus were measured using the Ultimate Testing Machine GoTech AI-7000 with a maximum load cell capacity of 20 KN at 10 mm/min speed.

2.4.2. Fourier Transform Infra-red analysis

The polymer blends were analyzed using the Fourier transform infrared (FTIR) spectrophotometer using Agilent/FTIR Cary 630. The measurements process was carried out at wave numbers 4000 to 650 cm^{-1}.

2.4.3. Thermal Analysis

The thermal stability of the polymer blends was evaluated by Thermogravimetric Analysis (TGA) / Differential Thermal Analysis (DTA) using Hitachi/TG/DTA 7300 series with heating temperatures from 30 to 600 °C and heating rate of 10 °C / min. The thermal properties of polymer blends were examined by Differential Scanning Calorimetry (DSC) using Hitachi/X-DSC 7000 with heating temperatures from 28 to 200 °C and a heating rate of 10 °C/min under nitrogen flow.

2.4.4. Morphological observation

The polymer blends were observed on its surface structure using Scanning Electron Microscope (SEM) ZEISS EVO @ MA 10. The sample is coated with gold to maintain good conductivity during analysis.

3. Results and Discussion

3.1. Mechanical properties analysis

Mechanical testing on polymer materials is one of the parameters used as references to determine the quality of polymer blends between LLDPE and CNR. The results of this test obtained values of tensile strength, elongation at break, and Young's modulus, as shown in Figure 2.

The LC blends with various ratios showed decreased tensile strength, elongation at break, and Young's modulus compared to the neat LLDPE. This decrease occurs due to brittle CNR in the polymer blends. Suppose the CNR ratio in the LC blends is increased. In that case, it can cause a decrease in mechanical properties, which indicates that the stiffness of the polymer blends decreases while the brittleness increases. Thus, the mechanical properties test results depend on the LLDPE ratio in the LC blends. A similar matter has been observed by Anjos *et al.* (2019), that the mechanical properties show a decrease with increasing the composition of one of the components in the incompatible blends. This indicates that the LC blends are incompatible between LLDPE and CNR. Incompatible blends were caused by molecular weight, density, polarity, and surface tension energy differences between the two polymer materials, in this case, LLDPE and CNR (Passador *et al.*, 2016). The LC blends for samples LC4 and LC5 showed a significant decrease against neat LLDPE compared to samples LC1, LC2, and LC3. The percentage decrease in the tensile strength of the LC4 and LC5 samples against neat LLDPE was 30.52% and 31.72%, respectively.

Meanwhile, the samples of LC1, LC2, and LC3 were only 7.95%, 11.06%, and 12.62%, respectively. This phenomenon shows that the maximum neat CNR content blended with neat LLDPE is LLDPE/CNR blends (70:30). The LCC blends for samples LCC2, LCC3, LCC4, and LCC5, respectively, showed an increase in tensile strength of 1.35%, 3.08%, 14.36%, and 9.14%. The optimum condition of LCC was in the LCC4 sample with the LLDPE-g-OA concentration of 4%. There was a decrease in LCC5 caused by the lubricating effect of the compatibilizer, in this case, oleic acid. A compatibilizer in the blends increases the tensile strength and the interface adhesion of the two materials. The increasing homogeneity affects the ductility of the polymer blends, which causes a decrease in their mechanical properties (Dobrovszky and Ronkay, 2016).

