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Enhancement of mechanical properties in unsaturated polyester via reinforcement with olive leaf particles

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HIGHLIGHTS

- A new type of polymer matrix composite, pls and plo, was used.
- Adding particles significantly enhanced impact resistance for all samples.
- Thermal conductivity decreased for all samples.
- FTIR spectra showed chemical adhesion between olp and the matrix, confirmed by SEM photomicrographs.
- The particles can be used in mechanical applications requiring high mechanical and thermal insulation properties.

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ABSTRACT

The use of olive leaf powder (OLP) as a filler material in unsaturated polyester composites brings a new avenue for the development of mechanical and thermal properties. The present work focuses on the impact of the relative proportions of OLP ranging from (0, 5, 10, 15, 20, and 25%) on the structural, electrical, and thermal properties of the composites.The specimens were prepared through the hand lay molding process, and the hardness strength, impact strength as well as compressive strength examination were conducted.These findings suggest that the characteristics of these composites improve as the content of OLP increases.The maximum impact strength found is 2.52 KJ/m², and the maximum hardness is 7.24 N/mm², while the compressive strength is around 48.7 MPa. Thermal conductivity decreased on the volumetric fraction of 25%, which is within the range of 0.101 W/m·℃. Based on the analysis of the FTIR results, it can be claimed that the change in intensities and positions of the peaks may be associated with the interaction between the OLP and the polyester matrix. The SEM data suggest that the OLP is dispersed throughout the matrix, and strong interfacial adhesion exists between them. In summary, this work highlights the prospects of using OLP-filled unsaturated polyester composites for several purposes due to the improved mechanical and thermal characteristics.

1. Introduction

Over the past few years, increasing attention has been directed towards the surface formation of polymeric composites that incorporate natural plant-based powders due to their inherent biodegradation properties [1]. Polymer matrix composites have been widely used in many applications due to their attributes, such as corrosion resistance and superior fracture and fatigue behaviors, as well as high modulus and specific strength [2,3]. Across the timeline, people have strived to maximize the use of available resources to improve the quality of life, which has spurred innovation and exploration for replacements for conventional products [4]. Specifically, composite material studies have produced new substitutes, including Routinely, MMCS:Metal Matrix Composites, CMCS: Ceramic Matrix Composites, and PSCS: Polymeric Composites, where the characteristics exhibit similarity to conventional materials in various industries [5,6]. Despite the limited usage of natural fibers or particles for reinforcing polymeric composites, recent studies reveal the way of applying them promising to offer better mechanical characteristics and even to compete with steel counterparts [7,8]. The technology behind composites is based on the ability to merge some materials at one time to produce a specified set of features that the individual components do not possess, which necessitates the invention of improved materials to meet industry demands [5]. Moreover, the incorporation of natural fillers, such as cellulose from sources like coconut shell, wood, pineapple leaf, sidr leaf, and palm kernel shell, in thermoplastic and thermoset polymer composites has garnered attention due to its potential to reduce costs, enhance productivity, and improve product mechanical properties [1-8].

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For instance, previous research has explored the effects of untreated and treated coconut shells on unsaturated polyester composites, revealing enhancements in mechanical and thermal properties [9]. Additionally, a study by researcher Russell Salah in investigated the mechanical characteristics of epoxy reinforced with powdered Ziziphus Spina-Christi bark, demonstrating improvements in mechanical properties with certain weight fractions of the reinforcement material [10]. The use of olive leaves reinforcement in polymer composites comes with the following benefits. Olive leaves are obtained easily from the olive industry as they are the product by-products, making them sustainable [11]. Using this agricultural waste involves managing the waste as well as supporting the use of the materials as green materials [1]. Furthermore, considering the usage of olive leaves in place of synthetic fillers and fibers further reduces the cost of production [8]. Olive leaves the mechanical properties of polymers; features such as tensile strength, compressive strength, and impact strength are raised by the cellulose, hemicellulose, and lignin present in them [12]. In addition, the uptake of olive leaf particles enhances the thermal resistance in addition to the thermal transmittance of the polymer matrix [13]. Composites with natural fiber reinforcement, such as olive leaves, are also more eco-degradable, making them environmentally sustainable [14]. Conducted research in the recent past has demonstrated that the use of olive leaves could improve the properties of polymer composites according to the study by [15], the incorporation of olive fibers into low-density polyethylene yielded improved flexural and impact properties, where the overall best performance was given at an optimum fiber concentration of 40 percent. Valvez et al. [16], demonstrated a literature review highlighting the mechanical advantages and pros of polymer filler via olive stones. Sarmin et al. [17] also explained that epoxy composites reinforced with olive tree branches experienced enhancements in tensile, flexural, and impact strengths because of established interfacial adhesion. Senthilkumar et al. [18] also concluded that the incorporation of natural olive tree leaves powder and pineapple fiber increased the impact strength. However, the bending strength of hybrid composites was slightly lesser. Senthilkumar et al. [19] analyzed the interactions between olive trunk leaves powder reinforcement and pineapple leaf fiber reinforcement in epoxy matrix composites and noted an enhanced tensile strength and thermal stability with the increment in the pineapple fiber reinforcement. Last but not least, Sarmin et al. [20] proved that the physical and mechanical properties of olive biomass/bamboo fiber hybrid composites presented the highest construction applications. Taken together, all these studies demonstrate the effectiveness of olive leaves or other types of biomasses in enhancing the performance of polymer composites, also in terms of mechanical and thermal properties while the sustainability is greatly enhanced. This study, therefore, seeks to add to this line of research by examining the mechanical and physical characteristics of unsaturated polyester reinforced with olive leaf powder.

