EVALUATION OF ECO-FRIENDLY UNSATURATED POLYESTER COMPOSITES REINFORCED WITH AGRO-WASTE FILLERS

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Received August 14, 2025

This research investigates the impact of incorporating agricultural waste materials, namely, date seed and walnut shell powder, into unsaturated polyester on its mechanical properties. Examination of the properties included tensile strength, flexural strength, impact resistance, and thermal conductivity. The date seeds and walnut shells were collected, crushed, and milled to produce powders with particle sizes of approximately 12.64 µm and 55.8 µm, respectively. The powders were mixed with unsaturated polyester at varying weight proportions (1%, 2%, and 3%); the samples were prepared by the hand lay-up method. The obtained results revealed that an increase in the content of powder enhanced the values of the characteristics studied. Specifically, the flexural strength of the composite with 2 wt% date seeds was 2.18 times higher compared to that of pure polyester. There was a variation in the tensile strength of the material with respect to the weight percentage of walnut shell powder, which was 1.5 times that of pure polyester. However, the hardness generally increased slightly and was likewise influenced by the percentage of filler, whereby the greatest improvement was achieved at 3% walnut shell loading. This study has shown that the reinforcement in composites can be sustainable, without requiring the use of expensive materials or complex processes.

Keywords: polyester, walnut shell, date seeds, composite materials, mechanical properties, biowaste

INTRODUCTION

Polymer materials are highly preferred in a wide range of applications due to their exceptional resistance, versatility in design, and low weight. As there is a tremendous number of polymers, whose structures vary depending on the building units and possible additives, the advantageous characteristics arise from their properties.[1,2] As polymers are indispensable in various applications and face limitations, polymeric composites are made to fill the gap and present new structures. In fact, these composites are creating a unique set of properties that cannot be achieved by the components individually.[3]

Currently, a worldwide effort driven by environmental awareness seeks environmentally friendly solutions across various aspects of life. Researchers are exploring locally available and cost-effective materials that can be applied in polymer reinforcement and evaluating their durability to meet the standards for well-reinforced polymer composites.[4] Efforts are increasing to exploit natural waste and fibers due to their abundance and ease of manufacturing.

Multiple factors contribute to global environmental issues, including the greenhouse effect, the use of toxic materials in industry, and the release of harmful waste (*e.g.*, microplastics) into the environment. Pollution resulting from emissions increases in tandem with rising energy consumption.[5,6] Recently, buildings have been accountable for more than one-third of worldwide greenhouse gas emissions in developed and developing countries.[7] Here, extending the age of plastics is crucial and is considered part of the solution.[8,9] Moreover, using eco-friendly materials based primarily on biomass represents a benign route,[10] as these materials have demonstrated their capabilities in many areas, such as water treatment, industrial applications, and polymer reinforcement.[11]

A promising and eco-friendly approach is the application of bio-waste to optimize material properties, hence contributing to solid waste reduction, while offering cost-effectiveness in production.[12] The application demonstrates the potential of plant waste as a resource that can help reinforce energy efficiency and material performance. Natural fibers and fillers are gaining preference during the last couple of decades in composite processing owing to their low cost, degradability, and ecological benefits. Various plant fibers, such as hemp, bamboo, walnut, coir, and sisal, are common in the preparation of polymer composites.[13,14] Walnut veneer possesses impressive mechanical properties, including very high strength and modulus. Walnut shell powder (WSP) has additional advantages: it is inexpensive, renewable, has low abrasion on machinery, has a low density with a high specific strength-to-weight ratio, and is an ecologically friendly reinforcement.[15]

Pradhan *et al.*[13] investigated the effect of the addition of WSP on the physical, mechanical, and thermal properties of polyester resin. It was noted that WSP addition improved microhardness and compressive strength; however, the addition caused a marginal reduction in tensile and flexural strength. Of importance, though, was the significant enhancement of thermal insulation, where 20 wt% WSP was found to reduce thermal conductivity by around 42%. A reduction in the coefficient of thermal expansion and an increase in the glass transition temperature further indicated increased thermal stability. Thus, the results suggested that WSP has limited mechanical benefits, but substantial thermal advantages at 20 wt% loading. With their lightweight, reduced thermal expansion, and associated superior insulation properties, such composites would be useful in various applications including insulation panels, food storage containers, thermos flasks, refrigeration equipment, building materials, and aircraft or automotive interiors.

