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Research Article

Characterization of Olive Seed Powder Incorporated Low Density Polyethylene Composites

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ABSTRACT

Global warming, increasing production and consumption rates, environmental concerns have revealed the need for some innovative material studies, and studies on the use of polymeric composites prepared with natural based fillers have become widespread to increase environmental awareness and ensure sustainable production. Composite materials prepared by using easily accessible, affordable, lightweight, high-strength plant based fillers can be used in many areas. In this study, composites of low density polyethylene (LDPE), which is one of the most widely used thermoplastics, were prepared by injection moulding process using the waste seeds of olives (OS), which have an important place in Turkey's agriculture and economy, and the density, hardness (Shore D), spectroscopic (Fourier transform infrared (FTIR) spectroscopy), morphological (Scanning electron microscopy (SEM)), mechanical, thermal (Differential Scanning Calorimetry (DSC), Heat Deflection Temperature (HDT) and Vicat softening temperature) analyses of OS filled LDPE composites were performed. As a result of the study, an increase in hardness and elastic modulus values of OS filled LDPE composites was observed, while no noticeable decrease in thermal properties was seen.

1. Introduction

Environmental and ecological awareness, which has emerged as a result of increasing concerns about sustainability and environmental issues, has led to efforts to develop innovative materials in various sectors. Increasing environmental sensitivity and the enforcement of environmental regulations with greater seriousness, coupled with competitive cost concerns, bio based composite materials produced from environmentally friendly and renewable materials have become increasingly widely used to replace composite materials obtained from synthetic materials in this context [1–3].

When it comes to bio based composite materials, the first thing that comes to mind is composite materials reinforced with natural based fillers. In this context, plant based additives, which are considered as natural based fillers, are cost-effective and environmentally friendly materials that have come to the agenda as an alternative to the use of synthetic additives, suitable for many industrial applications, and can be used as reinforcement in the composite structures [4–6]. Their environmental impact is remarkable in that they do not cause chemical emissions in production and processing processes such as synthetic additives and minimise both fuel consumption and greenhouse gas emissions due to their light weight [7].

In addition to being environmentally friendly, one of the biggest advantages of composites containing plant based fillers is that they have lower density compared to synthetic additives. They have significant advantages over synthetic additives such as favourable cost, easy accessibility, high strength, high hardness, low density, fatigue and corrosion resistance, easy processability, high thermal and acoustic insulation performance [8–12].

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Studies have shown that plant based fillers can strengthen polymeric composites in general due to their low density, elongation tendency and high strength. In particular, fillers with higher surface area can be distributed more homogeneously, enabling a more compatible interface between the matrix and the reinforcement phase [13]. Plant based fillers are recognised as an alternative to synthetic fillers in various industries due to their comparable physical and mechanical performance [7, 14].

The automotive, construction, energy, sports, electronics, aerospace and aeronautics industries are the main areas of use of plant based composites [15]. Automotive companies such as Ford, Volkswagen, General Motors, Honda have started to use plant based fillers in parts such as trunk lids, floor coverings, ceilings, dashboard coverings, seat backs [16]. It is possible to witness the use of plant based composites in a wide variety of sectors and parts such as insulation boards, roof panels, door frames in the construction field [17], turbine blades in the energy field [18], rackets, golf clubs, surfboards in the sports field [15], telephone and computer cases and bodies in the electronics field [15, 19], interior panels and outer bodies in the aerospace field [15]. Commonly used plant based fillers can be listed as jute, kenaf, hemp, flax, bamboo, sisal, muaz bark, sugar cane [20–24]. These fillers have been studied with various thermoplastics and thermosets and are generally known to provide high toughness, acoustic absorption, improved thermal properties and corrosion resistance [7, 25, 26].

