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A Sustainable Bio-Filler for Epoxy Composites: Use of Pistachio Shell Powder

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ABSTRACT

The use of natural materials has become a crucial concern for humanity due to the limited quantity of petroleum-based products and their harmful impact on nature. The use of natural materials is inevitable for a greener and more sustainable world. Transforming agricultural industrial waste into a high-value product or a source of fuel can significantly benefit our country's economy and environment. Pistachio shell is one of the agro-industrial wastes and is abundant in our country. Our country can benefit economically and environmentally by utilizing and repurposing discarded shells, whether as fuel or as a valuable commodity. In this study, the shells of pistachios grown in Şanlıurfa were crushed in a stone mortar, and reduced to certain sizes, and turned into powder. Later, this powder was used as a bio-filler material for epoxy resin. Powders were used at 1 % and 2 % additive ratios and homogeneous mixing was carried out using both magnetic and ultrasonic mixers. The mechanical properties of the obtained bio-filled epoxies were investigated and their microstructures were analyzed. Tensile tests, bending tests and hardness tests were performed as mechanical tests, and densities were measured. An improvement of 8 % and 15 % was detected in the maximum tensile and bending strength, respectively. The hardness and density values also increased by 8% and 1.7%, respectively. As a result of the microstructure analysis, no air bubbles were formed in the samples, and no agglomeration occurred. Obtained results revealed that pistachio shell grown in Şanlıurfa can be used as bio-filler.

Epoksi Kompozitler İçin Sürdürülebilir Bir Biyo-Dolgu: Antep Fıstığı Kabuğu Tozu Kullanımı

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ÖZ

Petrol tabanlı ürünlerin doğaya zarar vermeleri ve miktarlarının sınırlı olmaları, insanoğlu açısından doğal malzemelerin kullanımını oldukça önemli bir noktaya taşımaktadır. Doğal malzemelerin kullanımı daha yeşil ve sürdürülebilir bir dünya için kaçınılmazdır. Yakıt olarak kullanılan veya çöpe atılan tarımsal endüstriyel atıkların katma değeri yüksek ürün olarak ekonomiye kazandırılması ülkemiz açısından önemlidir. Antep fıstığı kabuğu tarımsal-endüstriyel atıklardan biridir ve ülkemizde bolca bulunur. Her yıl ya yakıt olarak kullanılan ya da çöpe atılan bu kabukların değerlendirilip ekonomiye kazandırılması ülkemiz açısından önemlidir. Bu çalışmada Şanlıurfa ilinde yetişen Antep fıstıklarının kabukları taş havanda dövülerek belli boyutlara indirilmiş ve toz haline getirilmiştir. Daha sonra bu toz epoksi reçinesi için biyo-dolgu malzemesi olarak kullanılmıştır. Tozlar %1 ve %2 katkı oranlarında kullanılmış ve hem manyetik hem de ultrasonik karıştırıcı kullanılarak homojen karıştırma gerçekleştirilmiştir. Elde edilen biyo-dolgu katkılı epoksilerin mekanik özellikleri incelenmiş ve mikroyapıları analiz edilmiştir. Mekanik testler olarak çekme testi, eğilme testi ve sertlik testi yapılmış ve yoğunluklar ölçülmüştür. Maksimum çekme ve eğilme gerilmelerinde sırası ile %8 ve %15'lik bir iyileşme tespit edilmiştir. Sertlik ve yoğunluk değerleri de sırası ile %8 ve %1,7 oranlarında artmıştır. Mikroyapı analizi sonucunda ise numunelerde herhangi bir hava kabarcığının oluşmadığı ve topaklanma meydana gelmediği tespit edilmiştir. Elde edilen sonuçlar Şanlıurfa şartlarında yetişen Antep fıstığı kabuğunun biyo-dolgu malzemesi olarak kullanılabilir olduğunu ortaya koymuştur.

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1. Introduction

Due to their excellent technical properties, epoxy resins have found extensive use across various industries (Demircan et al., 2020a; Neves et al., 2021; Wazalwar et al., 2021). Epoxy resins, which are especially used as matrix materials in fiber-reinforced composite materials, are materials with high mechanical strength, excellent chemical and electrical resistance, high-level thermal insulation, and high-quality adhesion (Earp et al., 2019; Özen et al., 2019; Ozen et al., 2022). Due to these properties, materials produced with epoxy resin are widely used in aerospace technologies, the defense industry, the automotive industry, marine vehicles, construction, and the energy industries (Ma et al., 2020; Margoy et al., 2021; Demircan et al., 2023a). Polymer materials, including epoxy resins, exhibit superior properties in industrial applications, but they also have various disadvantages (Prolongo et al., 2014). One of their most significant drawbacks is their resistance to natural degradation. Polymers that persist in the natural environment for an extended period of time pose a threat to living organisms. Additionally, their recycling rates are low, and various chemical reactions are required to obtain them. These negative aspects have prompted researchers to develop environmentally friendly, usable, sustainable, and functional materials (Matykiewicz et al., 2019; Demircan et al., 2020b).