Abu-Jdayil *et al.*[14] investigated the use of date seed waste in the manufacture of polyester-matrix composites with outstanding thermal insulation properties. FTIR testing indicated hydrogen bonding interactions between date seed powder and unsaturated polyester that favored the synthesis of stable composite materials. Replacement of the polyester with date seed powder up to 50 vol% produced composites that had good thermal insulation properties and were suitable for building applications owing to their low thermal conductivity. Mittal *et al.*[15] investigated date seed filler in biodegradable polyester matrices, where incorporation was found to enhance the mechanical properties as well as the biodegradation rates of the biopolymers. Similarly, Gharbi *et al.* [16] evaluated the effects of olive nut flour waste on the reinforcement of polyester by changing the weight fractions from 10% to 60%. They found there was significant enhancement of both the flexural strength and modulus.

Also, Alsaadi *et al.* [17] enhanced the performance of polymer composites by incorporating pistachio shells, which helped develop green composite materials. Sahari and Maleque[18] reported that good adhesion between the oil palm fillers and the polyester matrix enhances the distribution of stress, which increases the strength of the composite. In their study, though, this was achieved at the cost of water resistance when compared to pure polyester. Rosamah *et al.*[19] manufactured polyester hybrid composites by reinforcing oil palm shell nanoparticles in a mat made up of kenaf/coconut/kenaf fibers as fiber and particle reinforcements. The authors found that the composites exhibited better thermal, mechanical, and physical properties at 3 wt% loading of nano-oil palm shell. Increasing the oil palm shell content to higher values resulted in decreased mechanical properties.

Agricultural waste-based bio-filled polymers, such as polyester, contribute greatly to the development of sustainable material technology by the transformation of agricultural wastes into an environmentally friendly resource. The approach of utilizing agro-waste reduces waste production and leverages the rich carbon and nitrogen content of its composition.[20] In this work, walnut shell waste was combined with date pits in equal weight percentages (50 wt% each) to yield a hybrid powder. This hybrid powder was used in the development of composite materials, showing an efficient methodology for recycling agricultural waste into value-added and environmentally benign products.

EXPERIMENTAL

Materials

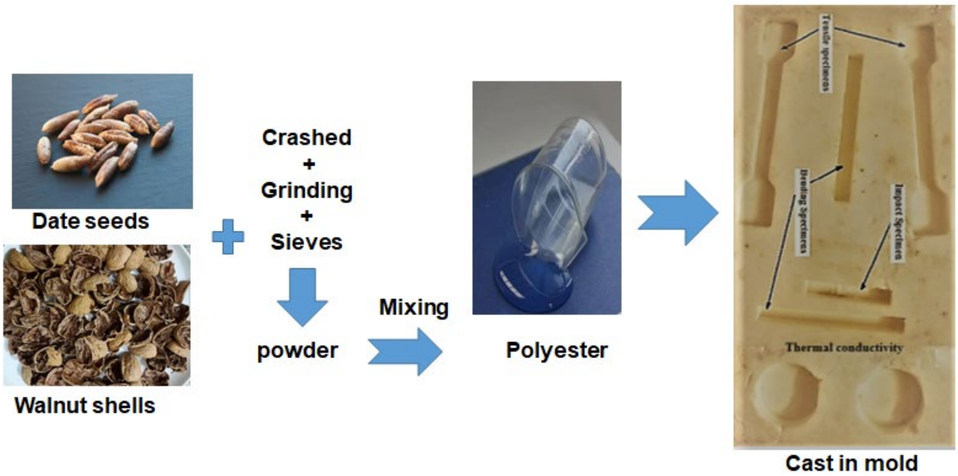
For the development of the composites, unsaturated polyester (ES-1060, esKiM casting type) and a hardener from esKiM Chemical Company, Turkey, were utilized, as detailed in Table 1. The hardener was used to replace 3 wt% of the polyester weight in the production of all specimens. Walnut shells and date seeds, sourced from Baghdad and Kurdistan local markets, Iraq, were cleaned and ground into powder using a jaw crusher.

Composite preparation

This study employed three types of fillers: walnut shell, date seed, and hybrid powders. A hybrid powder was prepared by combining equal proportions (50% each) of walnut shell and date seed powders. The particles were gathered, pulverized using a jaw crusher and an electric mill, and subsequently sorted using a vibratory sieve shaker to attain the desired size. The powders obtained had grain sizes of 12.64 µm for date seeds and 55.8 µm for walnut shells.

