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MECHANICAL PROPERTIES OF CLAY/LLDPE AND ORGANOCLAY/LLDPE NANOCOMPOSITES

KİL/LLDPE VE ORGANİK KİL/LLDPE NANOKOMPOZİTELERİNİN MEKANİK ÖZELLİKLERİ

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Abstract

Linear low-density polyethylene (LLDPE) is widely used in many areas in daily life because it is both light and flexible. In this study, clay/LLDPE and organoclay/LLDPE nanocomposites were obtained by melt intercalation method, incorporating small amounts (1- 5 wt.%) of nanosized clay and organoclay modified with a positively charged salt to enhance the mechanical properties of the polymer. Characterization and mechanical tests showed that the mechanical strength of the composites increased with additive content, though some flexibility was partially lost. FTIR and XRD analyses confirmed that clay and organoclay interacted with polymer chains and dispersed homogeneously. ANOVA results demonstrated that both filler type and concentration significantly influence the mechanical properties, with organophilic organoclay showing superior interaction to the LLDPE matrix compared to hydrophilic clay. Specifically, the addition of 5 wt.% organoclay resulted in increases of 56,67%, 58,73%, and 39,53% in elastic modulus, yield strength, and tensile strength, respectively. Additionally, the observed 5% rise in melting temperature suggests potential for expanding the application range of these nanocomposites to areas requiring thermal stability.

Keywords: Clay, LLDPE, mechanical properties, nanocomposite, organoclay.

Öz

Lineer düşük yoğunluklu polietilen (LLDPE), hem hafif hem de esnek olması nedeniyle günlük yaşamda birçok alanda yaygın olarak kullanılmaktadır. Bu çalışmada, polimerin mekanik özelliklerini iyileştirmek amacıyla pozitif yüklü tuz ile modifiye edilmiş organokil ve nano boyutlu kilin düşük miktarlarının (ağ. %1-5) kullanıldığı eriyik interkalasyon yöntemi ile kil/LLDPE ve organokil/LLDPE nanokompozitleri elde edilmiştir. Karakterizasyon ve mekanik testler, katkı miktarının artışıyla kompozitlerin mekanik dayanımının arttığını ancak esnekliklerinin kısmen azaldığını göstermiştir. FTIR ve XRD analizleri, kil ve organokilin polimer zincirleriyle etkileşime girerek homojen bir şekilde dağıldığını doğrulamıştır. ANOVA sonuçları, dolgu tipi ve konsantrasyonunun mekanik özellikler üzerinde önemli bir etkiye sahip olduğunu ve organofilik organokilin, hidrofilik kile kıyasla LLDPE matrisi ile daha üstün bir etkileşme gösterdiğini ortaya koymuştur. Özellikle, %5 ağırlık oranında organokil ilavesi ile elastik modülde %56,67, akma dayanımında %58,73 ve çekme dayanımında %39,53 oranında artış gözlenmiştir. Ayrıca, erime sıcaklığındaki %5'lik artış, bu nanokompozitlerin sıcaklık değişimlerine karşı dayanıklılık gerektiren uygulama alanlarında kullanım potansiyelini artırabileceğini göstermektedir.

Anahtar Kelimeler: Kil, LLDPE, Mekanik özellikler, Nanokompozit, Organokil.

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1. INTRODUCTION

Polyethylene (PE), a widely used petroleum-based polymer as a under the group of polyolefins, encompasses various types with distinct chemical structures, depending on production conditions like pressure and temperature. These types are categorized by density and branching characteristics and labelled such as high density polyethylene (HDPE), low density polyethylene (LDPE), ultra high molecular weight polyethylene (UHMWPE), and cross-linked polyethylene (PEX). Among these, linear low density polyethylene (LLDPE) is defined by its unique structure: it consists of a linear carbon backbone with short, evenly spaced alkyl branches, which disrupt crystalline formation (Peacock, 2000). As a result, LLDPE exhibits enhanced stiffness and strength than LDPE, and better ductility than HDPE while maintaining a density range of 0,915–0,940 g/cm^3.

Structurally, LLDPE combines the flexibility of LDPE with the strength of HDPE, leading to superior chemical and mechanical resilience. These qualities make it ideal for applications in flexible plastic sheeting, packaging, and transportation. Therefore, LLDPE is widely used in various industries for products such as packaging films, agricultural mulch, lay-flat pipes, water tubing, and low-temperature food containers, making it one of the most produced common plastics worldwide due to its flexibility, strength, and durability. Enhancing LLDPE’s properties can expand its usage, achieved by incorporating fillers or active functional particles to tailor characteristics as needed.

