



The Production and Characterization of Seashell Reinforced Epoxy Composite Material

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ABSTRACT

In this study, it was aimed to examine the production, characterization and mechanical properties of epoxy matrix composites added with seashells collected from different coastal locations of the Mersin Region, which is a natural raw material. Natural seashells with different mass ratios were ground into different sizes and added into epoxy, the matrix material. By performing physical and chemical characterization of the produced composites, the next step is to analyze and produce the product with the best and most superior properties.

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1. Introduction

The limited use of pure materials as a result of today's technology has led to the search for alternatives. As a result, composite properties with improved maintenance have been developed and their areas of use are changing. Composite materials are a group of materials that meet the industry's needs in large quantities. For this reason, for the integration or assembly of composite materials, studies are constantly carried out in different aspects of their performance with different reinforcement and material additions.

As a result of developing technology and a visionary perspective, improving conditions and high performance expectations from obtaining cheap materials, different searches have occurred in reinforcement material preferences during composite production in the industry. Thus, natural raw materials and wastes have become preferred as reinforcement materials. Preferences for composites produced with natural raw material reinforcement have emerged based on criteria such as environmental sensitivity, sustainability and reducing dependence on petroleum and derivative products. Composite production with waste reinforcement is a

preferred way to raise awareness by evaluating materials that negatively affect the environment or are thought to have a negative impact, to offer an alternative solution to waste storage problems and to obtain cheaper reinforcement materials.

In composite materials, the matrix, that is, the main material, holds the reinforcement together, while the reinforcement materials increase the strength by playing a stress-reducing role under the forces to which the composite material is exposed. Capillary crystals, short and long fibers with different arrangements, and particle and chopped ceramics can be used as reinforcement materials in composite materials. The main purpose of these reinforcement materials is to carry the incoming load and increase the hardness and durability of the matrix.

Composites can be classified under different groups, taking into account criteria such as materials, production methods, usage areas, advantages and disadvantages. In general, a classification is made according to the type of reinforcement or matrix found in its structure.

Composites are divided into three main groups according to matrix type. These; They are polymer matrix, metal matrix and ceramic matrix composites.

Polymer materials are materials with low electrical and heat conduction properties compared to metals. In addition, polymers have a much lower operating temperature range than metals and exhibit more plastic properties when placed under a load. In addition, most polymers are resistant to chemicals, but like other materials, polymers cannot resist fatigue. Polymer materials can be permanently colored during production and can be obtained in the desired shapes through determined patterns. Thus, they can visually imitate the desired materials and with these features, they eliminate additional surface and finishing processes. Thanks to their low density, polymers can be preferred and more economical designs can be obtained. Since their oxidation resistance is high, repair requirements are lower compared to metals. In addition, in mass production, it has the advantage of reducing the cost per piece, surface treatments such as coating, painting, and joining processes [1-15].

Polymer materials are the most widely used and widely used materials among composites. The reason for this can be explained as it is easy to produce and the raw materials are generally cheap.

The reasons why epoxy resin is preferred as a matrix can be listed as having high mechanical properties, chemical and abrasion resistance, and electrical properties. In addition, other types of materials can be added to eliminate problems that may arise in epoxy resins due to their low resistance to crack propagation, that is, they are a brittle material. These materials are polymers, inorganic fillers, reactive diluents and similar materials [1-15]. Another reason why epoxy resins are preferred is that they can be used with all kinds of fillers or reinforcements.

In this study, it was aimed to examine the mechanical properties of epoxy composites with natural raw materials and waste. Natural raw materials and wastes in different mass ratios (seashell, glass waste, palm tree, bird feather, loofah) will be ground into different sizes and added into epoxy, which is the matrix material. Physical and chemical characterization of each composite produced will be made and the next step is to analyze and produce the product with the best and most superior properties.

2. Material and Method

In the study, the grinding process was carried out on the "IKA" M20 brand and model grinding device. Ika M20 universal grinder allows grinding hard and brittle samples at 20,000 rpm up to a maximum volume of 250 mL. Figure 3.1 shows the outside of the grinder device (a) and the "M21" steel blade (b). In the experimental study, weighing was done with a precision scale (c) of the brand and model

"G&G JJ224BC" with a capacity of 220 g and an accuracy of 0.0001 g.