Due to the harmful effects of petroleum-based materials on the environment and their decreasing availability, researchers are focusing on natural materials. Natural materials are used as additive materials in various polymer resins due to their environmental friendliness, low cost, and easy availability (Puglia et al., 2005; Chang et al., 2021; Zaghloul et al., 2021). Researchers emphasize the use of easily degradable materials in nature to reduce the harm caused by petroleum-based polymers (Acikgoz et al., 2021). In particular, food waste such as pistachio shells, peanut shells, eggshells, walnut shells, and olive pits is annually discarded. These are referred to as agricultural-industrial wastes and are suitable for developing low-cost materials obtained from renewable sources (Prabhu et al., 2020). The use of natural additives obtained from agricultural-industrial wastes in polymer matrices is crucial to benefit from non-industrial materials and obtain composite polymers with specific mechanical and thermal properties (Dalbehera and Acharya, 2016; Erdoğan et al., 2019).

Antep pistachio shell is a natural additive material that is abundant in our country and classified as agricultural-industrial waste. These shells are obtained from the Antep pistachio. Antep pistachio is grown in 56 provinces in our country, and the largest producer is Şanlıurfa province (Alsaadi et al., 2018). Turkey has approximately a 15 % share of world pistachio production (Satil et al., 2003). Therefore, it is crucial to evaluate and utilize Antep pistachio shells in order to contribute to the economy. Pistachio shell is quite hard, and its chemical composition (%42 cellulose, %13.5 lignin, %3.11 cellulose lignin, % 1.26 ash, and % 0.18 extract) makes it a suitable material for various purposes (Al-Obaidi et al., 2020; Rizal et al., 2020). Additionally, it has high strength, hardness, and elastic modulus properties. Pistachio shells have a characteristic color range from beige to white. They are very light as a powder and, in some cases, even resemble natural marble granules. This unique color makes pistachio shell granules a versatile bio-based component.

Pistachio shell powders are commonly used as a bio-filler in the production of wood and plastic composites. Due to their lightweight, they increase the density/strength ratio of composites. Thiagarajan et al. (2021) used pistachio shell powder as an additive material (at weight percentages of 1 %, 2 %, and 3 %) to produce chopped glass fiber-reinforced epoxy composites. They used the vacuum-assisted resin infusion method for composite production and examined their mechanical properties. As a result, they found that the flexural strength of the composite material increased by 14 % with a 1 % addition. Alsaadi et al. (2018) added pistachio shell powders, which were reduced to micron sizes, to polyester resin and determined their tensile, flexural, and charpy impact properties. In addition, it is possible to evaluate pistachio shells by subjecting them to different processes. Çetin et al. (2022) produced flexible electrically conductive polymer composites by carbonizing shell residues and found that as the addition rate increased, the conductivity values increased. Salazar-Cruz et al. (2022) chemically modified shell particles in NaOH solutions and determined their thermal properties. They observed that as the shell amount increased, the thermal properties of the polypropylene composite improved.

In this study, the shells of pistachios grown in Şanlıurfa province were pulverized into powder form, and these powders were used as a bio-filler material for epoxy resin. The mechanical properties of the produced bio-filler additive epoxy composites were examined, and the results were evaluated. The main objective of this study is to enhance the economic value of pistachio shells, which are widely available in our country, by utilizing them as a high-value-added product.

2. Materials and Method

High-quality pistachios grown in Şanlıurfa province were selected for the production of pistachio shell powders. The mechanical properties of the shells were determined in the materials laboratory of Harran University and presented in Table 1.

Table 1. Mechanical properties of pistachio shell

Technical Specifications of Pistachio Shell Powder	
Density	1.311 gr/cm^3
Maximum Particle Size	75 µm
Hardness Value	31.9 HV

Commercially available F1564 epoxy resin and F3487 hardener were used as matrix materials. The technical properties of this resin are given in Table 2.