Polyethylene (PE), one of the most widely used thermoplastic materials in many sectors today, has various subclasses including low density PE (LDPE), linear low density PE (LLDPE), high density PE (HDPE) and ultra high molecular weight PE (UHMWPE) [27–29]. PE types, which have many features such as high processability, low cost, durability, chemical resistance, recyclability, can be used in many sectors such as packaging, construction, furniture, biomedical applications and industrial products by shaping with processes such as injection and extrusion [15, 30–33].

LDPE, is widely used in many industrial applications due to its versatile properties, usefulness and easy processability [34]. However, as a result of the insufficient properties of LDPE used alone in some applications and the increasing interest in the development of sustainable materials, studies have started to improve the properties of LDPE with various reinforcement phases. When the studies in the literature are examined, it is reported that the stiffness and tensile strength values of LDPE were improved in the study conducted by Rodriguez- Fabia et al. [35] by adding pulp fibre to the LDPE matrix, and in a study conducted by Taşdemir et al. [36], increases in the elastic modules and hardness properties of LDPE were observed in the use of wood fibre. In another study conducted by Gomes et al. [37] in which amazon palm fibre was used as the reinforcement phase, a decrease in strength was observed while an increase in the modulus of elasticity was reported.

Olive, which has an important place in the agriculture of Mediterranean countries, is widely used both with its fruit and oil and always stands out with its nutritional and economic value. While 90% of the world's olive cultivation is carried out in the Mediterranean basin, Turkey, with its approximately 170 million olive trees, ranks high in the world in terms of both the number of olive trees and olive production [38, 39]. Olives can be consumed as table olives after being harvested through various processes, or they can be obtained in the form of olive oil by separating the juice from the olive fruit. In high production and consumption cycles, olive seeds remain as plant waste without any added value. When the studies are evaluated, it is seen that olive seeds can be considered as biowaste and can be used especially in environmental studies such as water adsorption and heavy metal removal [40–43].

In this study, studies were carried out to evaluate olive seeds, a plant based filler, as a reinforcing phase and to prepare OS filled LDPE composites with OS concentrations ranging from 2.5% to 10% by injection moulding. In order to develop OS filled LDPE composites, the particle size and morphology of OS were first determined and its effect on the density, hardness, FTIR, SEM,

mechanical, DSC, HDT and Vicat temperature properties of OS filled LDPE composites were investigated. The aim of this study is to evaluate the use of OS waste from the agricultural sector as a reinforcing material for potential applications such as automotive, construction, sports and energy by producing LDPE based composites by injection moulding.

2. Experimental Details

2.1 Materials

Low density polyethylene (LDPE) polymer in powder form was supplied from the A+Plus Polimer in Izmir. Olive seeds (OS) were separated from olives collected from olive trees in the Marmara Region, dried and ground naturally and used.

1.1. Preparation of OS filled LDPE composites

Table 1 shows the sample codes and composite ratios used for OS filled LDPE composites. LDPE and OS powders were dried at 70 °C for 12 hours and after the moisture was removed, they were weighed in the specified proportions and blended in a ziplock bag until uniform according to the ratios given in Table 1. The OS filled LDPE composites were then injection moulded using a BOY/22A Pro Lab injection moulding machine at 205-210-205-200-200-200 ºC zone temperatures and 160 bar injection pressure. Samples were prepared according to the following standards: ISO 527-2 (Type 1A) for tensile test, ISO180 for impact test and ISO 75-2 for heat deflection temperature (HDT) tests.

1.2. Characterizations of OS, LDPE and OS filled LDPE composites

1.2.1. Density measurements

Density measurements of the LDPE and OS filled LDPE composites were calculated by taking into account the weights of the samples in air and water environments according to the Archimedes principle. Weight measurements were measured using Shimadzu-AUX321 brand/model precision balance. Three repetitions were performed for each sample.