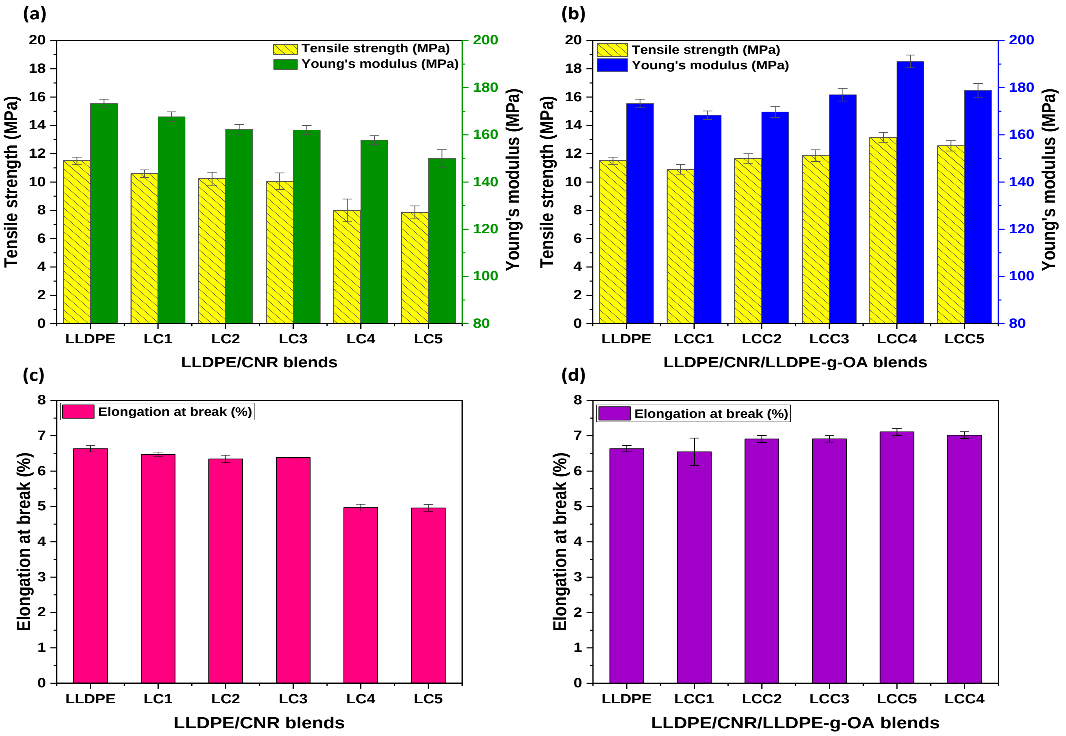

Figure 2 Tensile strength and modulus young's of (a) LC blends, (b) LCC blends, elongation at break of (c) LC blends, (d) LCC blends

3.2. FTIR Analysis

The FTIR spectrum of LLDPE, CNR, LLDPE-g-OA, LC blend with the variation of 70:30 (LC3), and LCC blends with the variation of 68:28:4 (LCC4) can be observed in Figure . The LC3 and LLC4 blends are considered the optimum variation in the polymer blends based on the results of the mechanical properties test.

In the CNR spectrum, there is a band at 2922.2-2847.7 cm^{-1} that indicates a stretching vibration of hydrocarbons alicyclic, followed by the appearance of the band at 1453.7 cm^{-1}, 1371.7 cm^{-1}, 745.5 cm^{-1}, indicating the presence of the vibration of CH_2 scissoring, CH_3 scissoring, and C-H rocking. The small band of 1654.9-1610.2 cm^{-1} indicates the presence of C=C in the CNR cyclic chain. A small band (a new peak) appeared in the LCC blends at 1654.9 cm^{-1}, indicating C=C from CNR. A small band with a wide absorption at 3100-3400 cm^{-1} was also visible, which is similar to the neat CNR spectrum characteristics. It showed that the LCC blends were compatible because the characteristics of the LLDPE and CNR are visible in the LCC blends spectrum, while the LC blends spectrum does not show any specific bands, which are the CNR characteristics.

The results of FTIR spectra indirectly confirmed that LLDPE-g-OA as compatibilizer was able to increase LLDPE and CNR into compatible blends, as evidenced by the appearance of the characteristics of the two materials in the LCC blends.