Table 2. Technical characteristics of the resin

	F1564 Resin	**F3487 Hardener**
Density	1.1-1.2 g/cm^3	0.94-0.95 g/cm^3
Appearance	Clear Colorless	Clear Colorless, Slight Yellow
Viscosity	1250-1450 mPa.s	10-20 mPa.s

To eliminate the moisture content, pistachio shells were dried in an oven at 80°C for 5 hours. Subsequently, the dried shells were crushed in a stone mortar to reduce their size. After grinding the shells thoroughly in the stone mortar, a ball mill was used to further reduce them to smaller sizes. The shells were then sieved using 0.25 mm, 0.15 mm, and 75 µm mesh sizes, successively, to achieve a maximum particle size of 75 µm. The process of converting the shells into powder is illustrated in Figure 1.

Figure 1. Powdering of pistachio shells

The reduced shell powders were weighed in mass ratios of 1% and 2% according to the epoxy resin. The resin/hardener ratio of 100/33 (gr) provided by the resin supplier was used. In order to achieve homogeneity in the resin-shell powder mixture, the simultaneous mixing method was preferred. This method involves immersing an ultrasonic mixing probe from the top and mixing magnetically from the bottom (Demircan et al., 2021). In this study, the mixture was stirred for 1 h with this method. To prevent excessive temperature rise during mixing, the ultrasonic mixer was used at 40% amplitude and the magnetic mixer at 300 rpm stirring speed. After mixing, the mixture was vacuumed in a vacuum chamber to remove air bubbles. The mixing method and vacuum chamber are shown in Figure 2. The mixture was then poured into wooden molds and left to cure at room temperature for 2 days. The cured plate was post-cured at 80 °C for mechanical stability and then cut into desired dimensions with a CNC router to prepare tensile, bending, and hardness samples for testing. Test samples are shown in Figure 3.

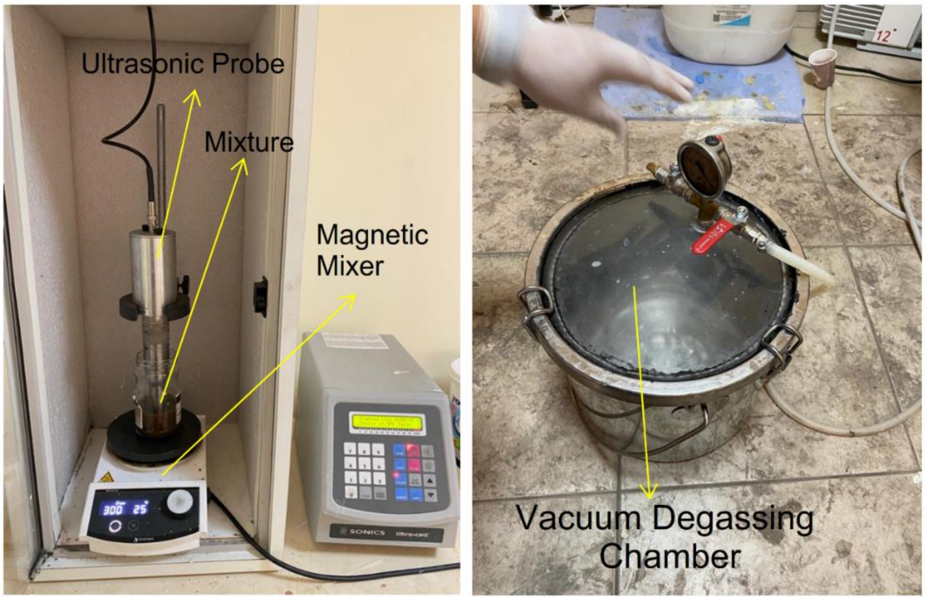

Figure 2. Mixing method and vacuum degassing chamber

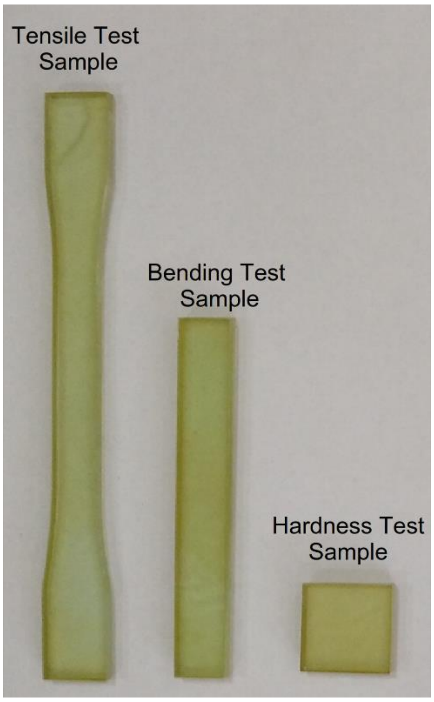

Figure 3. Test samples

3. Results and Discussions

3.1. Tensile Test

The tensile properties of a material are based on its resistance in the direction of applied axial forces. Determination of the tensile properties is crucial to providing information on elasticity modulus, tensile strength, strain, and other stress characteristics.