Loofah (a), palm fiber (b), seashell (c), glass waste (d) and bird feathers (e) to be used as reinforcement materials in the production of composite materials were obtained from various places. The obtained products were first subjected to a cleaning process. The cleaned materials were then ground and passed through a 500 μm sieve, the under-sieve products were used, and the over-sieve products were ground again and the same process was repeated. In the production of composite materials, "Armor chemical AC520" brand and model epoxy(f) with a curing temperature of 16 $^{\circ}\text{C}$, a burning temperature of 100 $^{\circ}\text{C}$, and casting type ultra-transparent properties was used [16]. For the tensile sample, mold samples were provided with a distance of 75 mm between jaws and a thickness of 4 mm according to "TS EN ISO 527-2" standards. "Front RTV2" brand mold silicone (a) was used to obtain the drawing molds. Tensile samples fixed on a flat mold (b) were poured mold silicone at room temperature and left to dry for 24 hours (c). Then, the mold and silicone were separated and the mold of the tensile samples was obtained (d).

In the study, the products to be used as reinforcement materials were first cleaned with pure water and left to dry. The dried materials were passed through a grinder with a speed of 20,000 rpm, then passed through a 500 μm sieve and the under-sieve products were separated for use. Epoxy was weighed on a precision scale, 2.5 g and hardener 1.25 g. After the epoxy, the product to be used as reinforcement material was added. After the reinforcement material, hardening resin was added and mixed until a homogeneous mixture was obtained. After the mixing process, the product obtained was poured onto the pre-heated mold, heated to prevent the formation of air bubbles inside, and the curing was waited for 24 hours. The resulting tensile sample was cast as 1%, 5% and 10% by weight for each reinforcement material [17].

Tensile test, which is widely used to investigate the mechanical behavior of the material; It is possible to apply it on many materials, including metals, plastics, composites, films, elastomers, rubber and fabrics. The tensile sample fixed to the jaws of the machine is subjected to a certain load with the help of the integrated computer and graphic data is obtained as a result of the changes observed in the material. It provides important information such as the percent elongation of the material, maximum tensile strength, and modulus of elasticity [18].

The produced composite tensile samples were analyzed with the 'SHIMADZU AG-XD' brand and model tensile test device in the laboratory of the Faculty of Technology of Firat University.

3. Results and Discussions

XRD curves of composites containing epoxy resin and pumpkin fiber in different mass ratios are given in the crystalline in nature, hemicellulose and lignin are amorphous. The composite doped with pumpkin fibers showed the main reflection peak at $2\theta = 19\text{--}22^\circ$ and the corresponding peak in cellulose. In the XRD spectra of

Figure. It can be seen in the figure that the XRD peaks of amorphous compounds have a lower intensity than pure epoxy. The lower intensity of the composite peaks can be interpreted as evidence of a good dispersion of the filler in the polymer matrix. Lignocellulosic materials represent amorphous and crystalline peaks. While cellulose is doped compounds, characteristic peaks of the filler material can be seen around the scanning angle of $2\theta = 19^\circ$ [19].