2. Experimental procedure

2.1 Unsaturated polyester resin

The unsaturated polyester resin (UPE) utilized in this study is a transparent liquid with low viscosity and a density of 1.2 g/cm³. To solidify the resin, a transparent hardener containing methyl ethyl ketone peroxide (MEKP) is added at a ratio of 2 g per 100 g of resin. Additionally, a cobalt catalyst, a dark-colored liquid in droplet form, is included at a ratio of 0.2 g per 100 g of resin to accelerate the curing process. After approximately 30 minutes, the resin undergoes a transition into a gel-like substance at ambient temperature.

2.2 Reinforcement material

Olive leaves (OL) were sourced from the Olive plant in Iraq, specifically in Anbar Governorate, using specialized mechanical techniques. Chosen for its favorable mechanical and thermal properties, widespread availability in nature, and economical price, as shown in Figure 1a the Olive leaves were subjected to washing with distilled water, cleaning, and subsequent drying in a hot air oven at 100 °C for 20 minutes. Following drying, the material was finely ground into a powder, as shown in **Error! Reference source not found.**b, and then deposited into the mold based on the specified volume fractions.

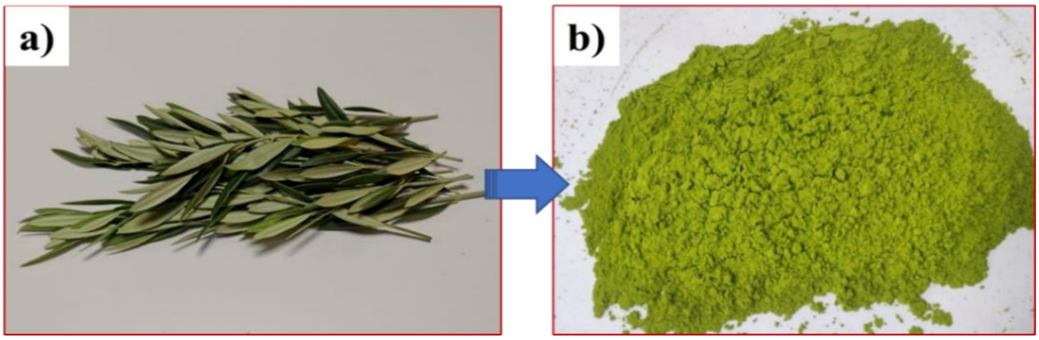

Figure 1: Olive leaves (a) before grinding: Raw olive leaves, and (b) after grinding: Olive leaves powder For composite reinforcement

2.3 Sample preparation

Manual molding was employed to prepare the samples using an aluminum mold. The mold was cleaned with ethyl alcohol and coated with a lubricating substance to prevent sample adhesion post-solidification and heat treatment. The unsaturated polyester resin was weighed using a sensitive balance according to the required quantity. Additionally, the weight of olive leaf powder and other additives, such as carbon fibers and copper oxide, was measured using the same balance after determining the volumetric fraction of particles ($V.f$) via the weight fraction of particles (Ψ). This calculation was performed using volumetric relationships and mathematical Equations (1, 2, 3) [21]:

$$V.f = \frac{1}{1+\frac{1-\Psi}{\Psi} x \frac{\rho_f}{\rho m}} \tag{1}$$

$$\Psi = \left(\frac{W_f}{W_c}\right) x100\% \tag{2}$$

$$W_c = W_f + W_m \tag{3}$$

where, W_c, W_m, W_f, are the weights in grams of the reinforced material, the matrix, and the composite material, respectively. ρ_f, ρ_m are the density of the reinforced and composite materials in g/cm³, respectively. When pouring the olive leaf particle samples, a small amount of resin is initially placed in the mold after adding the hardener and olive leaf particles.The resin is then poured into the mold, and the process is repeated according to the specified volumetric fractions. Four samples were poured in total. The samples are left inside the mold to solidify. Subsequently, heat treatment commences by placing the samples inside an electric oven at a temperature of 50 °C for 60 minutes to complete the solidification process and achieve optimal interweaving of polymer chains, eliminating any stresses that occur during the pouring process.The samples remain in the oven until the gradual cooling process is complete and the oven temperature reaches room temperature. Following this, the samples are removed from the oven and the mold, preserved in storage bags and prepared for inspection using testing devices.