In this study, tensile tests were conducted on a Shimadzu AGS-X Plus tensile machine according to the ASTM D638-14 standard. Tests were carried out at a speed of 5 mm/min for each parameter with five samples, and the average values were used for analysis. The stress-strain curve obtained from the tensile test is shown in Figure 4. The maximum strength, strain, and modulus of elasticity values are presented in Table 3. The results indicate that the increase in the amount of shell powder increases the maximum strength value and modulus of elasticity while decreasing the strain value. This shows that the shell powder improves the rigidity of the epoxy. It is known from the literature that an increase in rigidity results in an increase in toughness and mechanical properties (Sun et al., 2019).

The modulus of elasticity of the pure resin was found to be 2.36 GPa, while the 1% pistachio shell-added resin showed an increase in the modulus of elasticity to 2.56 GPa. The highest modulus of elasticity was obtained with 2 % pistachio shell-added resin, which was found to be 2.72 GPa. These results indicate that the addition of pistachio shell to epoxy resin improves the mechanical properties of the resulting biofilled resin. The findings of this study suggest that pistachio shell could be a promising filler material for enhancing the mechanical properties of epoxy resin.

The increase in modulus of elasticity with the addition of pistachio shell to the epoxy resin can be attributed to the rigid and strong structure of the pistachio shell. The shell contains cellulose, hemicellulose, and lignin, which are all tough and fibrous materials that can enhance the mechanical properties of the resulting biofilled resin. When the pistachio shell particles are incorporated into the epoxy matrix, they act as reinforcing agents, which can improve the rigidity, strength, and toughness of the material. The interlocking of pistachio shell particles within the epoxy matrix increases the contact area between the two materials, resulting in better stress transfer and improved load-bearing capacity.

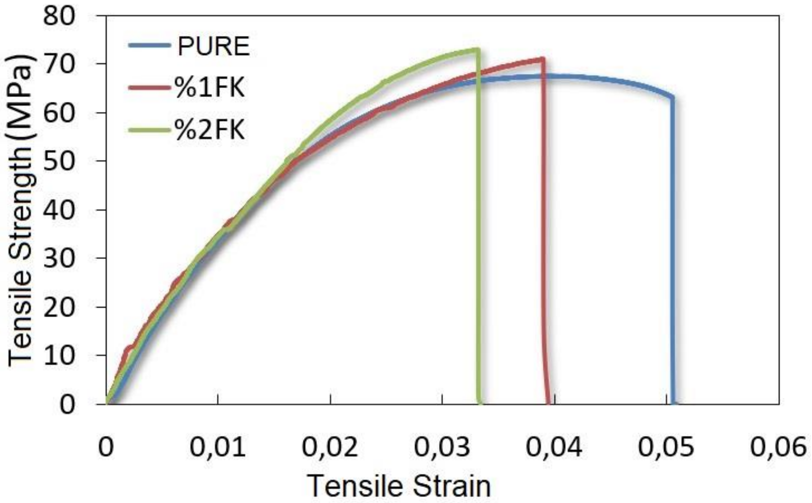

Figure 4. Tensile test strength-strain curve

Table 3. Tensile test results

	Maximum Strength (MPa)	Strain (-)	Modulus of Elasticity (GPa)
Pure	67.51	0.05085	2.36
% 1FK	71.01	0.03943	2.56
% 2FK	73.02	0.03342	2.72

3.2. Three Point Bending Test

Due to the ease of specimen preparation and simplicity of test configurations, the three-point bending test is an important method for determining bending properties. This test involves placing a rectangular cross-sectional test specimen on two supports in a straight position and subjecting the specimen to damage by a loading head located in the middle of the supports. Typically, a support width-depth ratio of 16:1 is used (Demircan et al., 2023b). The bending stress value obtained from the test can be calculated using Equation 1.

$$\sigma_f = \frac{3PL}{2bd^2} \tag{1}$$

P (N) represents the maximum load applied to the center of the specimen, L (mm) represents the distance between the lower supports, b (mm) represents the width, and d (mm) represents the thickness of the specimen.