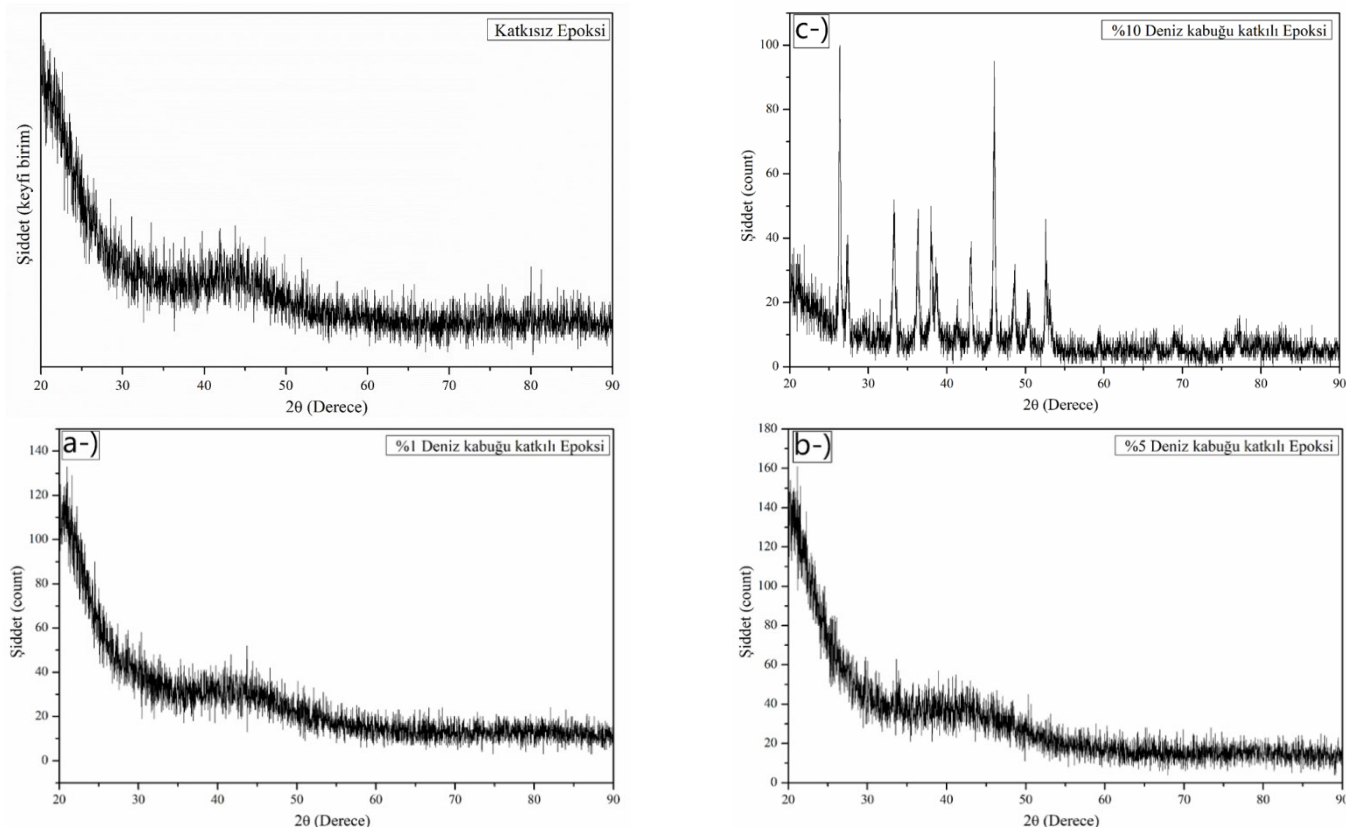


Figure 1 XRD result of pure and a-)1%, b-)5%, c-)10% shell reinforced epoxy composites

FTIR spectrograms of pure epoxy and seashell powder reinforced in different mass ratios into epoxy resin are given in Figure 4.19. From the spectrograms, the first small 'U' bend at 2921 cm^{-1} and 2854 cm^{-1} indicates the OH and CH regions. The second intensity peak is observed at 2364 cm^{-1} . The subsequent intensity peaks correspond to asymmetric stretching and vibrations of $\text{C}=\text{O}$, attributed to the presence of CaCO_3 . The final intensity peaks are interpreted as bending of the CO_3^{2-} plane, attributed to the presence of calcite. As the seashell powder content increased, the peak intensity gradually increased and

completely disappeared at 1% seashell powder content. This was due to the interaction with the polymer matrix of the increased seashell powder. The characteristic $\text{C}=\text{O}$ (Amide) tensile vibration peak was observed for all composites except the composite containing 1% seashell powder. Asymmetric stretching vibration of carbonate ion was found in all composites. As a result, it was understood by FTIR analysis that the aragonite phase of calcium carbonate was dominant [20].