Table 1. The ratios and sample codes of OS filled LDPE composites

Sample Code	LDPE, wt. %	OS, wt. %
LDPE	100	0
2.5OS	97.5	2.5
5OS	95	5
7.5OS	92.5	7.5
10OS	90	10

1.2.2. Hardness (Shore D) analysis

Shore D hardness tests of LDPE and OS filled LDPE Composites were carried out using the AMITTARI-HSM-SD-ST device at 23 °C temperature and 55% humidity in accordance with the ASTM D2240 standard, with 5 measurements for each sample.

1.2.3. Fourier transform infrared (FTIR) spectroscopy analysis

FTIR analyses, which were carried out to observe the existing functional groups and their changes in LDPE and OS filled LDPE composite structures, were performed with a Thermo Scientific Nicolet iS50 brand model FTIR spectrophotometer device. Spectra were recorded in the wavenumber range of 4000–600 cm^{-1} with 32 scan numbers and 4 cm^{-1} spectral resolution.

1.2.4. Particle size analysis

Particle size distributions of OS powders were characterized using a Malvern/Mastersizer 3000E brand/model laser particle size analyzer in the water phase as dispersant, and D10, D50 and D90 values representing 10%, 50% and 90% distributions of particle amounts were determined.

1.2.5. Scanning Electron Microscopy (SEM)

The morphological structures of LDPE and OS filled LDPE composites were examined with a Carl Zeiss/Gemini 300 brand/model scanning electron microscope and for this purpose, the fracture surfaces formed after the impact test were analyzed under 1000× magnification and 5 kV acceleration voltage. In order to provide conductivity to the samples, a thin gold/palladium layer coating process was performed before the analysis.

1.2.6. Mechanical tests

Tensile tests of the LDPE and OS filled LDPE composites were carried out at a speed of 100 mm/min according to the ISO 527-2 (Type 1A) standard. As a result of the tensile tests performed using the Shimadzu AGS-X universal testing machine, tensile strength (TS), elongation at break (EB) and modulus of elasticity (EM) values were analyzed. Impact tests were carried out using the Instron Ceast 9050 brand/model testing machine and a 5.5 J Izod hammer in accordance with the ISO 180/A standard, and as a result of the test, the Notched Impact Strength (NIS) values were evaluated.

1.2.7. Differential Scanning Calorimetry (DSC) analysis

DSC analyses of OS powder, LDPE sample and OS filled LDPE composites were carried out using TA Instruments/DSC250 brand/model device to investigate melting and crystallization behaviors and crystallinity ratios (Equation 1). Tests were carried out in the temperature range of -80 °C to 150 °C with a heating rate of 10 °C/min.

$$X_c = \frac{\Delta H_m}{\Delta H^\circ_m (1-w)} \quad (1)$$

While the ΔHm value in Equation 1 represents the melting enthalpy of LDPE, the ΔHom value represents the melting enthalpy of the completely crystalline form of the polymer, and this value is accepted as 285 J/g in the literature [44]. The *w* in the equation represents the weight ratio of the polymer in the mixture.

1.2.8. Heat Deflection Temperature (HDT) and Vicat softening temperature measurements

HDT tests of LDPE and OS filled LDPE composites were conducted according to the B method of the ISO 75-2 standard with a flexural stress of 0.45 MPa and a heating rate of 120 °C/h, and the Vicat softening temperature tests were carried out according to the A50 method of the ISO 306 standard with a force of 10 N and a heating rate of 50 °C/h using an Instron/Ceast HV3 brand/model testing device.

2. Results and Discussion

2.1. Characterization of OS powders

Particle size distribution and SEM image results of OS powder used in the composites are presented in Figure 1.(a-b), respectively. A single peak is seen in the particle distribution graph of OS powder in Figure 1.a. The D10, D50 and D90 values of OS powder were found to be 15.4 µm, 53.4 µm and 125 µm, respectively. In the SEM image given in Figure 1.b, it is seen that OS powder particles have an irregular shape and consist of particles of different sizes.