Table 1
Characteristics of polyester

Product description	Casting type polyester
Chemical structure	Orthophthalic
Density 20 °C g/cm^3	1.16 ± 0.02
Viscosity (cPs)	450 ± 50
Monomer content (%)	34 ± 3
Gel time (min)	5 ± 1
T max (°C)	150 ± 10
Tensile strength (MPa)	65 ± 6
E-modulus, tensile (GPa)	3 ± 0.3
Elongation at break / tensile (%)	7 ± 0.7
Flexural strength (MPa)	120 ± 10
E-modulus (GPa) flexural	2.7 ± 0.3



Scheme 1: Stages of polyester composites preparation

The particles were filled within the polyester at different concentrations, namely, 1%, 2%, and 3%, and compared to the plain one. The composite samples were prepared by mixing unsaturated polyester with varying amounts of the hybrid powder. The powders were introduced into the polyester and hardener and thoroughly blended at ambient temperature. Subsequently, the concoction was poured into molds in accordance with the specified testing criteria and allowed to undergo a 48 h curing process. The preparation steps of the modified composites are illustrated in Scheme 1.

Characterization

FTIR spectroscopy

Fourier-transform infrared (FTIR) spectroscopy was carried out using a Bruker Tensor 27 (Germany) to examine the functional groups added to the polymer by embedding the additives. The spectra were recorded in the range of 600-4000 cm^{-1}.

Thermal conductivity test

The thermal conductivity of the polyester composites with reinforcing particles was measured using Lee's disc method in accordance with ASTM E-

1530.[21] The specimens used in this test were cylindrical, with a thickness of 5 mm and a diameter of 40 mm.

Impact and hardness test (Shore D)

The composites' impact strength was assessed using a low-velocity Izod impact-testing machine. For this measurement, samples measuring 64 mm × 12.7 mm × 3.2 mm were utilized; this procedure adhered to the ISO 180 [22] test standards. The nominal energy of the pendulum hammer was 5.5 joules, and the samples were used without notching.

The Shore D hardness test was performed at room temperature, following the ASTM D-2240 protocol.[23] The specimens used in this test had a diameter of 40 mm and a thickness of 5 mm. Three measurements were taken for each specimen, and the average hardness value was calculated; the force applied in Shore D hardness tests was 5 Kg.

Tensile and flexural strength test

The composite specimens were tested for tensile and flexural properties using an Instron 1195 Universal Testing Machine. For the tensile tests, specimens were prepared according to ASTM D 638,[24] with dimensions of 150 mm × 10 mm × 3 mm, and the crosshead speed was set to 5 mm/min. All tests were performed on three samples for each test, and the results represent the average data.

For the flexural tests, the specimens were cut to dimensions of 105 mm × 15 mm × 3 mm according to ASTM D 790 25. The flexural test (or bending test) characterizes the behavior of a structural element under a load applied perpendicularly to its axis. Flexural strength refers to the strength at which the beam section fractures and is measured using the "Three-Point Test" method. The test was carried out at room temperature with a Laryee Model 1031 machine manufactured in China, in which the load applied varied from 0 to 45 kN. The data from the three-point bending test were acquired with the same tensile machine at a crosshead speed (strain rate) of 5 mm/min, in which the load was applied until the specimen was fractured. The flexural strength can be calculated by the following equation:

$$F.S. = 3\,FL/\,2bd^2 \qquad (1)$$

where F.S.: flexural strength (MPa), F: force at fracture (N), L: length of the support span (mm), b: width of the sample (mm), and d: thickness of the sample (mm).

RESULTS AND DISCUSSION

The mechanical properties of the samples, including bending strength, resistance to impact, hardness, and tensile strength, were analyzed as a function of the filler weight percentage variation. These properties show how the addition of date seed and walnut shell powders may affect the polyester composites. Data from each test were obtained from the average results of three standard specimens.