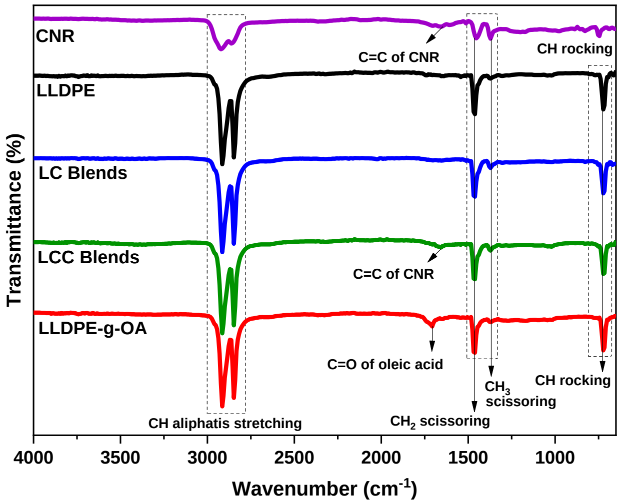

Figure 3 FTIR spectra of LLDPE, CNR, LLDPE-g-OA, LC blends, and LCC blends

The result of the FTIR spectra of the LCC blends is that there has been an interaction between LLDPE and CNR with the presence of an LLDPE-g-OA compatibilizer. The low concentration of the compatibilizers in polymer blends makes it challenging to determine that a chemical reaction occurs between the compatibilizer and the matrix in the polymer blends. So it is possible to determine the compatibility of polymer blends based on mechanical properties tests only (Anjos *et al.*, 2019).

A small band (a new peak) at 1707.1 cm^{-1} appears in the LLDPE-g-OA spectrum, indicating the presence of a carbonyl group (C=O) from OA grafting onto the LLDPE. The results of measuring the grafting degree (GD) percentage, LLDPE-g-OA purified obtained a value of 13.42%, and LLDPE-g-OA unpurified obtained a value of 16.95. Based on the measurement of the GD percentage and the small band that appeared at 1707.1 cm^{-1}, it is known that OA has been successfully grafted onto the LLDPE main chain using the BPO initiator (Figure) (Liu *et al.*, 2003).

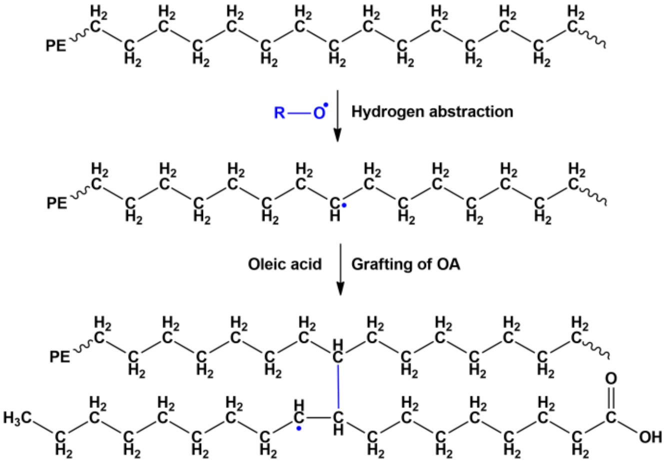

Figure 4 Reaction mechanism for the formation of LLDPE-g-OA

3.3. Thermal Analysis

Thermal analysis was carried out to investigate the degradation characteristics and thermal stability of LLDPE, CNR, LLDPE-g-OA, LC blends, and LCC blends, as shown in Figure 5. This thermal analysis includes thermogravimetry analysis (TGA), derivate thermogravimetry (DTG), and differential thermal analysis (DTA). And differential scanning calorimetry (DSC) (Abdullah *et al.*, 2014).