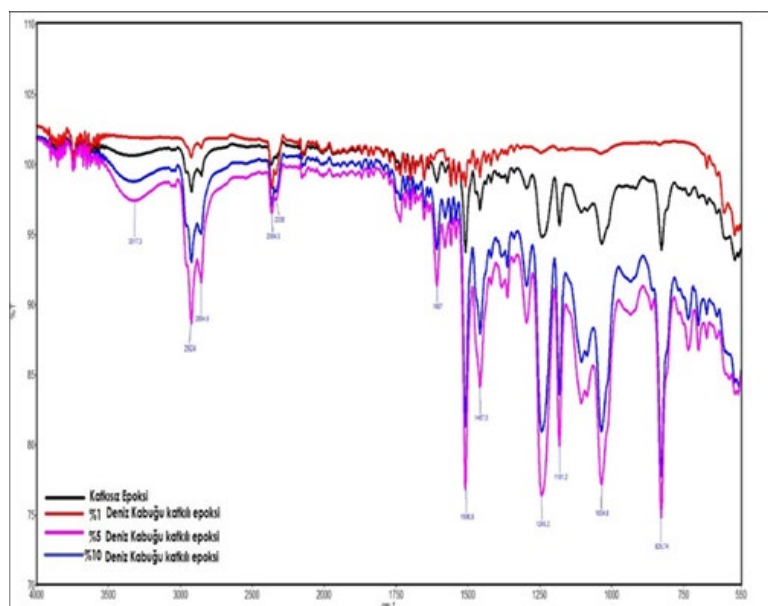


Figure 2 The transmittance spectra vs. wavenumber of the samples

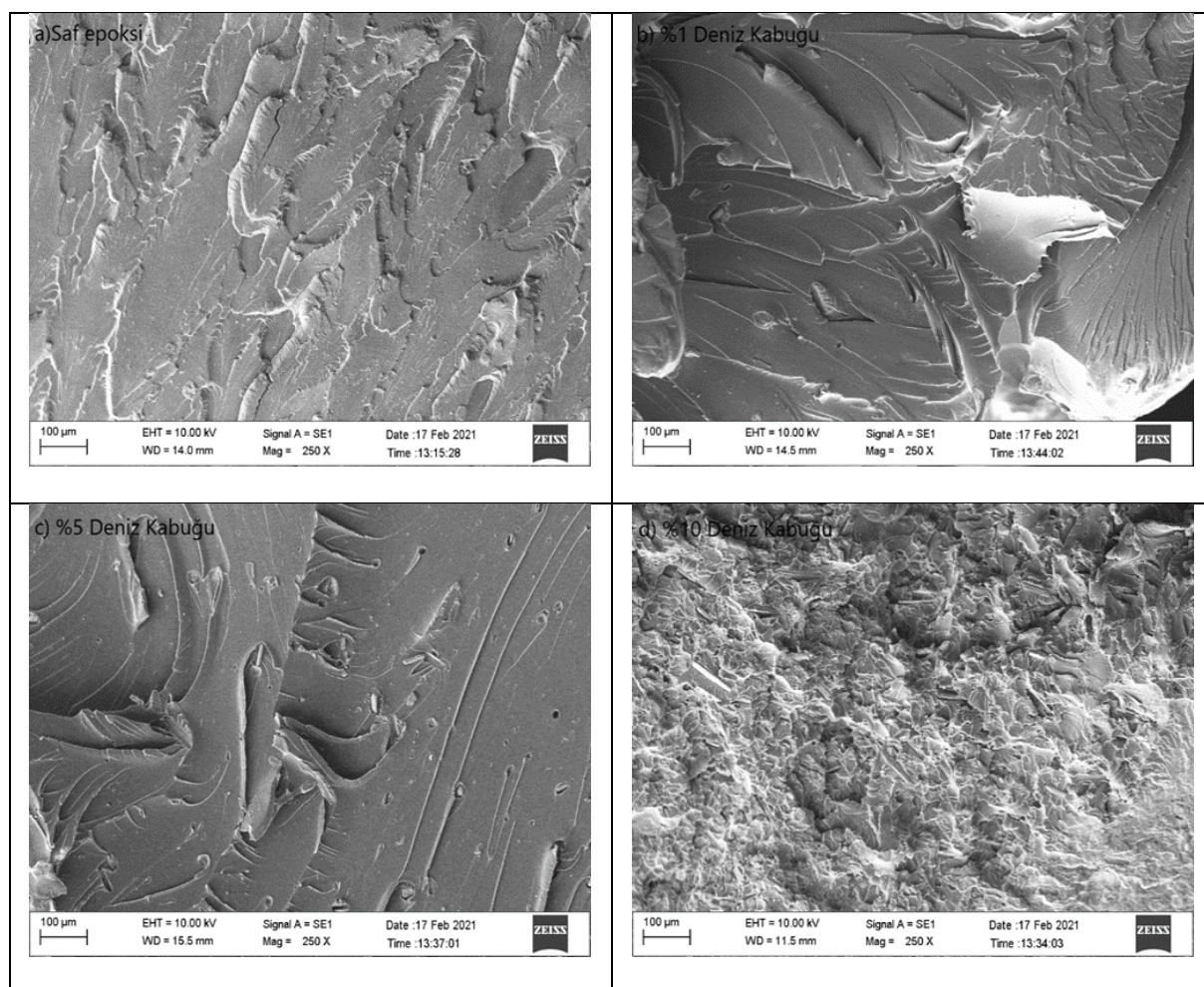


Figure 3 a-)Pure epoxy, b-)1%, c-)5%, d-)10% SEM images of seashell reinforced epoxy composites

Homogeneous distribution of reinforcement materials within matrix materials can affect the mechanical and structural properties as well as thermal properties of composite materials. As can be seen in Figure 4.14, structures with a high content of layered CaCO_3 particles contained in the reinforcement material, the seashell, were observed more specifically. While the filling caused roughness in the cross-section of all samples, agglomeration was also observed in some regions. This agglomeration tendency with increasing filler content is due to the high hydrophilicity and high surface energy of CaCO_3 . It was observed from the SEM images that the epoxy composite containing 1% seashell had a rough and irregular surface, while the presence of many small and irregular seashell particles was observed on the surface of the composite containing 5% seashell, and the surface texture of this naturally reinforced composite was relatively smooth compared to other composites. From the SEM images, no significant differences were observed in the surface morphology of the composites containing 10% seashell. It can be seen in SEM images that these composites have heterogeneous surface morphology with small particles [21].