Research on improving LLDPE’s application potential has surged since the 1980s, especially as nanoparticles became popular additives in the 1990s (LeBaron et al., 1999). Recent studies underscore LLDPE's role in agriculture, where its use as a mulching film enhances soil protection, water retention, and weed control, while also extending durability under weathering (Liu et al., 2002; Mansoor et al., 2021; Wadgaonkar & Mehta, 2019). To enhance LLDPE economically, various additives—such as clay (As’Habi, 2013; Durmus et al., 2007; Mir et al., 2017; Mousavi et al., 2016; Sabetzadeh et al., 2016; Said et al., 2020; Stoeffler et al., 2011), silica (Kontou & Niaounakis, 2006), carbon (Liu et al., 2002), carbon nanotubes (Mezghani et al., 2011), and graphene oxide (Li et al., 2019)—have been explored for their effects on LLDPE’s gas barrier, thermal stability, UV resistance, and fatigue properties. Hotta and Paul (2004) demonstrated that organoclays, within a 0,8–6,9 wt.% range, improve the mechanical performance of LLDPE.

In this study, the montmorillonite clay from Turkiye was used to investigate the effect of nanofiller on the mechanical properties of LLDPE. Given its hydrophilic nature, the clay was converted to an organoclay via interaction with a cationic surfactant (Gunister et al., 2004) to enhance compatibility with the LLDPE matrix. Besides mechanical improvements, this study also targets enhanced thermal stability. While prior studies incorporated up to 10 wt.% of nanosized clay, this research aims to achieve comparable or superior mechanical performance at lower concentrations.

2. NANOCOMPOSITES PREPARATION AND TESTING

2.1. Materials

The clay sample was sourced from the bentonite deposits in Edirne-Lalapasa, Turkiye. Analysis identified the dominant clay mineral as 98 wt.% montmorillonite, with minor components of 0,5–1 wt.% illite, 1–2 wt.% calcite, 0–1 wt.% feldspar, and 0–0,2 wt.% quartz. The chemical composition, determined using atomic absorption spectroscopy (Perkin Elmer 3030) except for silica (measured gravimetrically), was as follows: 56,77% SiO_2, 19,27% Al_2O_3, 3,50% CaO, 4,54% Na_2O, 4,48% MgO, 2,33% Fe_2O_3, 0,41% K_2O and 0,22% TiO_2. The clay particles consist of two tetrahedral silica sheets sandwiching a central octahedral alumina sheet, with exchangeable Ca^{2+} cations between the layers. To modify the clay's surface properties, Na^+ activated clay was treated with the cationic surfactant hexadecyltrimethylammonium bromide (HDTABr, $C_{19}H_{42}NBr$, molecular weight 364,46 gr/mol, supplied by Merck) following the procedure outline by Canbaz & Gungor (2009). This treatment expanded the clay layers by exchanging Na^+ ions with HDTABr, converting the clay from hydrophilic to organophilic; the resulting product is termed organoclay.

The LLDPE was supplied by Borouge (Abu Dhabi, UAE) with its density 0,931 g/cm^3. The melt flow index measured per ASTM D 1238-10 procedure A at 190°C with a 2,16 kg load was 0,2 g/10 min.

2.2. LLDPE Nanocomposites Preparation

The samples were prepared by melt intercalation method, adding 30 mg, 90 mg, and 150 mg of clay (Clay) per 3 g of LLDPE in mini-compounding machine to achieve 1 wt.%, 3 wt.%, and 5 wt.% Clay/LLDPE nanocomposites. The extrusion parameters were optimized based on the pretesting results of pure LLDPE samples with the LLDPE pellets melted and mixed in a Haake Minilab Rheomex CTW S mini-compounder at temperatures 180-200 °C, rotation speed of 50-100 rpm and mixing times of 5 – 10 minutes. The same procedure was followed using organoclay (OClay) to produce 1 wt.%, 3 wt.% and 5 wt.% OClay/ LLDPE nanocomposites.

2.3. Methods for Characterization and Testing of Nanoparticles, LLDPE and LLDPE Nanocomposites

The expansion of clay layers in the presence of LLDPE was analyzed using X-ray diffraction (XRD). PANalytical's X'Pert PRO Materials Research Diffractometer with copper X-ray source was used to determine the expansion of clay layers caused by cationic salt and LLDPE chains. Shifts of the characteristic peaks of LLDPE, Clay and OClay were identified using Bragg's Law from the intensity vs. diffraction angle (2θ) plot. This peak shift provided evidence of expanded interlayer spacing, indicating the interaction between LLDPE and the nanoparticles covering its surface. Moreover, the intensity drop of the peaks also implies the disturbing of crystalline structures.