3. Experimental characterization and mechanical analysis

3.1 SEM test

FE-SEM using a Philips XL30 (Philips, UK.) characterization tests were conducted at the Nanotechnology and Advanced Materials Research Center at the University of Technology, Baghdad- Iraq. FE-SEM characterization entails imaging of the samples in high resolution and features the surface morphology of the sample at the nanoscale.

3.2 FTIR test

In this study, the FTIR measurements were conducted using a Shimadzu FTIR-1800, a high-precision spectrometer manufactured in Japan. This device, known for its accuracy and reliability, was employed to analyze the chemical structure of the polymer composites. The measurements were carried out at the BPC Analysis Center in Baghdad, Iraq, ensuring rigorous standards and precision in capturing the infrared spectra of the materials. This facilitated a detailed comparison of the molecular interactions within the pure unsaturated polyester and the polyester reinforced with olive leaf powder.

3.3 Impact test

The impact strength (G_c) was calculated for all samples using an impact testing apparatus based on the energy required for fracture divided by the sample's cross-sectional area. The tests were conducted at room temperature. Samples for the impact test were prepared to standard dimensions of 4 x 10 x 80 mm without notches in accordance with ISO 179, using the Charpy impact testing device. This device measures the energy required to fracture a sample using a hammer that strikes the sample at room temperature. The impact strength (G_c) is calculated using the following Equation 4 [22]:

$$G_c = \frac{U_c}{A} \tag{4}$$

where, G_c is impact strength (kJ/m²), U_c refers to fracture energy (k J), determined from the Charpy impact test instrument, and A is the cross-sectional area (mm²). **Error! Reference source not found.** shows how samples affect the result before the test and after the test. In (a), the samples are captured when they are in their original state before being tested in the impact test. In contrast, (b) displays the samples as they are after testing with the effects of the impacts shown by the alterations. This comparative visual also helps to determine the level of impact and overall score and compare the results of the samples with impact conditions.

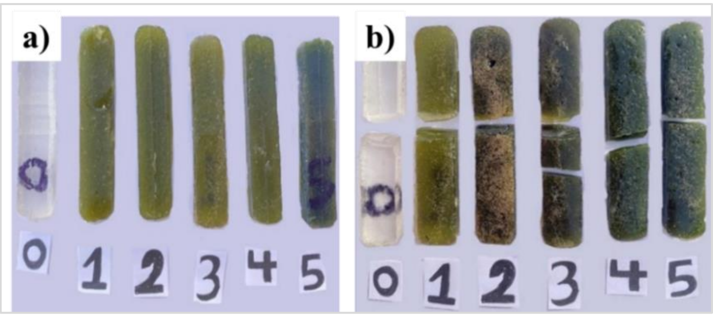

Figure 2: Impact samples (a) before the test and (b) after the test

3.4 Hardness test

The hardness of the samples was measured using the Shore D method with the HUATEC GROUP Hardness Tester HT-6600C Shore D, manufactured by HUATEC, China. The device features a needle-shaped penetration instrument that pierces the sample's surface to measure its hardness rating. Hardness tests were conducted at a laboratory temperature of 27 °C. The samples were prepared according to ASTM-D 2240 specifications [23]. Hardness testing of polymer samples helps assess material cohesiveness and durability. This experiment aimed to evaluate the surface hardness of polymer composites with varying reinforcing ratios. The hardness value of each sample was determined by averaging ten readings per sample.The tests were conducted at 27 °C.

3.5 Compression test

The compression test measures the strength and rigidity of a vertical columnar sample when pressure is applied to its ends. The equations and symbols for stress ($\sigma\sigma$), strain ($\epsilon\epsilon$), and modulus (E) used in tensile force analysis are also applicable to compression force analysis, referred to as compression strength and compression modulus, respectively [24]. The samples were tested using the Laryee Yaur testing solution device, manufactured by Laree Technology Co. Ltd., China, and located in the Advanced Materials Laboratory at Anbar University's College of Education for Pure Sciences. This test calculates the compressive resistance of the samples when a compressive load is applied at a strain rate of 5 mm/min, yielding resistance results directly from the device's graph. Samples were prepared to standard dimensions in accordance with American standard specifications.