The unit strain that occurs during bending is expressed as the nominal fractional change in length of an element on the outer surface of the test specimen, located at the midpoint of maximum stress. This value can be calculated using Equation 2.

$$\varepsilon_f = \frac{6Dd}{L^2} \tag{2}$$

D (mm) represents the maximum deflection at the center of the specimen, L (mm) represents the distance between the lower supports, and d (mm) represents the thickness of the specimen.

The bending modulus can be obtained using Equation 3.

$$E_f = \frac{L^3 m}{4bd^3} \tag{3}$$

In addition to the formulas mentioned above, the value m (N/mm) represents the slope of the tangent at the initial linear portion of the load-deflection curve. The principle of the three-point bending test is shown in Figure 5.

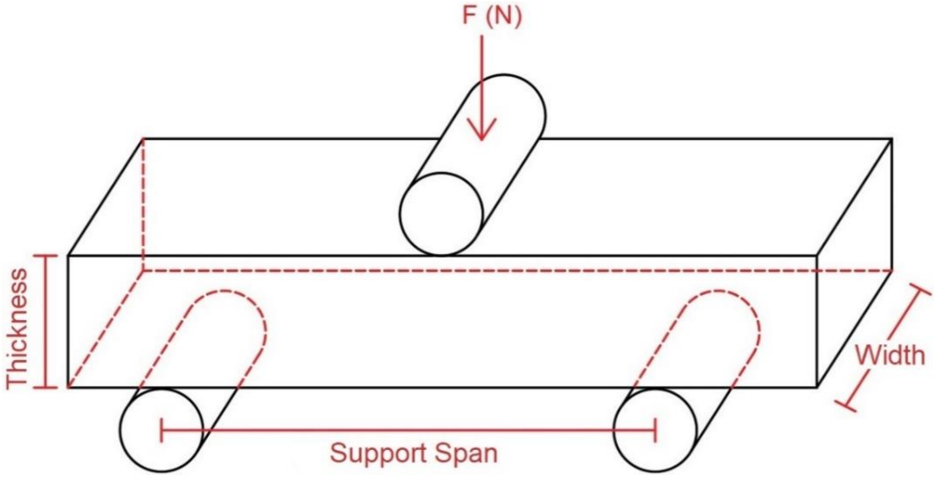

Figure 5. Three-point bending test principle

In this study, bending tests were conducted on a Shimadzu AGS-X Plus universal testing machine according to the ASTM D790-17 standard. Tests were performed at a speed of 2 mm/min for each parameter with five samples, and the average values were used for data analysis. The stress-strain curve obtained from the bending test is presented in Figure 6. The maximum stress, unit strain, and modulus of elasticity are provided in Table 4.

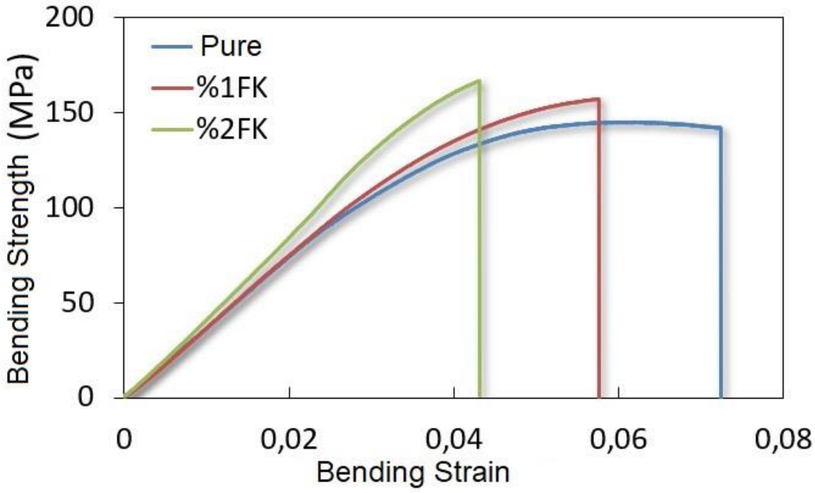

Figure 6. Three-point bending test stress-strain curve

Table 4. Three-point bending test results

	Maximum Strength (MPa)	Strain (-)	Modulus of Elasticity (GPa)
Pure	145.18	0.07244	3.46
% 1FK	157.01	0.05768	3.92
% 2FK	167.04	0.04313	4.32

The table presents the bending maximum strength, strain, and modulus of elasticity values for pure resin and epoxy resins with varying percentages of pistachio shell powder. As the percentage of shell powder increased, the bending maximum strength increased from 145.18 MPa for pure resin to 157.01 MPa for 1 % FK and 167.04 MPa for 2 % FK. The strain values decreased as the percentage of shell powder increased, with the highest strain value of 0.07244 for pure resin and the lowest value of 0.04313 for 2 % FK. The modulus of elasticity also increased from 3.46 GPa for pure resin to 3.92 GPa for 1 % FK and 4.32 GPa for 2 % FK. This indicates that the material became more rigid in structure.