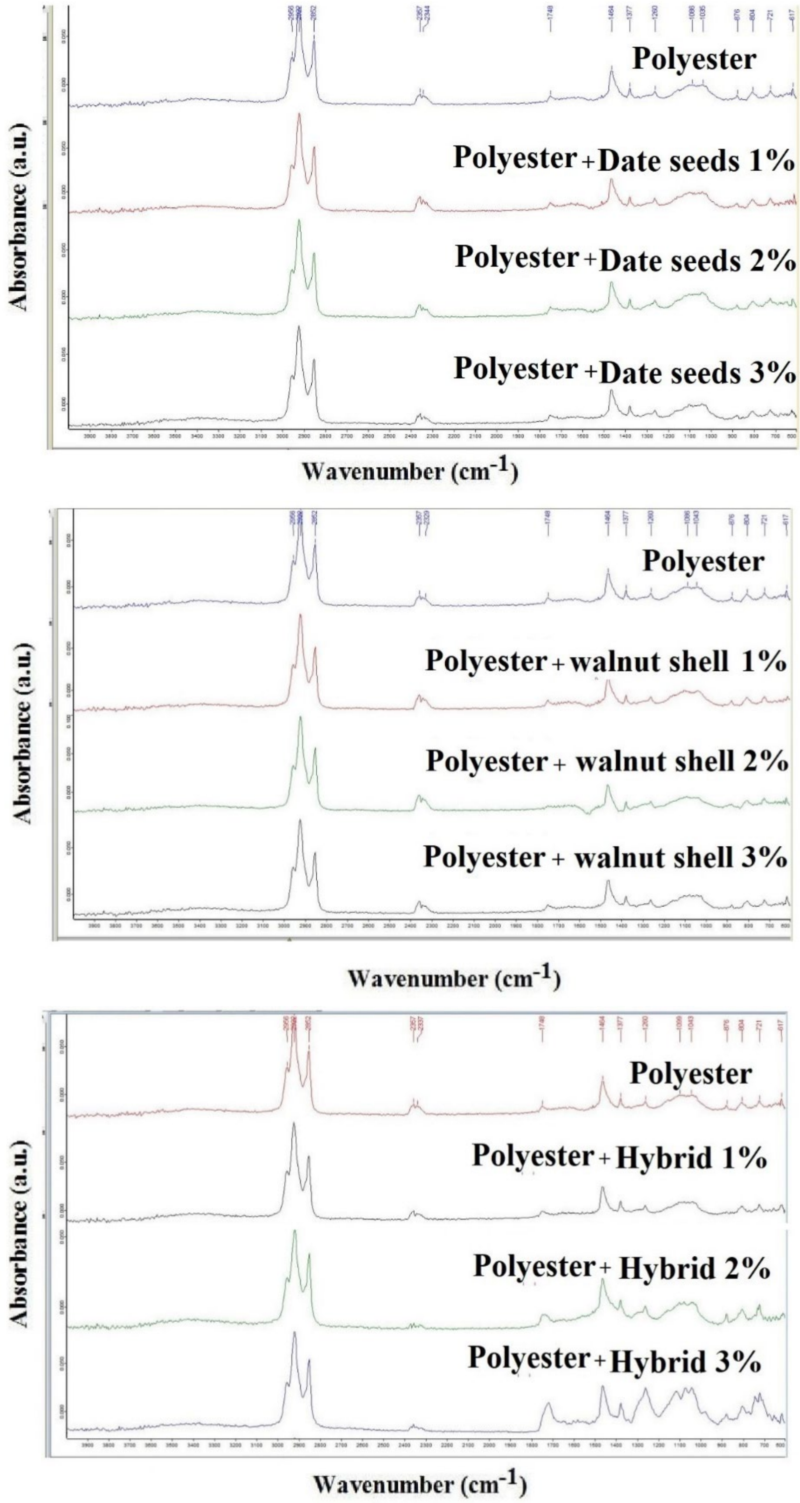
FTIR examination

The samples were examined via FTIR; the spectra recorded for the polyester reinforced with the date seed powder, the polyester reinforced with walnut shell powder, and that incorporating a mixture of both additives are illustrated in Figure 1. The spectra show no major difference, which could be attributed to the small amounts of the additives. Yet, mixing 3% of the hybrid mixture indicated the presence of small new peaks, which could be discussed.