The bonding interactions between additives (Clay and OClay) and LLDPE were examined by using Thermo Scientific Nicolet iS10 FT-IR spectrometer. The samples for FTIR analysis were prepared as pallets with KBr. The transition temperatures of

LLDPE and its composites were determined using differential scanning calorimetry (DSC) performed on a DSC 204 F1 Netzsch at a heating rate of 10 °C /min.

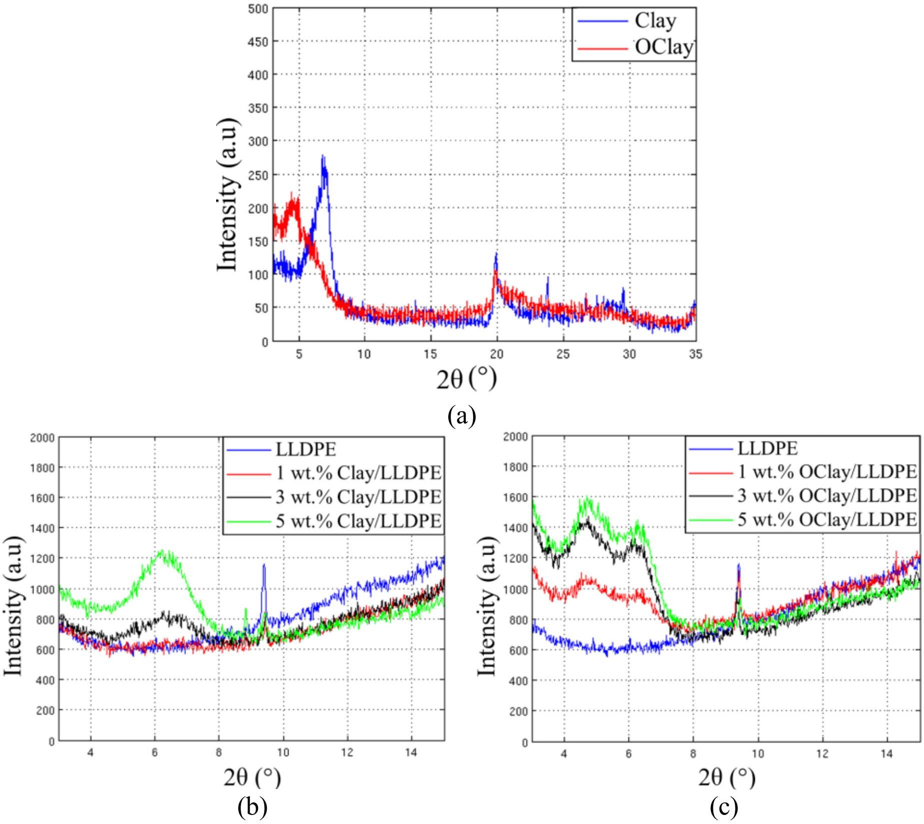
The dumbbell shape specimens for the mechanical testing were prepared according to ISO527 standard using a Haake Minijet II Thermo Scientific mini-molding machine at 600 bar and 50 °C. Prior to testing, the uniform distribution of the clay particles in the LLDPE matrix, as well as agglomerations and air bubbles was examined under an optical microscope. The tensile properties of LLDPE and its nanocomposites were determined by using the Testometric M350-10CT tensile tester at a strain rate of 5 mm/min, following ISO527. Additionally, the hardness tests were conducted on 2 mm thick specimens of LLDPE and its nanocomposites using a Shore Durometer Model 902-51 Operating Stand, following ASTM D2240. Each test was repeated at least 10 different points across the sample surface to ensure consistent measurements.

The two-way analysis of variance (ANOVA) was applied to evaluate the effect of filler (Clay and OClay) presence and filler material content on the mechanical properties of LLDPE. The two factors are: filler type (Clay and OClay), and amount of filler (1, 3, 5 wt.%).
The mechanical properties assessed included elastic modulus, yield strength, and tensile strength. The ANOVA analysis was based on repeated measurements obtained for each filler concentration. A 95% confidence level was used in all analyses, and factors were considered statistically significant when the p-value was less than 0,05 (Yassin & Gunister, 2023).

3. RESULTS AND DISCUSSION

3.1. The Structural and Thermal Characterization

The d-spacing for each scattering angle in all the samples was calculated using Bragg's equation, following the same steps given in the previous work (Li at al. 2019). XRD patterns of clay, organoclay, polymer and polymer nanocomposites are given in Figure 1 a, b and c respectively.