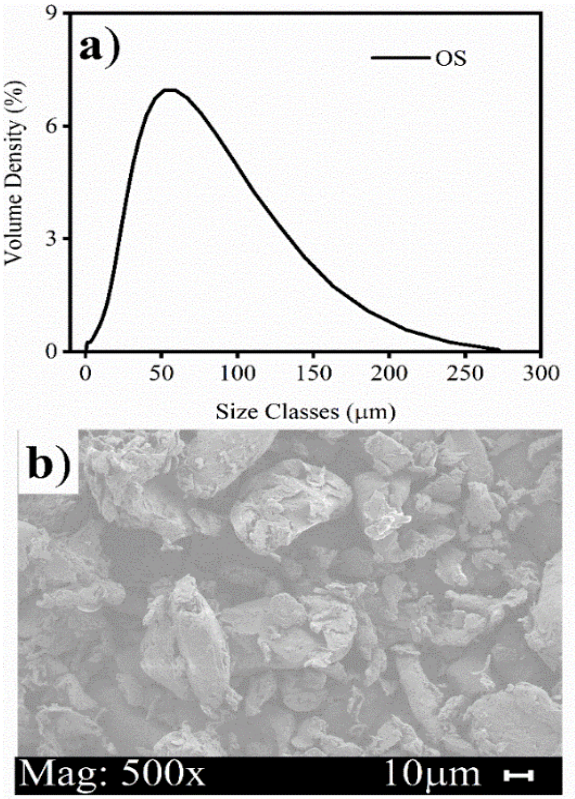

Figure 1. a) Particle size distribution and b) SEM image under 500× magnification of OS powder

2.2. Characterization of LDPE and OS filled LDPE composites

2.2.1. Physical properties

Density and hardness (Shore D) values of LDPE and OS filled LPDE composites are given in Table 2. It is observed that density values increase as OS amount increases in composites. Since the densities of plant based additives are generally in the range of 1.10-1.60 g/cm^3, the increase obtained in the study was found to be consistent with the literature [45, 46]. When the

hardness values in Table 2 are examined, although a small increase was observed with the addition of 2.5% OS, no significant change was observed in the other filled composites. This small increase is associated with an increase in the surface hardness of the composite when the surface hardness of the natural fibre is harder than that of the matrix [36, 47].

Table 2. Density and hardness test results of LDPE and OS filled LDPE composites

Analysis	LDPE	2.5OS	5OS	7.5OS	10OS
Density (g/cm^3)	0.9161±0.0005	0.9243±0.0005	0.9329±0.0002	0.9421±0.0010	0.9493±0.0016
Hardness (Shore D)	42.8±0.6	45.2±0.9	45.0±1.2	45.6±1.1	46.0±0.6

2.2.2. Fourier transform infrared (FTIR) spectroscopy analysis

FTIR spectra of LDPE and OS filled LDPE composites are given in Figure 2. When the spectra are examined, the characteristic peaks of the LDPE polymer are clearly observed. Among these peaks, the most characteristic one is the peak at 2918 cm^{-1} which represents the asymmetric stretching vibration of the CH2 molecule and the peak at 2851 cm^{-1} which represents the symmetric stretching vibration of the CH_2 molecule. Apart from this, the bending deformation at the wave number 1468 cm^{-1}, the symmetric deformation at the wave number 1370 cm^{-1} and the rocking deformation peaks at the wave number 716 cm^{-1} are also related to the functional groups in the LDPE structure [48, 49].