In the spectra corresponding to the date seeds, the peak around 1750 cm^{-1} stands for C=O carbonyl stretching vibrations characteristic of esters, aldehydes, ketones, and carboxyl groups. A band around 1670 cm^{-1} is attributed to C-H stretching, indicating the presence of an aromatic ring. The small bands at 1500-1700 cm^{-1} are associated with the O-H bond in carboxylic acids. The peak at 1050 cm^{-1} corresponds to C-O stretching. The bands detected at 1000 and 780 cm^{-1} are indicative of esters such as CH_3CO-O- and cyclic C-O-C groups that are conjugated with C=C-O-C double bonds in olefinic or aromatic frameworks, signifying the principal constituents of lignocellulosic materials. Moreover, the spectral bands around 750 cm^{-1} and 600 cm^{-1} correspond to aromatic (C-H) substitution vibrations.[26]

In the spectra corresponding to the walnut shell, the band at 1745 cm^{-1} confirms the C=O stretching vibrations in the acetyl group of hemicelluloses. Supplementary hemicellulose bands, coinciding with those of cellulose, are barely present around 1375 cm^{-1}. Cellulose could be characterized by the 1500 and 780 cm^{-1} bands.[27] Because cellulose (crystalline, phonon-conducting) raises λ while lignin (amorphous, phonon-scattering) lowers it, the cellulose:lignin ratio must be controlled to tune the filler's thermal conductivity to the application's insulation or heat-dissipation requirement.

Implementing ATR correction restores the authentic intensities of spectral peaks, baseline correction eradicates background slopes and scattering effects, and mild smoothing aids in mitigating noise. Collectively, these preprocessing measures facilitate accurate peak localization, enable precise quantification, and enable reliable spectral analysis of polyester composites. The ester carbonyl peak around 1720 cm^{-1} broadens and shifts to lower wavenumbers, while a C–O stretching band near 1240 cm^{-1} appears, signifying hydrogen bonding and chemical coupling between polyester carbonyls and the hydroxyl groups of the lignocellulosic filler. The spectrum is predominantly characterized by the intense polyester ester C=O (~1720 cm^{-1}) and C–O–C (~1260 cm^{-1}) peaks, which obscure the relatively feeble bands of cellulose and lignin.[26-28]



(a)

(b)

(c)

Figure 1: FTIR spectra of polyester reinforced with (a) date seed powder, (b) walnut shell powder, and (c) a mixture of both additives

Thermal conductivity and density

Figure 2 presents the density and thermal conductivity of composites with different contents of walnut shell, date seed, and hybrid filler particles. The general result indicates a significant reduction in thermal conductivity when increasing the amount of particles, compared to the pure polyester. This could be because of the lower thermal conductivity of the walnut shell, date seed, and hybrid filler particles themselves compared to the polyester resin; an idea which was also reported in previous studies.[28]

The addition of particles also affects density, but the general trend is a slight reduction from that of the pure polyester. Worth noting, the maximum density was found with a 1% addition of walnut shell powder, as also reported in the literature.[29] Moreover, the addition of walnut shells, date seeds, and hybrid fillers introduces more porosity and air voids in the polymer, which contributed to the lower densities obtained.

The incorporation of WSP into the polyester matrix reduced thermal conductivity, enhanced thermal stability, and lowered thermal expansion. Such characteristics make these composites useful in various fields, including thermal insulation boards, food packaging, refrigeration, and even construction.[30] Despite limited mechanical properties, the overall performance suggests that the WSP-polyester composite has great potential to serve as an eco-friendly thermal insulation material.[26] Thus, lignocellulosic cell walls and inner pores serve as a natural insulator, and disturbing the path for heat transfer.

It was also reported in another study that an epoxy based composite made with Indian almond fiber and fruit-shell particles had a Shore D hardness of 89 and a density of 1.18 g cm^{-3}. This agrees well with our findings, considering that the addition of 3% walnut shell yielded a Shore D hardness of 80, whereas incorporating 3% date seed resulted in a slightly higher density of 1.20 g cm^{-3}.