(a)

(b)

(c)

Figure 1. XRD Analyzing Results of a) Clay and OClay, b) LLDPE and Clay/LLDPE Nanocomposites, and c) LLDPE and OClay/LLDPE Nanocomposites

Due to surfactant interaction through cation exchange, the interlayer spacing of clay expanded from 1,26 nm to 3,65 nm (Figure 1a). In the Clay/LLDPE nanocomposites, polymer chains were intercalated between the clay layers, expanding the interlayer spacing from 1,26 nm to 1,47 nm (Figure 1b). For the OClay/LLDPE nanocomposites, the polymer chains similarly intercalated with the organoclay layers; however, interactions were more prevalent on the larger organophilic surfaces of OClay, resulting in LLDPE chains encapsulating the organoclay particles (Figure 1c). If exfoliation of additives within the LLDPE chains occurred, the crystalline peaks of additives would disappear in the characteristic crystalline peak regions of Clay and OClay. However, in both Clay and OClay samples, the XRD results indicate predominantly intercalated structures, as the additives' crystalline peaks remain visible in Figure 1. For both Clay and OClay, the intensity of the LLDPE crystalline peak slightly decreases inversely with the additive weight %, as shown in Figures 1b and Figure 1c.

Based on the DSC results given in Figure 2, the melting temperature of LLDPE was determined to be 129 °C, with degradation beginning above 200 °C. Accordingly, the optimal processing conditions were set at 180 °C, mixing time of 7 minutes, and screw rotation speed of 50 rpm to prevent degradation and avoid air bubbles in the composites. All LLDPE nanocomposites were prepared under these optimized conditions. In Figure 2, the heat flux (W/g, with endothermic reactions shown as upward peaks) is plotted vs. temperature up 200 °C, as measured by DSC.

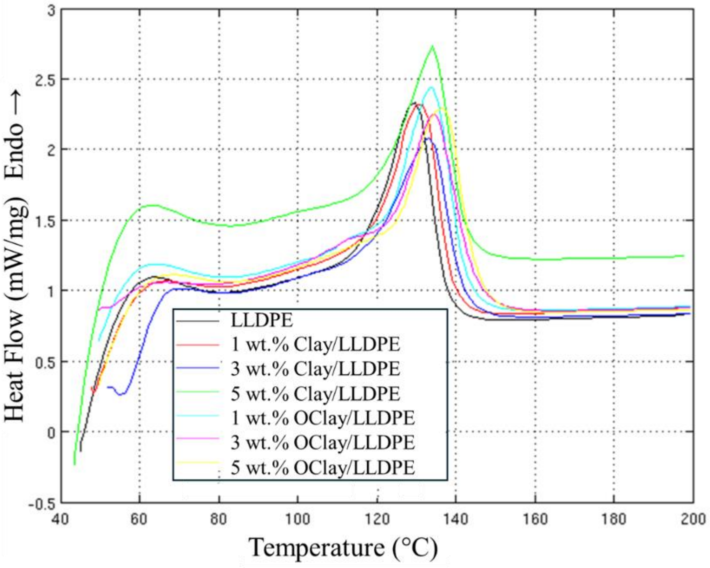


Figure 2. Heat Flux vs. Temperature Graphs of Pure LLDPE, Clay/LLDPE and OClay/LLDPE Nanocomposites

The derivatives of the DSC curves were analyzed to determine the transition temperatures. The melting temperature of pure LLDPE was found to be 129,8 °C. With the addition of clay at concentrations of 1, 3, and 5 wt.%, the melting temperature increased to 130,9, 132,5, and 134,1 °C, respectively. When OClay was added to LLDPE in the same concentrations, the melting temperature increased slightly more, shifting to 133,8, 134,3, and 136,3 °C, respectively. As a result, the melting temperature of LLDPE is improved 5 % with addition 5 wt.% of OClay.

The FTIR spectrum of LLDPE, and its composites are given in Figure 3.

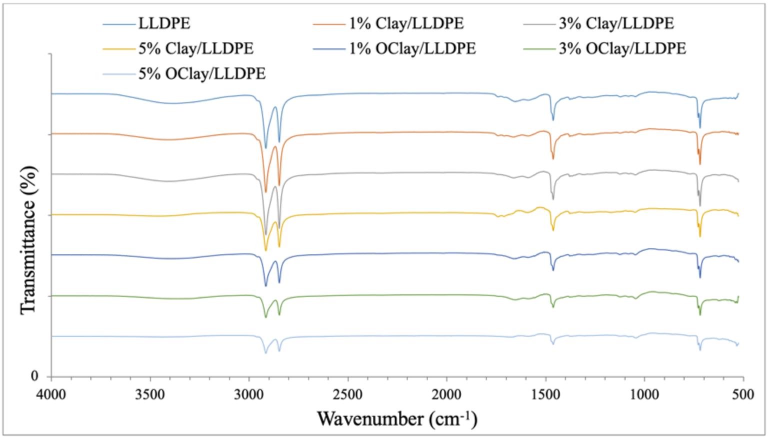


Figure 3. FTIR Results of Pure LLDPE, Clay/LLDPE and OClay/LLDPE Nanocomposites

Analyzing FTIR spectrums, the characteristic peaks band of LLDPE and its composites are given in Table 2.