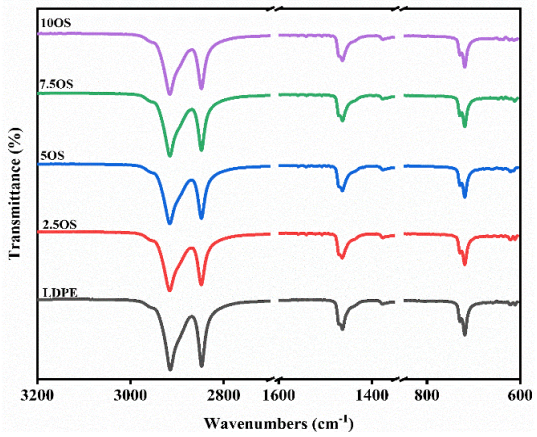

Figure 2. FTIR spectra of LDPE and OS filled LDPE composites

It can be mentioned that the addition of OS powder into the LDPE matrix does not cause a significant change in the FTIR spectrum. This situation shows that there is no chemical interaction between LDPE and OS powders. The absence of significant differences in the peaks and peak intensities supports the existence of physical intermolecular interactions such as hydrogen bonding and Van der Waals between LDPE and OS powders [50, 51].

2.2.3. Scanning electron microscopy (SEM)

SEM images of the LDPE and OS filled LDPE composites under 500× magnification are presented in Figure 3.

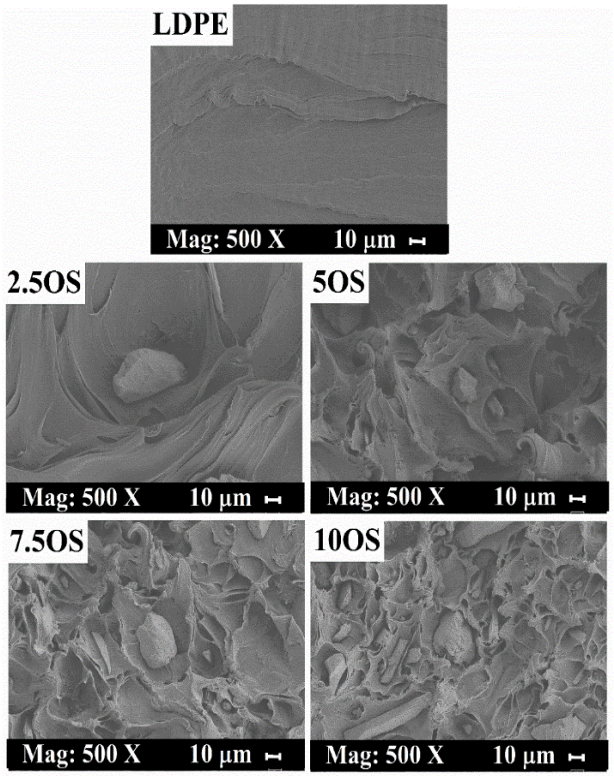

Figure 3. SEM micrograms of LDPE and OS filled LDPE composites at 500× magnification

When the SEM images in Figure 3 are examined, the fracture surface of LDPE exhibited a flatter fracture performance, while the addition of OS powder to the composite structure made the surface rougher. The SEM images of the

composites with OS powders show that there are large gaps between the OS particles and the matrix. In addition, the absence of LDPE matrix residues on the OS powder particles indicates that the surface interaction between LDPE and OS powders is low [52]. In composites containing higher amounts of OS powder, the increase in the amount of OS on the fracture surface caused these gaps to increase. This situation supports the decrease in TS, EB and NIS values from the mechanical test results in Table 3 and Figure 4 [52, 53].

2.2.4. Mechanical properties

The results of tensile and impact mechanical tests of LDPE and OS filled LDPE composites are given in Figure 4.(a-b) and Table 3.