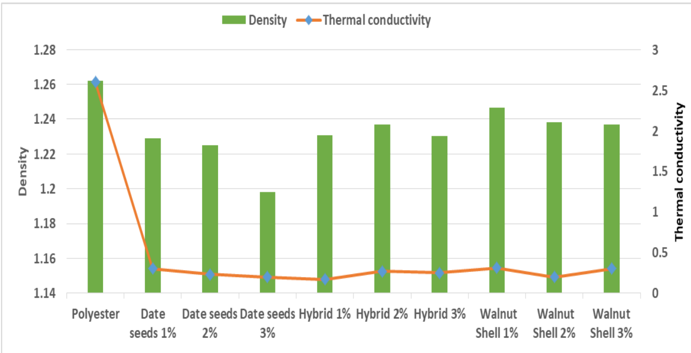


Figure 2: Comparison of density and thermal conductivity in polyester based composites with additive particles

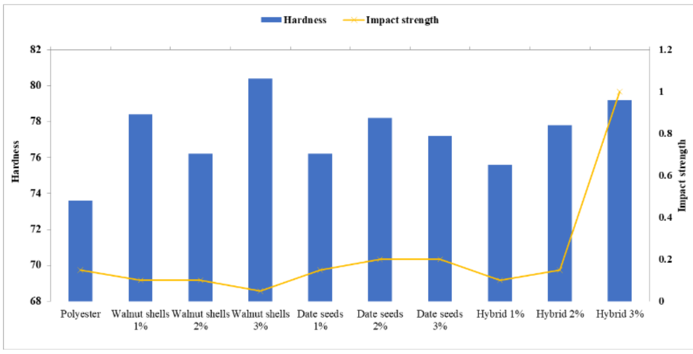


Figure 3: Comparison of impact strength and hardness in polyester based composites with reinforcing particles

Impact and hardness test (Shore D)

Figure 3 presents a comparison of hardness and impact resistance in polyester composites containing added particles. The addition of particles in any ratio improves the hardness value in comparison to that of pure polyester. This is explained by the structure of the added particles, which resists permanent plastic deformation, improving the material's resistance to plastic deformation. The shell particles occupy voids within the polymer matrix, thereby densifying and stiffening the structure. Hardness testing is relatively simple and offers useful information on the interactions between the microstructures of polymer composites.[26]

However, the impact strength decreases with increasing percentages of walnut shell, date seed, and hybrid particles. This reduction is because these particles generally have lower impact resistance than the composite materials and lower impact resistance than the unsaturated polyester matrix. The above observation agrees with the results obtained by Nasif *et al.*[31]

Effect of filler content on tensile and flexural strength properties

Figure 4 presents the effects of different fillers and loadings on the tensile and flexural strength of polyester. Results show a considerable increase in tensile strength for particle-loaded samples compared to the pure polyester samples. The highest tensile strength was achieved by the addition of 1% walnut shell powder. This improvement is due to the excellent interfacial interaction between the polyester matrix and WSP, because of the high lignin content in the powder and a decrease in the crystallinity of the composite. Powder particles improve interfacial bonding between resin and filler, enhancing mechanical properties as a whole. In addition, they hinder the growth and development of inner cracks during fracture and improve the resistance of the composite against outer forces. This is in agreement with studies reported earlier.[26] The fracture surfaces were also relatively flat without necking, indicating a brittle fracture mechanism during testing. Moreover, no remarkable long-time reduction in the cross-sectional area was recorded and the tensile strength was measured at its maximum value. The addition of 3% date seed particles enhanced the reinforcement effect of natural fillers in the polyester matrix, resulting in improved interfacial bonding between the particles and the resin. Analysis revealed that the presence of specific functional groups (–OH, CH_2, CH_3, Si–O–Si, C=C, and Si=H) increases the strength of interlinking between these particles and the matrix. The presence of active groups in date seed particles confirms the findings of previous studies.[26]

The flexural strength decreased from 38 MPa for pure polyester to 25 MPa for 2 wt% WSP, and this was attributed to poor stress transmission and agglomeration of particles at high filler loading. Particles tend to be agglomerated rather than uniformly dispersed; thus, leading to voids and defects that badly deteriorate the composite's integrity. The addition of 2 wt% of date seed particles improved the flexural strength from 38 MPa to 83 MPa, which exhibited a more pronounced reinforcing effect within the polyester matrix. Date seeds contain a high amount of fibers that include cellulose and lignin. These, when combined in a polymer matrix, enhanced stress tolerance and were in agreement with previous literature.[26] In our work, a tensile strength of 63 MPa was found in 1% walnut shell reinforced composite, and a flexural strength of 83 MPa was found in the 2% date seed composite.