Table 2. FTIR Results of Pure LLDPE, Clay/LLDPE and OClay/LLDPE Nanocomposites

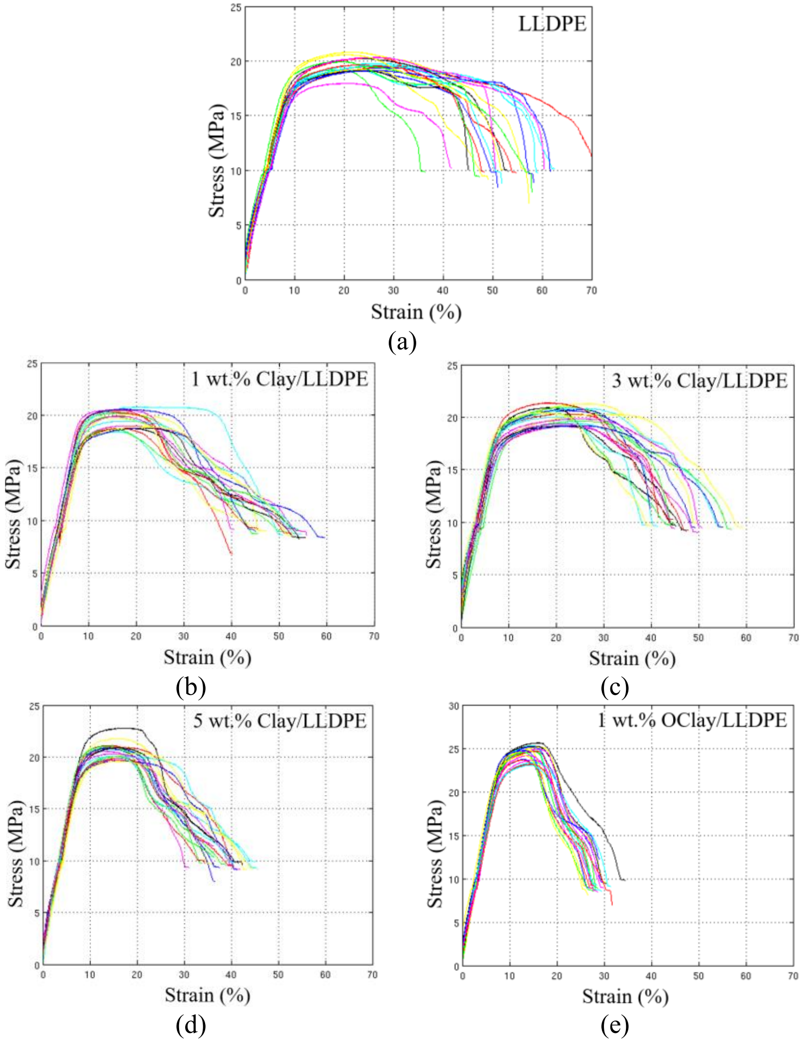
Amount of additive	**H-OH**	**C-H**	**C-H**	**H-O-H**	**C=C**	**C-C**	**C-C**	**Si-O**	**C-H**	**Al-OH**	**Si-O**
0	3384-3386	2916	2848	1654	1587 -1591	1462	1378	-	719	-	-
1 wt.% Clay	3404-3409	2916	2848	1660-1666	1588 -1592	1462	1378	1048 -1048	719	631-632	525
3 wt.% Clay	3407-3411	2916	2848	1660	1588 -1591	1462	1378	1046 -1048	719	624-625	525
5 wt.% Clay	3457- 3459	2916	2848	1711	1591 -1593	1462	1378	1048 -1048	719	628	525
1 wt.% OClay	3388-3405	2916	2848	1651	1588 -1592	1462	1378	1046 -1047	719	623-625	525
3 wt.% OClay	3352-3366	2916	2848	1651	1587 -1588	1462	1378	1045 -1046	719	622-622	525
5 wt.% OClay	3427-3445	2916	2848	1651	1586 -1592	1462	1378	1044 -1045	719	625	525