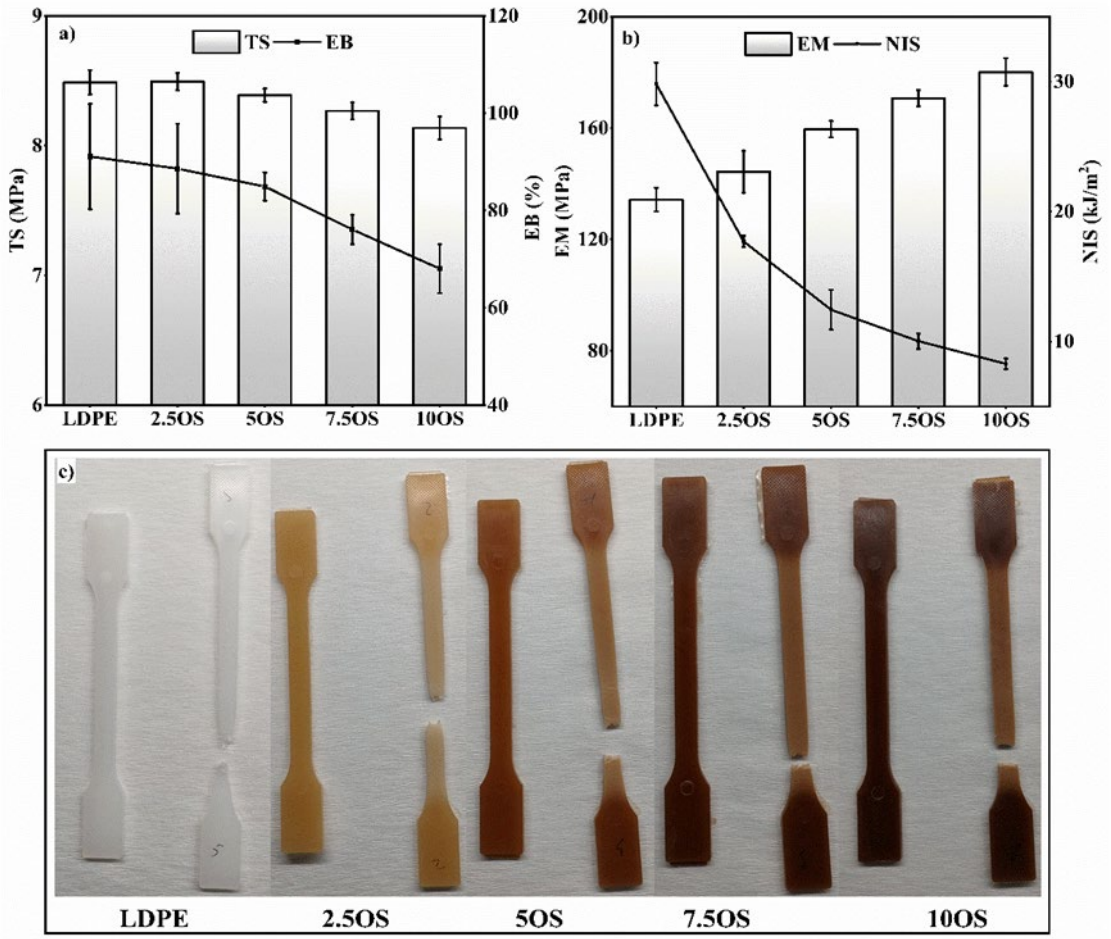

Figure 4. Changes in a) TS-EB, b) EM-NIS values according to varying OS ratios in LDPE composites and c) images of the tensile test bars

As shown in Figure 4.c, the colour of the composites changed from mustard to dark brown as the amount of OS increased. In addition, the test bars showed a homogeneous distribution in colour. Examining the EM values of the composites, it can be seen that the EM value of LDPE is 134 MPa, while it increases to 180 MPa in the 10% OS filled material, an increase of approximately 50%. A regular increase was observed as a function of the amount of OS in the composites containing 2.5, 5 and 7.5% OS powder. Tronc et al. [54] reported in their study that significant EM increases occurred. Arrakhiz et al. [55] also found in their study that the EM value of pure PP material increased from 1034 MPa to 1541 MPa with the addition of 25% pinecone dust. It was found that the reason for these increases obtained in both studies was due to the use of a harder filler material than the matrix. On the other hand, as the amount of OS in the LDPE matrix increases, the TS, EB and NIS values decrease. The decrease in these values is associated with the negative effect of the matrix on the force distribution under stress,

as seen in the SEM images of OS powders (see Figure 1 and Figure 3) [56]. In addition, the brittle structure of natural fibers also supports the decrease in EB values [54, 57]. When the rupture end points in Figure 4.c are carefully examined, it can be seen that while ductile rupture is observed in LDPE, more brittle rupture occurs as the amount of OS increases.