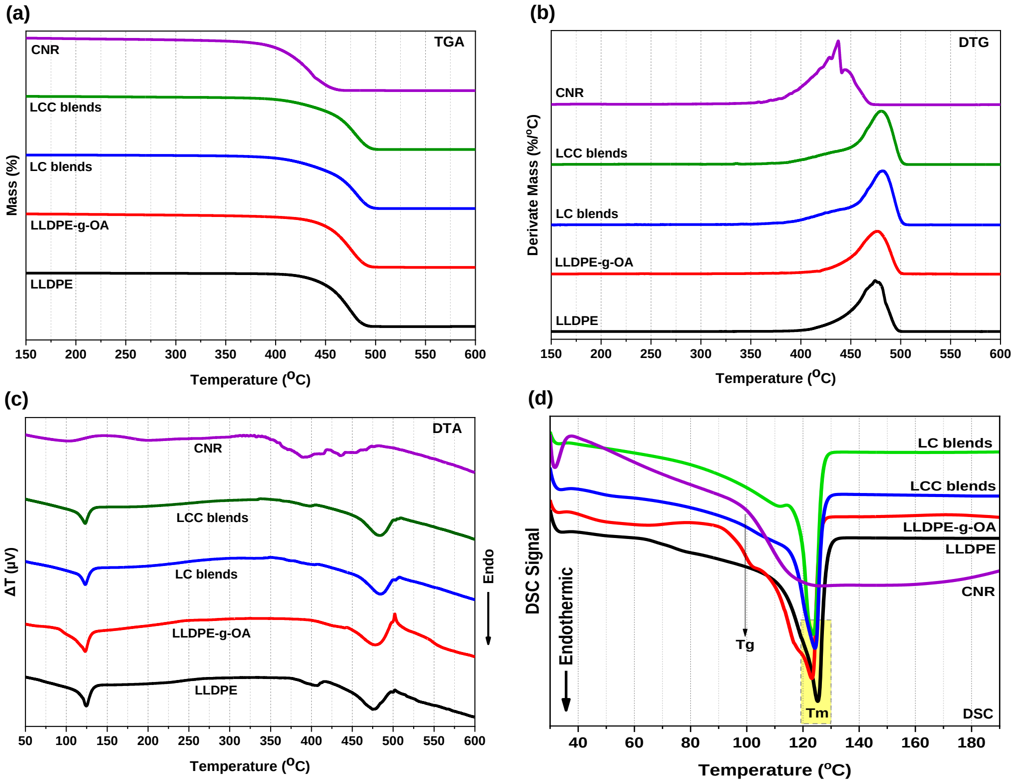

Figure 5 (a) TGA, (b) DTG, (c) DTA, (d) DSC curves of neat LLDPE, LLDPE-g-OA, LC blends, LCC blends, and neat CNR

Based on the results of the TGA analysis (Table 2), at the beginning of the mass degradation at $T_{1\%}$ and $T_{5\%}$, there was no significant difference between neat LLDPE and LC blends. The mass degradation characteristics of the neat CNR influence the temperature difference. The significant temperature difference occurred in the LCC blends, which confirmed the existence of LLDPE-g-OA because it had the same characteristics at $T_{1\%}$. At $T_{5\%}$, mass degradation of the LLC blends was between LLDPE-g-OA and LC blends. At $T_{95\%}$ of the LCC blend was above the other samples. It indicates that the blending process between LLDPE and CNR has increased the thermal stability of the blends. The decomposition temperature (T_d) of all samples from the DTA analysis did not show any significant difference from the T_{max} of mass degradation. Mahendra *et al.* (2019) reported that the presence of graft copolymers as compatibilizers in the blends not only functions to reduce interfacial tension. It can increase the compatibility of the blends between polyolefin and CNR, where there is a physical interaction on the surface between two polymer phases which has an impact on increasing thermal stability.

The increase in thermal stability after the addition of compatibilizer is due to the formation of chemical bonds between the polymer chain. And the compatibilizer, where the end groups of the carboxylic acid (in this case, oleic acid) in the graft polymer directly affect its thermal stability (Lima *et al.*, 2019).

Based on the results of the DSC analysis, which showed the endothermic peak of neat LLDPE, LLDPE-g-OA, LC blends, and LCC blends. The endothermic peaks indicate each sample's melting point temperature (Tm). The polymer blends have a lower T_m than the neat LLDPE. The presence of LLDPE-g-OA compatibilizer in the blends directly affects the melting point of the LCC blends, although the results are still below the T_m of the neat LLDPE sample. The neat CNR did not show the presence of an exothermic peak, but there was a glass transition (T_g) at the temperature of 101.1 ^{o}C. It means that the visible peak is moisture. The T_g temperature of neat CNR has similarities with previously reported research (Aritonang *et al.*, 2020).

Table 2 Thermal analysis data of LLDPE, CNR, LLDPE-g-OA, LC blends, and LCC blends

Sample	$T_{1\%}$ (°C)	$T_{5\%}$ (°C)	T_{max} (°C)	(%)	$T_{95\%}$ (°C)	T_m (°C)	T_d (°C)
LLDPE	277.4	419.0	474.3	68.4	487.0	125.0	474.6
LLDPE-g-OA	220.0	385.4	477.0	70.1	490.6	123.1	478.1
LC blends	276.3	393.0	482.0	75.5	492.7	123.6	484.1
LCC blends	220.0	391.3	479.9	71.1	493.1	124.0	483.7
CNR	145.9	317.2	437.5	70.5	458.1	-	435.0

3.4. Morphological Observation

The surface morphology of the LCC blends (Figure e) has shown a smooth surface structure with small phases compared to the LC blends (Figure d). There is an inter-surface reaction between the two polymer phases (LLDPE and CNR). A compatibilizer in these blends has reduced the interfacial tension between the two polymer phases (Jia *et al.*, 2015). It can be observed from the surface structure that it has miscible blends because CNR in the blend has been evenly dispersed in LLDPE as the matrix.