3.6 Thermal conductivity test

The thermal conductivity test was conducted using Lee's disk apparatus, manufactured by Griffin and George Company in England. The thermal conductivity (K) was calculated using Equation 5, and the heat flow rate per unit time (H) was determined via the sample's cross-sectional area using Equation 6 [24].

$$K\left(\frac{T_B - T_A}{T_S}\right) = e\left[T_A + \frac{2}{r}\left(d_A + \frac{1}{4}d_s\right)T_A + \frac{1}{2r}d_S d_B\right] \quad (5)$$

$$H = IV = \pi r^2 e(T_A + T_B) + 2\pi re\left[d_A T_A + d_s \cdot \frac{1}{2}(T_A + T_B) + d_B T_B + d_C T_C\right] \quad (6)$$

E is the heat flux measured in watts per square meter per degree Celsius (W/m²·°C). H is the time rate of energy transfer to the inductance. T_{A},$_{B}$,T_C are Temperatures of disks A, B, and C in °C, d_A, d_B, d_C are the thicknesses of the copper discs A, B, and C in mm. d_s is the sample thickness in mm, V is the voltage supply to the circuit in volts, and r is the radius of each disk in mm. Figure 3 provides the layout of the test for the thermal conductivity equipment. The following shows the experimental setup, which consists of the apparatus for the thermal conductivity measurement connected to a DC power supply, wires, and three disks: A, B, and C.

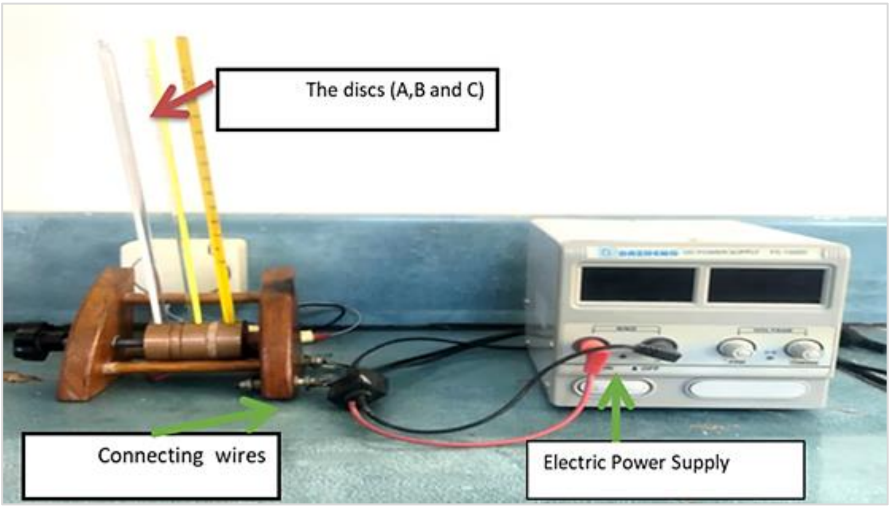

Figure 3: Thermal conductivity equipment's test

4. Results and discussion

4.1 SEM results

The change in the surface topography illustrated in FE-SEM images of UP before and after the addition of olive leaf powder at 5% indicates remarkable modifications in the composite structure (see Figure 4). As it can be observed before the addition of OLP see **Error! Reference source not found.**a. the surface of the UP seems rather smooth, implying the presence of a homogenous polymer matrix. However, the introduction of OLP see **Error! Reference source not found.**b is marked by the formation of some new protrusions and cracks illustrated by the yellow arrows clearly pointing toward different structures and compositions [25]. Some of the factors that may have led to these changes include the following: The nature of OLP particles' morphology as being heterogeneous disrupting the homogeneity of the matrix; there is increased surface area that encourages reaction and development of cracks; the application of mechanical stress during processing results into internal stress; and lastly there could be chemical interaction that changes the property of a polymer. These changes may also be attributed to the thermally induced expansion of the UP and OLP material coefficients, changes to crystallinity, or planar molecular orientations. In general, the observed major surface roughness and cracks within the UP-OLP composite can be attributed to the incorporation of OLP and processing parameters [26-28].