2.2.5. Differential Scanning Calorimetry (DSC) analysis

DSC analyses were performed to examine the temperature dependent heat transfer performances of the composites, DSC thermograms are presented in Figure 5 and the analysis results obtained from DSC thermograms are presented in Table 4.

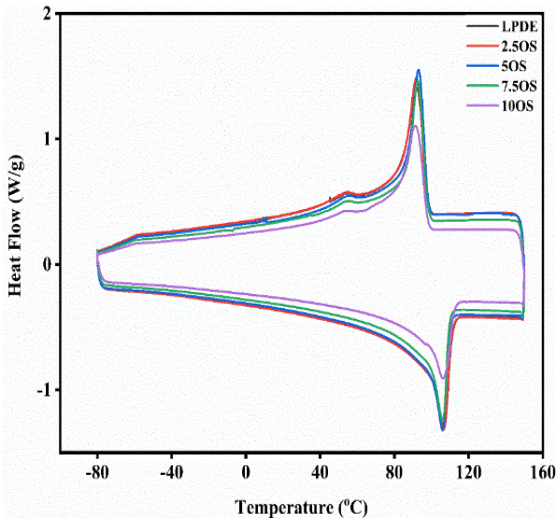

Figure 5. DSC thermograms of LDPE and OS filled LDPE composites with heating and cooling curves

As the amount of OS in the LDPE matrix increases, fluctuations are observed in both Tc and Tm temperature values. When we examined the Xc values, there were no significant changes in the Xc value in OS filled materials with a ratio of 2.5-5%, while decreases were observed in materials with a ratio of 7.5-10%. This can be explained by the fact that the use of OS filler at 2.5-5% has no significant effect on crystal formation in LDPE polymer, while the use of OS filler at 7.5 and 10% has an inhibitory effect on crystal formation [58]. Furthermore, the cellulose content of the plant based filler impedes heat transfer and diffusion between the LDPE molecular chains within the composite [59].

3.2.6. Heat Deflection Temperature (HDT) and Vicat softening temperature analysis

Graphical representation of HDT and Vicat softening temperature is given in Figure 6. In both values, no significant change was observed due to the increase in the amount of OS. Especially since it is known that the HDT temperature change depends on the thermal transition temperature of the matrix material and the change in the crystal structure, it can be interpreted that results are obtained in parallel with the Xc values obtained as a result of DSC analysis [60].

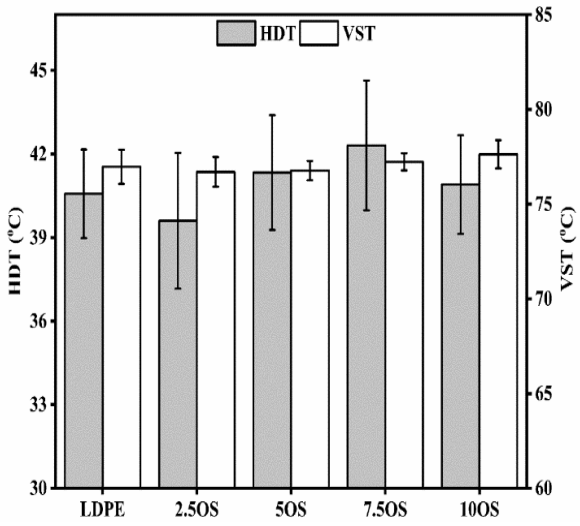

Figure 6. Changes in HDT and Vicat softening temperature according to varying OS ratios in LDPE composites