According to Table 2, the FTIR spectrum of pure LLDPE shows the characteristic CH_2 asymmetric and symmetric stretching bands, with peaks at 2916 cm^{-1} and 2848 cm^{-1}, respectively. The peak at 1462 cm^{-1} was attributed to the bending deformation of the C-H bond. The CH_3 symmetric deformation band appeared at 1378 cm^{-1} and the C-H rocking deformation occurs at 719 cm^{-1}. (Gulmine et al. 2002)

For the clay and organoclay, the shifts around 621 - 630 cm^{-1} indicated the presence of central cations (such as Mg) in the octahedral sheet. Peaks in the range of 1045–1050 cm^{-1} were associated with the stretching of Si–O bonds. A strong peak at 525 cm^{-1} is attributed to the Si–O–Al bending (Madejova 2003; Durmus & Macosko, 2007). Characteristic absorption peaks of the clay structure are shifted in the FTIR spectrum of the LLDPE nanocomposites, suggesting an interaction between the LLDPE chains and clay layers. In Table 2, a broad band 3351 - 3459 cm^{-1}, observed clearly in the spectra of montmorillonite is due to hydrogen bond between LLDPE branches and H–O–H vibrations of adsorbed water.

3.2. The Mechanical Testing

Testometric M350-10CT was used for the tensile tests of pure LLDPE and its composites following ASTM638 standard and each test was repeated twenty times. The mechanical properties such as elastic modulus, yield strength, tensile strength, tensile strain, elastic storage energy (resilience) and durability of materials (toughness) are determined from the stress-strain diagrams of LLDPE (Figure 4a), Clay/LLDPE nanocomposites (Figure 4b-d), and OClay/LLDPE nanocomposites (Figure 4e-g).



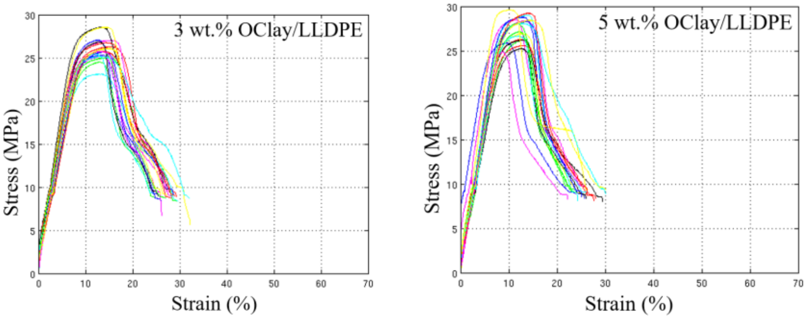
(a)

(b)

(c)

(d)

(e)



(f) (g)

Figure 4. The tensile stress – strain diagram of samples for a) LLDPE, b) 1 wt.% Clay/LLDPE, c) 3 wt.% Clay/LLDPE, d) 5 wt.% Clay/LLDPE, e) 1 wt.% OClay/LLDPE, f) 3 wt.% OClay/LLDPE, g) 5 wt.% OClay/LLDPE nanocomposites.

The tensile (elastic) modulus of the samples was determined by the slope of linear portion in elastic region of the curves. The yield point is the where noticeable plastic deformation starts on the stress-strain graph leaving from the linearity. The yield strength is the stress value at the yield point. Tensile strength is the maximum stress of the samples under the applied load, and corresponding strain is called tensile strain. The stored elastic energy is called resilience which is the area under the stress-strain up to yield point, and stored energy up to rupture is called toughness which is the area under the stress-strain curve up to failure. Stiffness, the resistance to elastic deformation, is the key parameter for many applications of LLDPE measured by elastic modulus, higher resilience means better stiffness.

The resistance to localized plastic deformation of LLDPE and its nanocomposites was determined through hardness testing, with the results presented in Figure 5. The type and amount of filler had a slight effect on the hardness values of LLDPE. The observed variability in hardness can be attributed to factors such as minor inconsistencies in filler dispersion during the mixing process or localized microstructural differences within the composite matrix, including variations between amorphous and crystalline regions. To ensure reliable measurements, all samples were carefully examined under an optical microscope prior to testing, and any specimens containing air bubbles were excluded. As a result, air bubbles are not considered a contributing factor to the observed spread in values. The variability is more pronounced at lower filler concentrations, likely due to greater challenges in achieving complete homogeneity in the composite matrix, which may also influence mechanical properties.