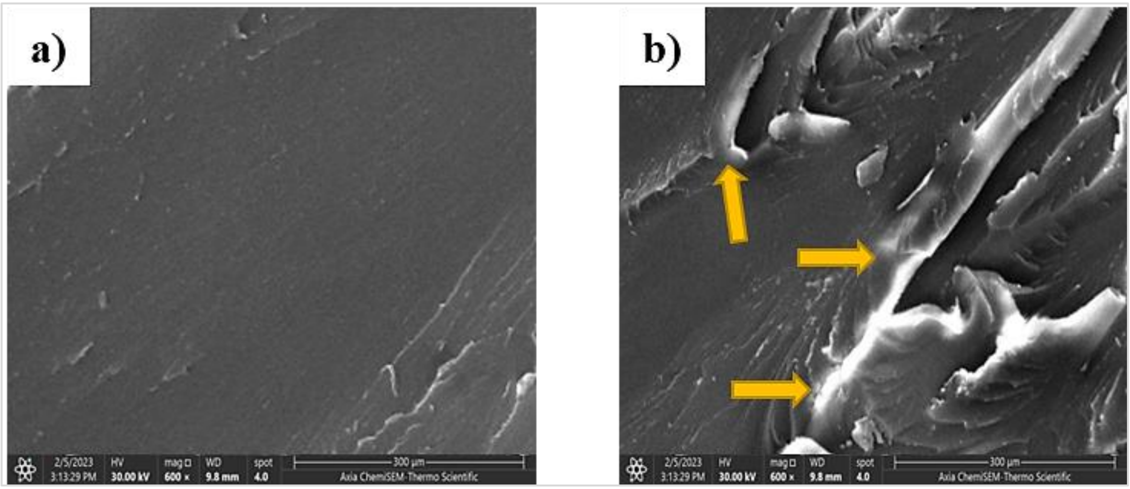

Figure 4: FE-SEM images representing the surface structural of unsaturated polyester (UP) without (a) and with (b) 5% of olive leaf powder (OLP)

4.2 FTIR results

In the studies done to compare the FTIR of pure unsaturated polyester with that of polyester reinforced with 15% olive leaf powder, as an example, it was observed that there were significant shifts and alterations in peak intensities in the range of 2800 cm^{-1} to 3000 cm^{-1} [29] as shown in **Error! Reference source not found.**. For the pure polyester, the first peak at 2877 cm^{-1} with a height of 5.2 shifts to 2852 cm^{-1} with a reduced height of 4.9 upon reinforcement.This change shows there is physical contact between the olive leaf particles and the polymer matrix, leading to a change in the frequency of the vibrational energy of C-H stretching bonds.The reduction of the height of the peak is an indication that the concentration or interaction strength of these bonds is reduced by the incorporation of olive leaf particles.

The second peak, coming from wavenumber 2917 cm^{-1} with height 3.1, moves slightly down to 2918 cm^{-1} and increases in height to 7. This change indicates that the bond characteristics between the polyester and the olive leaf particles are strengthened due to increased interaction or new bond formations, thus increasing the absorption intensity.The third peak changes from 2950 cm^{-1} with an intensity of 7 to 2960 cm^{-1} with an intensity of 1.6, meaning that the olive leaf powder interferes with the order of the polymer matrix and decreases the amount of bonding capacity of the related groups. Altogether, these observations show that the introduction of olive leaf powder into polyester alters the chemical configuration and intermolecular forces and, hence, the vibrational quantum states and the absorption profile of the resultant composite.

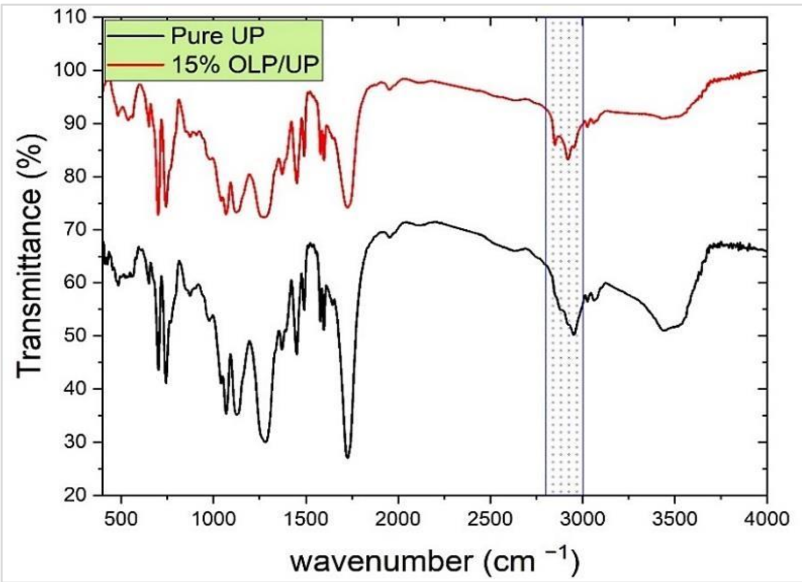

Figure 5: FTIR curves of pure unsaturated polyester and polyester reinforced

with 15% olive leaf powder

4.3 Impact results

The impact test evaluates the energy absorbed by polymer materials and their composites with varying reinforcement ratios during fracture, determining their ability to withstand external impact forces. **Error! Reference source not found.** illustrates the improvement in impact strength for all samples reinforced with powdered olive leaves. Specifically, incorporating olive leaf particles as reinforcement at a volume fraction of 5% resulted in an increase in impact strength by 0.89 kJ/m² compared to the pure sample. The energy required to fracture the unsaturated polyester without reinforcement is lower than that for the composites reinforced with olive powder.The increase in fracture energy for the composite materials is attributed to the olive particles absorbing some of the impact force and acting as barriers to fracture propagation.This barrier effect is determined by the strength of the interface between the particles and the matrix, influencing the transmission of the fracture through the interface.