The use of processed agricultural waste in the production of lightweight and eco-friendly composites has been reported in many studies, such as the one conducted by Babu *et al.*[27] They achieved a tensile strength of 72 MPa, while the flexural strength achieved was 118 MPa. Another study[32] used Tulsi (holy basil) fibers as reinforcement and mango seed particles as filler within an epoxy resin matrix. The tensile strength achieved was 60 MPa and flexural strength was 90 MPa. Naik *et al.*[33] prepared a mango seed shell short fiber reinforced/epoxy composite, and obtained a flexural strength of 75 MPa. Finally, Kumar *et al.*[34] also studied mango seed shell/epoxy composites, and obtained tensile and flexural strengths of 58 MPa and 108 MPa, respectively.

Stress and strain relationship

The relationship between stress and strain is important because it helps understanding how materials respond to forces, providing insight into their mechanical properties. It is important in the development of reliable structures, optimization of material's efficiency, and avoidance of failure; hence, it is at the heart of innovation and practical engineering solutions. Figure 5 shows a significant improvement in stress resistance compared to that of pure polyester for all the additive percentages and types. However, this is at the cost of ductility, as in the case of the composite with the walnut shell content of 1% and the highest stress resistance. This is because the lignocellulosic particles behave like stress concentrators, which weaken the polyester matrix. The addition of rigid filler particles may restrict the movement of the chains of the polymer, hence making the composite more brittle and less ductile. Thus, it reduces its elongation at break; that is, it is more prone to fracture without any remarkable deformation. Shell particles have been also found to fill the micro-voids, reduce capillary water ingress, and slow microbial attack.[27]

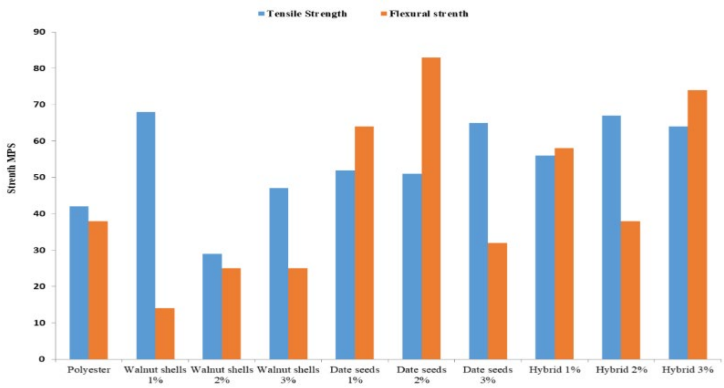


Figure 4: Comparison of tensile strength and flexural strength with polyester reinforcing particles

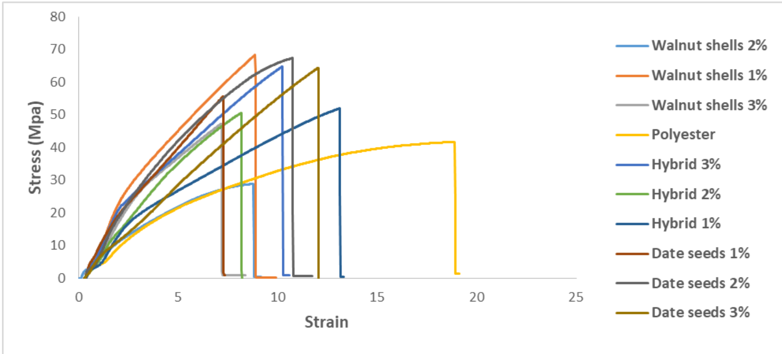


Figure 5: Stress-strain relationship of the studied composites

CONCLUSION

Composites were fabricated, using walnut shell and date seed powder, with particle sizes of 55.8 µm and 12.64 µm, respectively, and their mechanical properties were tested. The tests revealed that the addition of the fillers decreased the density of the materials and increased the mechanical properties up to a certain loading. Increasing the filler concentration beyond that threshold caused a reduction in the strength of the composites due to particle agglomeration and loss of interfacial bonding.

Interestingly, higher walnut shell content resulted in lower flexural strength. The maximum flexural strength of the neat polyester was significantly higher compared to that achieved for the walnut shell composites. However, the results showed that the flexural strength of the composite with 2 wt% date seeds was 2.18 times that of pure polyester. The tensile strength of the walnut shell composites decreased for higher filler loadings, while hardness generally increased slightly. The maximum improvement in hardness was observed at a 3% filler loading of walnut shell.

ACKNOWLEDGMENT: The authors thank the Department of Materials Engineering at the University of Technology, Iraq, for partially supporting this work.

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