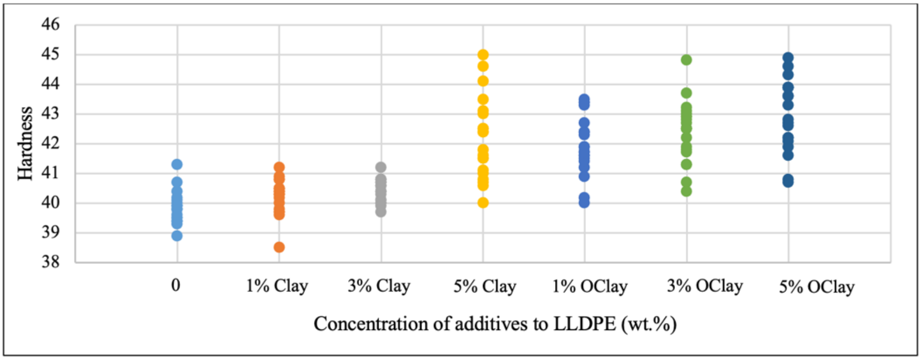


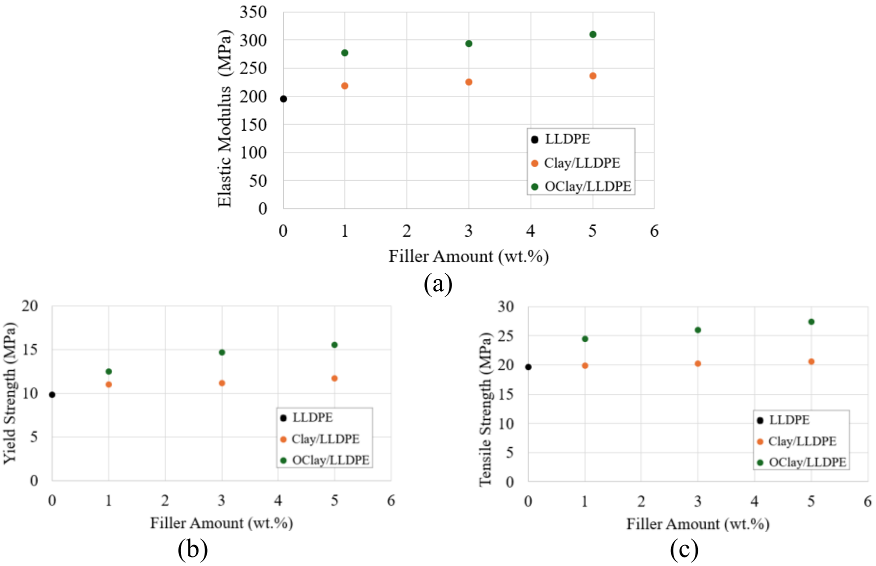
Figure 5. Shore D hardness values of pure LLDPE and its nanocomposites

All twenty samples of each LLDPE and its nanocomposites were examined from their stress-strain diagram, the results are given in Table 3 including their standard deviations for each mechanical property, and hardness values.

Table 3. Mechanical Properties of LLDPE and LLDPE Nanocomposites

Additives to LLDPE	**Elastic Modulus (MPa)**	**Yield Strength (MPa)**	**Tensile Strength (MPa)**	**Tensile Strain (%)**	**Resilience (MPa)**	**Toughness (MPa)**	**Hardness**
0	195,89±8,88	9,79±0,44	19,63±0,65	17,82±2,64	0,24±0,01	8,85±1,50	39,84±0,54
1 wt.% Clay	218,77±12,44	11,01±0,89	19,85±0,90	17,15±1,69	0,27±0,02	8,16±0,75	40,24±0,59
3 wt.% Clay	224,89±11,83	11,12±0,68	20,18±0,74	15,70±1,86	0,28±0,01	7,84±0,97	40,44±0,38
5 wt.% Clay	236,78±10,76	11,68±0,64	20,63±0,78	13,14±1,35	0,30±0,01	6,16±0,64	42,18±1,38
1 wt.% OClay	277,45±14,02	12,49±4,33	24,44±0,76	9,73±0,64	0,35±0,02	5,09±0,45	41,95±1,00
3 wt.% OClay	294,11±24,21	14,71±1,21	26,01±1,27	9,23±0,73	0,37±0,03	4,86±0,46	42,40±1,03
5 wt.% OClay	310,81±28,15	15,54±1,41	27,39±1,43	8,67±0,74	0,39±0,04	4,50±0,55	42,98±1,21

By addition of Clay and OClay up to 5 wt.% significantly most mechanical properties of LLDPE, except for tensile strain and toughness. Specifically, the addition of 5 wt.% Clay enhanced the elastic modulus, yield strength, tensile strength, resilience, and hardness of LLDPE by 20,87%, 19,31%, 5,09%, 25,00%, and 5,87 %, respectively. Similarly, incorporating 5 wt.% OClay increased these properties by 56,67%, 58,73%, 39,53%, 62,50%, and 7,88%, respectively. The stiffness of LLDPE evaluated based on elastic modulus improved from 195,89 MPa to 310,81MPa is the key parameter for many applications as given in Figure 6. 58,67% improvement on LLDPE's stiffness without using any compatibilizers or modifiers, just by adding 5 wt.% organoclay is a huge effect comparing to previous works done by other researchers (Hotta & Paul, 2004). However, tensile strain and toughness of LLDPE decreased by 26,27% and 30,40% with 5 wt.% Clay, and by 51,38% and 49,15% with 5 wt.% of OClay.