The physical mechanism behind this improvement can be explained by the interaction between the olive particles and the polyester matrix. When an external impact force is applied, the particles disrupt the continuity of the matrix, causing the crack to deflect and absorb more energy before propagation.This crack deflection and energy absorption mechanism significantly enhance the material's toughness. Similarly, other samples showed increased impact strength at volume fractions of 10%, 15%, 20%, and 25%. The highest impact strength was observed at 25%, with an increase of 1.99 kJ/m² compared to the pure sample. This improvement is due to the strong adhesion between the reinforcing powder and the polymer matrix, creating a robust interfacial surface. This enhances the material's ability to withstand external stresses and loads, ultimately improving its mechanical properties, such as impact strength, as noted by the researcher in reference [30].

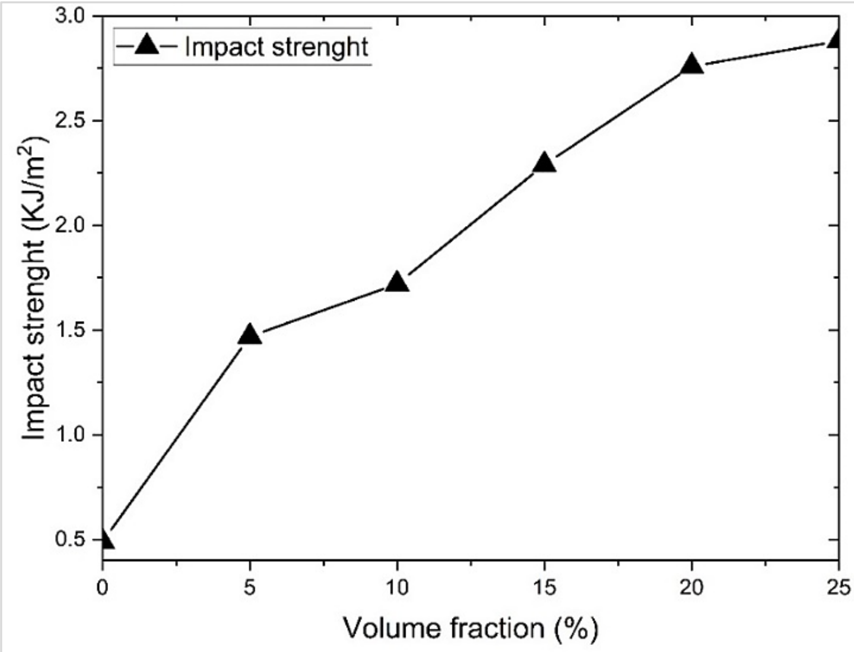

Figure 6: Impact test results of specimens

4.4 Hardness test

Conducting a hardness test on polymer samples helps ascertain the material's cohesiveness and durability.This test was performed to determine the surface hardness of polymer materials and their particle composites at different reinforcing ratios. Recorded measurements for each sample were averaged to establish the hardness value.The experiment was carried out at a laboratory temperature of 28 °C. **Error! Reference source not found.** demonstrates that adding olive leaf particles at a 5% volume fraction to the matrix material increases the sample's hardness by 11.8 units compared to the pure sample.This increase indicates that the added particles effectively penetrate the polymer chains, enhancing the composite material's hardness. Similar trends were observed for samples with volume fractions of 10%, 15%, 20%, and 25%. The sample with 25% olive leaf particles exhibited the highest hardness value, showing an increase of 32.3% compared to the pure sample.

The observed improvement in hardness can be attributed to several physical mechanisms. Olive leaf particles themselves are hard, and they restrict the ‘free flow’ of polymer chains in the composite, thus contributing toward enhanced overall hardness of the composite. The present complex interface between the olive leaf particle and the polyester matrix also limits the diffusion of the polymer chains. It hinders deformation arising out of stress, thereby increasing hardness. This results in evenly distributing the olive leaf particles in the matrix, and hence providing a reinforcing effect across the whole surface, which in turn enhances the overall mechanical properties of the composite, such as hardness. Furthermore, the dispersion of hard particles within the polymer matrix ensures a good stress distribution between the softer polymer phase and the stronger hard particles to reduce indentation and deformation of the material.These physical explanations align with the findings of the researcher [31], who reported that the inclusion of filler material enhances the composite material's resistance to plastic deformation.