Table 3. Mechanical test results of LDPE and OS filled LDPE composites

Analysis	Parameter	LDPE	2.5OS	5OS	7.5OS	10OS
Tensile	TS (MPa)	8.490 ± 0.09	8.490 ± 0.07	8.390 ± 0.05	8.270 ± 0.07	8.140 ± 0.09
	EM (MPa)	134.3 ± 4.26	144.3 ± 7.53	159.6 ± 2.95	170.7 ± 2.93	180.1 ± 4.95
	EB (%)	91.10 ± 10.8	88.60 ± 9.22	84.91 ± 2.9	76.09 ± 3.03	68.02 ± 5.02
Impact	NIS (kJ/m^2)	29.82 ± 1.64	17.70 ± 0.44	12.45 ± 1.53	10.03 ± 0.59	8.290 ± 0.4

Table 4. DSC analysis results of LDPE and OS filled LDPE composites

Parameter	LDPE	2.5OS	5OS	7.5OS	10OS
Tc (°C)	92.10	91.91	93.000	92.60	91.50
ΔHc (J/g)	108.0	108.5	106.3	98.61	94.12
Tm (°C)	106.7	106.5	106.1	105.9	106.1
ΔHm (J/g)	108.9	108.3	105.9	100.0	84.20
Xc (%)	38.20	39.00	39.10	37.90	32.80

4. Conclusion

LDPE is one of the most widely used thermoplastic matrices worldwide. In some application areas, the production of composite materials with natural fillers added LDPE composites has gained importance when its properties are insufficient and the need for improvement is combined with both environmental pollution and cost reduction concerns.

In this study, in order to reduce the unit cost and to obtain more environmentally friendly material, OS was added into the LDPE matrix and characterisations were carried out. It was observed that as the amount of OS in LDPE increased, the density increased slightly in line with the densities of natural based fillers. While the hardness changes of LDPE composites filled with OS at varying ratios were close to each other, an increase was obtained compared to LDPE polymer. No chemical interactions were observed in the FTIR spectra, indicating that there are intermolecular physical interactions between OS powder and LDPE. In the SEM images, the gaps between the OS particles and the LDPE matrix interface supported the decrease in TS, EB and NIS values. In order to increase the weak interfacial interactions between the matrix and the filler material, it has been observed in the literature that binding agents are used and the interactions are increased.

There is a requirement for the use of a binding agent to achieve improved mechanical performance. When the EM values were analysed, it was observed that while the EM value of LDPE polymer was 134.3 MPa, this value increased up to 180.1 MPa with the use of 10% OS. When the thermal properties were analysed, small changes were observed in both Tc and Tm values. However, while there was no significant change in Xc values up to 7.5% filled OS, a decrease was observed in 10% OS composite. No significant change was observed in HDT and Vicat softening temperature values. Once the materials have been characterised, it will be possible to determine the areas in which such composites can be used, depending on their performance, their intended use and their cost. For example, if impact resistance is not desired, but instead cost-effectiveness is desired, the 4.2% decrease in TS value of 10OS material compared to LDPE polymer can be ignored. At the same rate, the EM value was also found to be maximum. In the future studies, it will be possible to obtain higher mechanical performance at higher OS ratios, taking into account the cost with the use of the necessary binding agents.

Article Information Form

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Authors' Contribution
Sibel TUNA: Conceptualization, Investigation, Methodology, Formal analysis, Writing, Review & editing.
İbrahim ŞEN: Conceptualization, Investigation, Methodology, Resources, Review & editing.

The Declaration of Conflict of Interest/ Common Interest
No conflict of interest or common interest has been declared by authors.

The Declaration of Ethics Committee Approval
This study does not require ethics committee permission or any special permission.

The Declaration of Research and Publication Ethics
Authors of the paper declare that they comply with the scientific, ethical and quotation rules of SAUJS in all processes of the paper and that they do not make any falsification on the data collected. In addition, they declare that Sakarya University Journal of Science and its editorial board have no responsibility for any ethical violations that may be encountered, and that this study has not been evaluated in any academic publication environment other than Sakarya University Journal of Science.

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