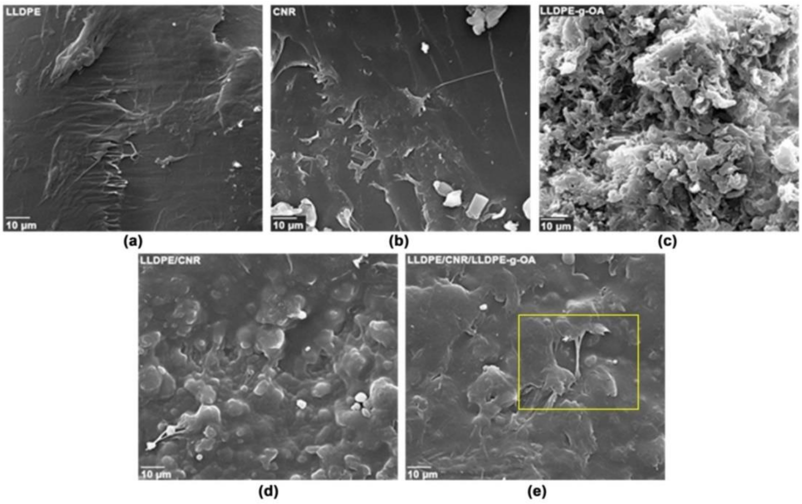

Figure 6 SEM micrographs of (a) LLDPE, (b) CNR, (c) LLDPE-g-OA, (d) LC blends, and (e) LCC blends

As Das *et al.* (2012) reported, the compatibilizer plays a role in reducing the size of the dispersed substance phase to be evenly dispersed on the matrix in the blends. The surface morphology of the LC blends has shown the presence of a rough surface structure with large phases. The LC blends have shown that the two phases cannot blend well (immiscible). It is due to the large interfacial tension between the two phases, which results in poor surface adhesion. The low surface polarity of LLDPE is associated with low surface energy, which causes poor surface adhesion. CNR is incompatible with LLDPE due to differences in surface energy. The difference in surface energy produces a large interfacial tension and poor surface adhesion (Mahendra *et al.*, 2019). The absence of compatibilizer in the LC blends has shown a significant difference in the microstructure of the LCC blends. The surface morphological characteristics of LLDPE, CNR, and LLDPE-g-OA have shown significant differences in the microstructure of the LC blends and the LCC blends. Many studies have reported that the addition of compatibilizer in polymer blends can increase the compatibility of the immiscible blends (Oskooie *et al.*, 2017).

4. Conclusions

LLDPE-g-OA as a compatibilizer in the LLDPE/CNR blends has influenced the polymer blends' mechanical, thermal, and morphological properties. Tensile strength testing on the LLDPE/CNR blends after adding the LLDPE-g-OA compatibilizer has increased tensile strength value, elongation at break, and Young's modulus. The optimum results for the increase in mechanical properties were the compatibilizer content of 4% with a tensile strength value of 12.93 MPa, elongation at break of 7.11%, and Young's modulus of 191.13 MPa. The thermal stability of the LLDPE/CNR blends in the presence of LLDPE-g-OA has shown an increase characterized by mass degradation of 95% at 493.1 ^{o}C and caused a change in thermal properties with the melting point occurring at a temperature of 124 ^{o}C. The FTIR spectra results on the LLDPE/CNR/LLDPE-g-OA blends showed a difference (a new peak) compared to the constituent material and incompatible blends, which are characterized by the presence of C=C bonds from CNR. The morphological results showed significant microstructural changes and a smoother surface compared to the incompatible blends. LLDPE-g-OA compatibility, which is quite good in polymer blends of different polarities, can certainly play a role in polymer composites in the presence of inorganic fillers.

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