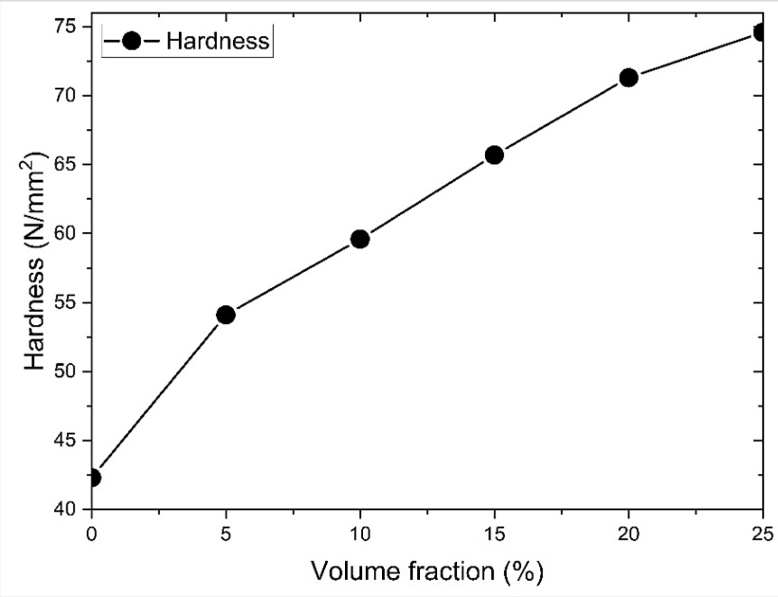

Figure 7: Hardness test results of specimens

4.5 Compressive test

The objective of the compressive test is to determine the maximum stress that polymeric materials and particle-reinforced samples can withstand when subjected to vertical pressure. This stress is computed by dividing the applied force by the sample's cross-sectional area. All samples were tested at room temperature. Error! Reference source not found. shows that the compressive strength at a volume fraction of 5% increased by 13.1 MPa compared to the pure sample. This increase in compressive strength is attributed to the presence of olive leaf particles, which disperse the load and transfer it from the base material to the particles via the interface. At a volume fraction of 10%, the compressive strength improved by an additional 8.9 MPa compared to the 5% volume fraction sample. Further increases were observed at volume fractions of 15%, 20%, and 25%, indicating a consistent enhancement in compressive strength with higher reinforcement ratios. Several physical mechanisms contribute to the observed improvement in compressive strength. The olive leaf particles act as stress raisers that help in transmitting the applied load from the polymer matrix to the harder reinforcing particles which in turn increases the overall compressive strength. Increased hardness of the olive leaf particles also increases the reinforcement within the composite by entering the polymer matrix and occupying the space, thus decreasing the polymer chain mobility thereby improving the material's ability to withstand compressive deformation.The use of strong interfacial adhesion between the olive leaf particles and polyester matrix enables efficient stress transfer, hence increasing the mechanical properties of the composite material. Moreover, due to the uniform distribution of the olive leaf particles in the matrix, they are fully effective in reinforcing the composite and increasing, in this case, the general compressive strength of the flow. These physical explanations align with the findings of the researcher [32], who reported that the inclusion of filler material enhances the composite material's resistance to compressive forces.