(a)

(b)

(c)

Figure 6. Comparison of a) Elastic Modulus, b) Yield Strength, and c) Tensile Strength of LLDPE and LLDPE Nanocomposites

The ANOVA results confirm that filler type, filler amount, and their interaction significantly influence the mechanical properties of LLDPE composites, with all p-values far below 0,05, providing strong evidence against the null hypothesis (Table 4). Among these factors, filler amount exhibits the strongest effect, as indicated by the highest F-values across elastic modulus (F = 13208,16), yield strength (F = 2412,40), and tensile strength (F = 26703,41), highlighting its dominant role in enhancing mechanical performance. Filler type also shows a notable effect, with F-values ranging from 59,23 to 191,11, while the interaction between filler type and filler amount, though statistically significant (F-values from 47,53 to 87,43), has a comparatively smaller influence. P-values are presented in scientific notation (e.g., $p < 1E-30$) to clearly convey their extreme significance. All p-values in Table 4 are well below conventional thresholds (e.g., 0,05 or 0,0001), underscoring the robust effects of filler type, filler amount, and their interaction on the mechanical properties of LLDPE composites. These findings highlight the critical role of optimizing filler amount to achieve improved mechanical properties in LLDPE composites.

Table 4. Analysis of Variance (ANOVA) for Elastic Modulus, Yield Stregth and Tensile Strength of LLDPE and LLDPE Nnaocomposites

	Elastic Modulus		Yield Strength		Tensile Strength	
Source of Variation	F	P-value	F	P-value	F	P-value
Filler Type	59,23	$p < 0,001$	106,14	$p < 0,001$	191,11	$p < 0,001$
Filler Amount	13208,16	$p < 0,0001$	2412,40	$p < 0,0001$	26703,41	$p < 0,0001$
Filler Type x Filler Amount	53,77	$p < 0,001$	47,53	$p < 0,001$	87,43	$p < 0,001$

Organophilic clay (OClay) layers, due to their finer dispersion within the LLDPE matrix, interact more readily with the non-polar LLDPE chains than do hydrophilic clay (Clay) layers. This results in improved compatibility of OClay with the LLDPE matrix. However, both Clay and OClay dispersed in an intercalated rather than exfoliated in LLDPE matrix revealed by XRD results caused increment in the standard deviation for each mean value of improved properties. Consequently, OClay exhibited a more positive effect on the mechanical properties of LLDPE compared to Clay at the same amount of addition.

4. CONCLUSION

In this study, LLDPE nanocomposites were fabricated via melt intercalation using 1-5 wt.% of nanosized clay and organoclay to enhance mechanical properties. The results showed that increasing additive concentration improved mechanical strength, albeit with a slight decrease in flexibility. FTIR and XRD analyses confirmed homogeneous dispersion and interaction between the fillers and polymer chains. The ANOVA findings further revealed that both the type and amount of the fillers have a statistically significant effect ($p<0,05$) on mechanical properties, providing a comprehensive understanding of the comparative performance of clay and organoclay at different levels. Specifically, organophilic organoclay exhibited superior interaction to the LLDPE matrix compared to hydrophilic clay, leading to greater mechanical enhancements. At 5 wt.% organoclay, the elastic modulus, yield strength, and tensile strength increased by 56,67%, 58,73%, and 39,53%, respectively. Additionally, the 5 wt.% OClay/LLDPE nanocomposite showed a 5% rise in melting temperature suggests expanded application potential in thermally demanding environments. Adding organoclay effectively enhances the durability of LLDPE and may help reduce microplastic generation, making it suitable for high-performance applications. Future studies could further explore its long-term stability and environmental resistance.

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Contribution of the Authors
Ebru Gunister: Conception, literature review, preparation and characterization of materials, analysis, and interpretation, writing, editing and funding.
Edgar Alejandro Ayala Iracheta: Preparation, testing, analysis, writing and editing.

Conflict of Interest
The authors declare that there is no potential conflict of interest for this manuscript.

Research and Publication Ethics Statement
Research and publication ethics were observed in this study.

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