The adhesion between the epoxy matrix and the pistachio shell particles can affect the strength and stiffness of the composite. With increasing levels of pistachio shell addition, the adhesion between the particles and the matrix may increase, leading to improved mechanical properties. The interaction between the filler particles and the epoxy matrix can also affect the mechanical properties of the composite. With higher levels of pistachio shell addition, the filler-matrix interaction may become stronger, resulting in improved load transfer and increased stiffness. On the other hand, the decrease in strain values can be attributed to the reduced mobility of the polymer chains in the resin matrix, resulting in a more rigid structure.

3.3. Vickers Hardness Test

The Vickers hardness test is a widely used method for determining the hardness of materials. The test is preferred due to its simplicity, requiring small amounts of material, generally being non-destructive, and being repeatable (Schneider et al., 1999). It is an optical method, meaning that the indentation left by the test on the surface of the specimen is measured to determine its hardness value. The indentation is made by an equilateral pyramid with a square base made of diamond, with a plane angle of 136°. In this test method, the surface of the specimen is first cleaned and polished, and then it is placed on the device perpendicular to the indentation axis. The clarity and brightness of the measuring microscope are adjusted, and the area to be tested is selected. An appropriate load is determined, and the device is operated to ensure that the indentation is made. The indentation is then retracted, and the optical image is analyzed to examine the indentation left. Then, the diagonals of the indentation are calculated. If one diagonal is more than 5 % longer than the other diagonal, or if the four corners of the indentation are not in sharp focus, it means that the test surface is not perpendicular to the indentation axis. Such an indentation can give incorrect data, and deviations may occur in the hardness values (HV) calculated based on this. In such a case, the specimen alignment should be checked, and the test should be repeated. If there is no problem with the diagonal lengths, the diagonals are measured with a precision of 0.1 μm, their averages are taken, and the hardness value is calculated by substituting them into Equation 4.

$$HV = 1.8544\frac{P}{d^2} \tag{4}$$

The applied load P (N) represents the load, and d (mm) represents the average of diagonal lengths. The measurement principle with this test method is shown in Figure 7.

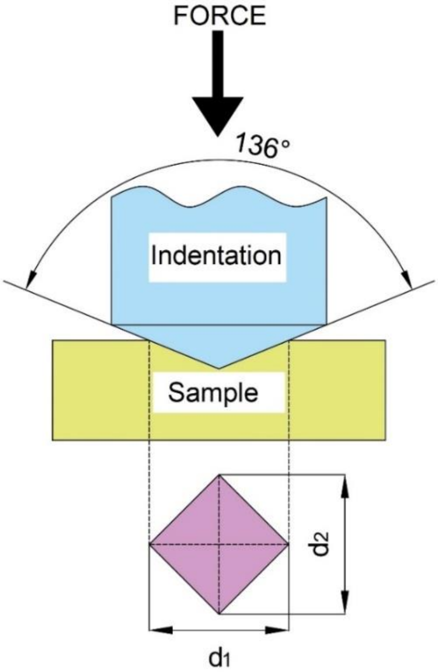

Figure 7. Vickers hardness principle

In this study, Vickers hardness tests were conducted on each sample by taking 5 indents according to the ASTM E384-17 standard. A 0.5-kg load was applied for 5 seconds during the indentation process. The indentation was then analyzed under a microscope, and the lengths of the diagonals were measured to calculate the Vickers hardness values (HV) using Equation 4. An example of an indentation mark is shown in Figure 8, while the Vickers hardness values are presented in Figure 9.