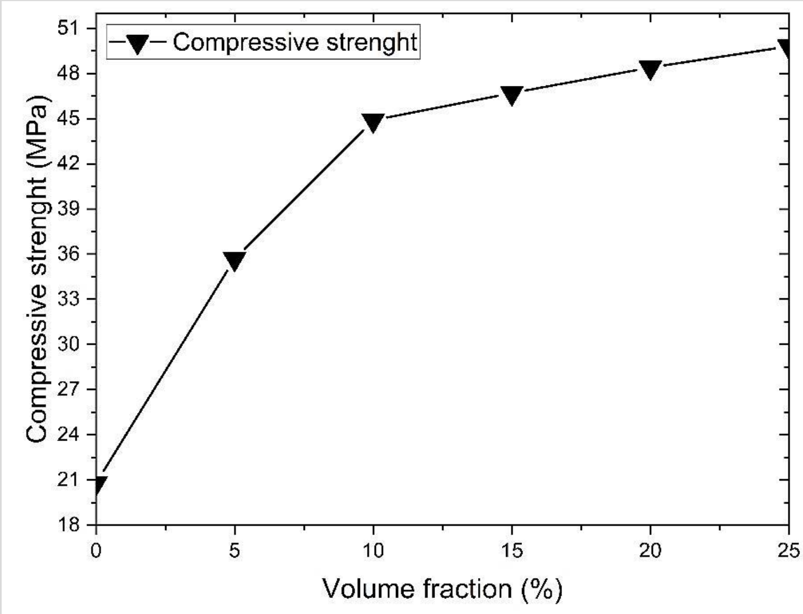

Figure 8: Compressive test results of specimens

4.6 Thermal conductivity results

Error! Reference source not found. represents the thermal conductivity of the unsaturated polyester reinforcement enriched with Olive Leaf Particles with volume fractions of 0%, 5%, 10%, 15%, 20% & 25%. From the data presented here it can be seen that the thermal conductivity of the UP reinforcement reduces with the increase in the volume fraction of OLP particles. For example, the thermal conductivity of the reinforcement is 0.321 W/mK at 0 vol.% of the olive leaf particles and reduced to 0.101 W/mK at 25 vol.% of the olive leaf particles. Olive leaf particles are shown to lower the thermal conductivity of unsaturated polyester composites. This can be attributed to two key factors: This is due to the following two factors: 1) inherently lower value of thermal conductivity of the particles as compared to the matrix [33], and 2) interruption of the continuous pathways for heat transfer in the matrix due to the presence of the particles, which causes the heat to transfer through the longer path [34]. This situation is further enhanced by the presence of interfacial thermal resistance between the particles and the matrix to transfer heat effectively [35].

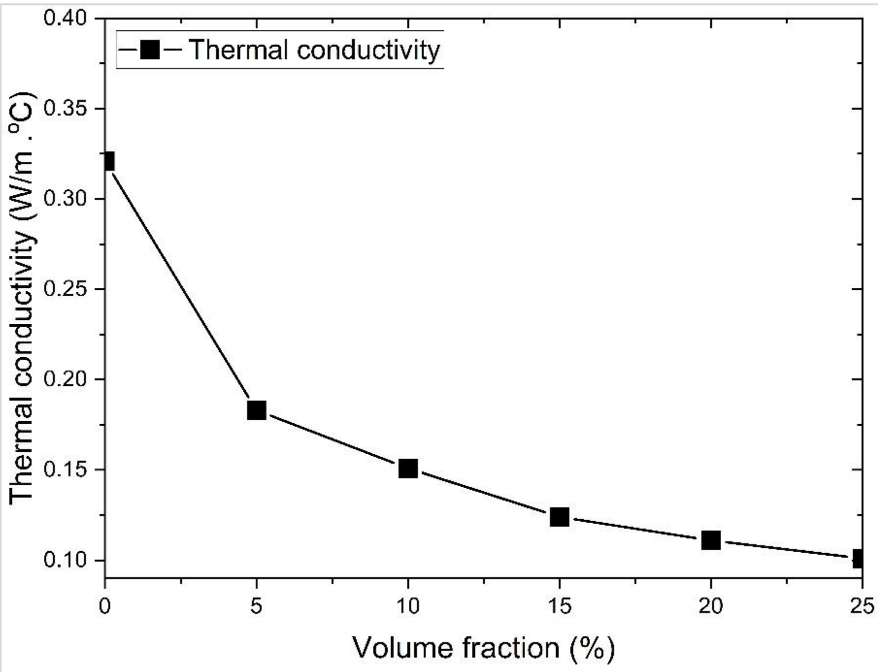

Figure 9: Thermal conductivity test results of specimens

5. Conclusion

The use of olive leaf powder as a filler in unsaturated polyester composites is a potential approach to developing mechanical and thermal performances. This research elucidates how the incorporation of different proportions of OLP (0%, 5%, 10%, 15%, 20%, and 25%) influences the mechanical, electrical, and thermal properties of the composites. The samples were prepared using hand lay molding, and the mechanical properties such as hardness, impact strength, and compressive strength were also tested. The examined results show consistently higher values of these properties as the amount of OLP incorporated increases.The result of the impact test shows the highest value of impact strength of 2.52 KJ/m², the maximum hardness of 72.4 N/mm², and the maximum compressive strength of 48.7 MPa. Thermal conductivity was reduced to 25% volumetric fraction and was found to be approximately at 0.101 W/m·°C. The peak intensities in the FTIR spectra were also changed, indicating the chemical adhesion between the OLP and the polyester matrix, and the SEM photomicrograph confirmed the effect of OLP in the matrix and strong interfacial adhesion.Altogether, this investigation elucidates the possibility of developing OLP-filled unsaturated polyester composites for multiple applications that provide better mechanical and thermal performance.

Author contributions

Conceptualization, E. Ibraheem; data curation, E. Ibraheem.; formal analysis, E. Ibraheem.; investigation, E. Ibraheem.; methodology, E. Ibraheem.; project administration, W. Bdaiwi, resources, E. Ibraheem.; supervision, W. Bdaiwi.; validation, E. Ibraheem..; visualization, E. Ibraheem.; writing—original draft preparation, E. Ibraheem.; writing—review and editing, E. Ibraheem and W. Bdaiwi. All authors have read and agreed to the published version of the manuscript.

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Data availability statement

The data that support the findings of this study are available on request from the corresponding author.

Conflicts of interest

The authors declare that there is no conflict of interest.

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