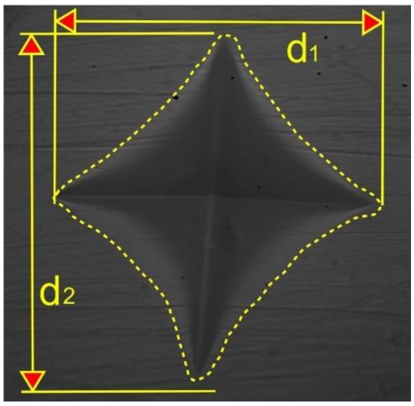

Figure 8. The diagonals of the indentation

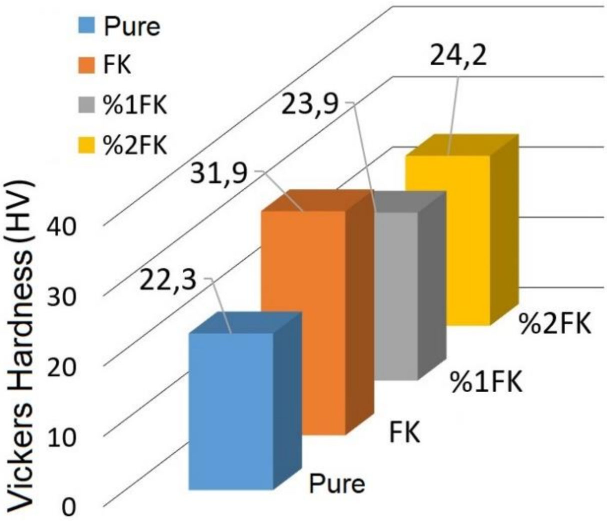

Figure 9. Hardness values

In this graph, the hardness value of the pistachio shell is also given with the FK notation. As can be understood from the graph, since the hardness value of the shell powder is higher than that of the epoxy resin, an increase in the amount of powder causes an increase in the hardness value of the epoxy. This value was measured as 22.3 HV in pure epoxy and 24.2 HV in a sample with 2 % powder content. Changes in the microstructure of the composite may have caused this due to the addition of a filling material that can lead to changes in the arrangement of molecules in the composite and change its mechanical properties. Fadhil and Hadi (2021) added pistachio shell powder to the epoxy resin in proportions of 5 %, 7 %, and 9 %, and examined their wear and hardness values. They measured the hardness values on the Hardness Shore D scale. They found that as the amount of powder increased, the wear and hardness values also increased, and the highest values were observed in samples with 9% powder content. Chandrakar et al. (2021) used pistachio shells in proportions of 5 %, 10 %, 15 %, 20 %, 25 %, and 30 % compared to epoxy resin and examined their physical and mechanical properties. They observed that the hardness values increased as the proportion of pistachio shell increased. The mechanism behind this increase can be explained by the addition of a filler material, which increases the elasticity modulus of the polymer composite material. The increase in modulus led to an increase in the hardness of the composite (Cheng and Cheng, 1998).

3.4. Microstructure and Density

Microstructure analysis is crucial in examining the compatibility between bio-fillers and polymers. Microscopic images are an important method of examination, showing how the filler material is distributed within the matrix material (Mohajer et al., 2017). In this study, micro-images were taken at 100x magnification to determine the compatibility of the pistachio shell powder used with the polymer, and they are shown in Figure 10. As seen from the images, no particles were found in the pure resin. In the sample with 1% filler, particles are clearly visible, and their sizes are found to be variable. This is because the powdered particles were passed through a sieve with a size of 75 μm.

Since particles smaller than this size were present in the powder, these particles were not filtered out. When the sample with 2 % filler was examined, similar particle sizes were observed, but the number of particles was found to be greater than in the 1% filler sample as the filler ratio increased. Additionally, it is evident from the images that the distribution of particles is nearly homogeneous, and there is no indication of agglomeration. The filler material is nicely delimited by the matrix body, and there is no visible void between the two phases. Furthermore, it was observed that no voids were present in the composite during the fabrication process, which is usually encountered in composites. This is because the mixture was placed in a vacuum chamber to remove air bubbles. Therefore, it can be concluded that there is good compatibility between the filler and matrix phases (Ji et al., 2002).

In order to examine the density of epoxy composites, the Archimedes principle was used to measure the density according to the ASTM D792-91 standard. This measurement method is based on the principle of measuring the weight of the sample in air and in water. The first step is to measure the temperature of the pure water used in the method, as the density of water changes with temperature. Once the temperature is determined, the weight of the sample is measured first in air and then in water, and these values are used in Equation 5 to calculate the density.

$$\rho = \frac{\rho_w}{1-(W_w/W_a)} \tag{5}$$

ρ_w (g/cm^3) represents the density of water at the measured temperature, W_w (g) is the weight of the sample in water, and W_a (g) is the weight of the sample in air. To obtain accurate results, five measurements were taken, and the average values are presented in Figure 11. It was observed that the density value increased as the amount of pistachio shell powder added to the resin increased, as the density of the pistachio shell powder was higher than that of the resin. The density of the pistachio shell was measured at 1.311 g/cm^3, while the maximum density of the composite with powder additive was measured at 1.122 g/cm^3 at a 2 % powder additive ratio.

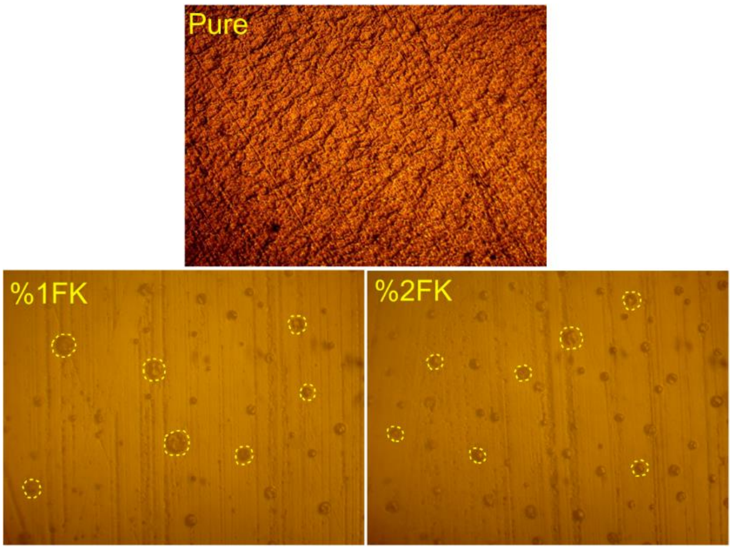

Figure 10. Microscope images

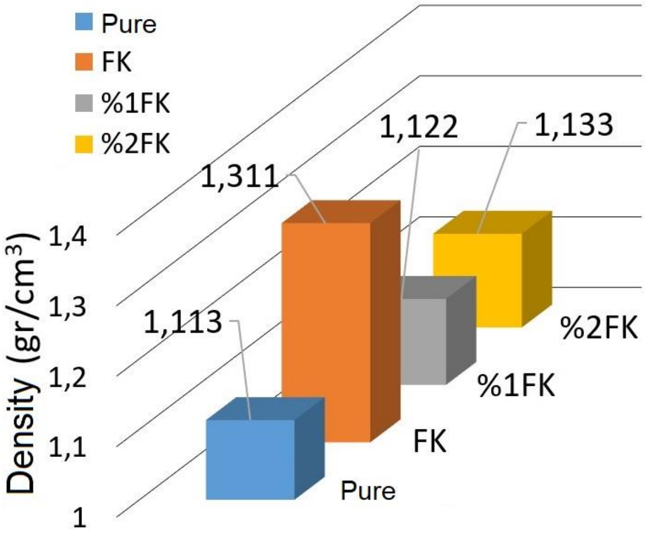

Figure 11. Density values

4. Conclusion

The environmental impact of petroleum has been increasing every year. Researchers are using biofillers as additives to minimize this impact. In this study, epoxy composites were produced using Antep pistachio shells grown in Şanlıurfa as a bio-filler material. The pistachio shells were first reduced to a powder size of 75 µm and added to the epoxy matrix at 1% and 2% ratios using the simultaneous mixing method. Samples were then taken from the prepared plates according to various ASTM standards and subjected to mechanical tests. As the ratio of the additive increased, it was found that the stress and modulus of elasticity values increased and the strain decreased in the tensile and bending tests. A maximum improvement of 8 % was observed in the tensile test, while a 15 % improvement was observed in the elasticity modulus. In the bending test, a 15 % improvement was observed in the maximum strength, and a 24 % improvement was observed in the bending modulus. Hardness values were determined in Vickers hardness units, and it was found that hardness values increased with the increase in powder content. The hardness of the pure epoxy was found to be 22.3 HV, whereas the epoxy composite with the maximum result containing 2 % additive exhibited a hardness of 24.2 HV, signifying a notable 8% increase. As the powder content increased, the densities of the samples also increased. The density of pure epoxy was measured at 1.113 g/cm^3, while the density of the sample with 2% additive was measured as 1.133 g/cm^3, corresponding to an increase of 1.7%. The reason for the small increase in density is that the density of the shell powder is close to the density of the epoxy. Microstructure analysis revealed that there were no air bubbles in the structure and the shell powder was homogeneously distributed within the matrix material. The data obtained from this study shows that Antep pistachio shells are suitable as a bio-filler and can be used as industrial waste.

Competing Interests

The authors declare that there is no competing interest to disclose that is directly or indirectly related to the work submitted for publication.

Author Contribution

The authors declare that they have contributed equally to the article.

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