Table 1. Stress-Strain values of the samples

Samples	E(MPa)	σ (MPa)	amount of extension (%)
Seashell 1	1720.38	34.2667	4.60514
Seashell 5	1124.00	19.2277	6.03699
Seashell 10	1676.05	47.2462	5.50162
Pure	2087.24	31.2460	4.20931

The Stress-Strain graph of the sample obtained using pure epoxy resin is shown in Figure 4.1. The graph of the sample containing 1% seashell powder supplement by mass is shown in Figure 4.3 a-). While the tensile strength of the pure epoxy sample was determined as 31.2460 MPa, the percent elongation amount was 4.2093% and the elasticity modulus was 2087.24 MPa, the tensile strength of the sample containing 1% seashell powder reinforcement by mass was determined as 34.8464 MPa and the percent elongation amount was 4.6051%. It is observed that supplementing the samples with 1% seashell powder by mass causes an increase in the tensile strength and elongation of the sample compared to pure epoxy [22].

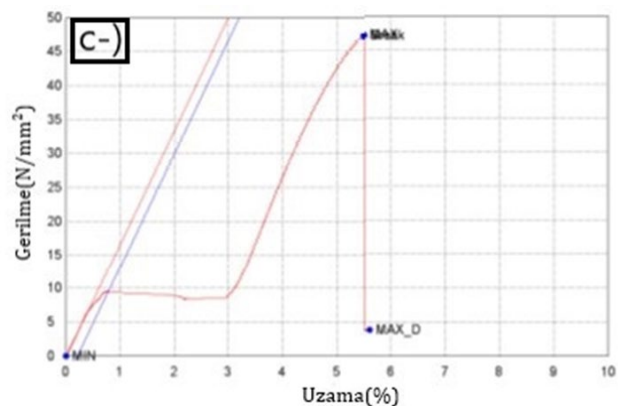
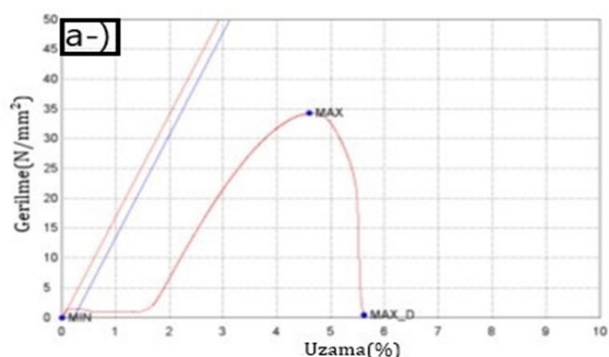
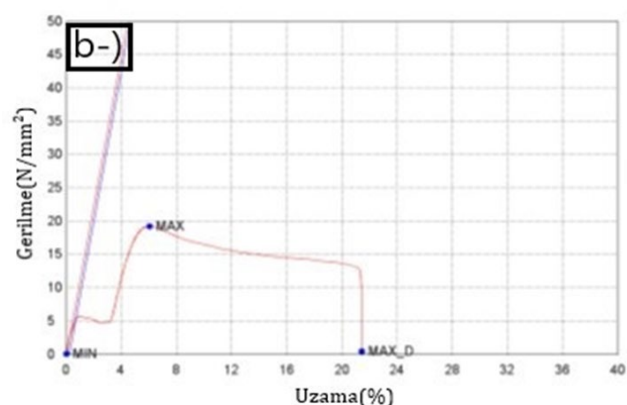
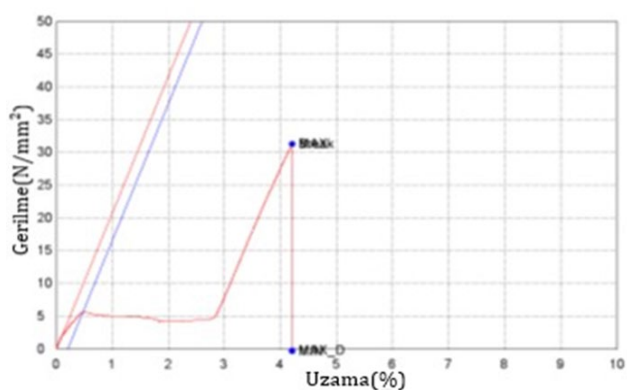


Figure 4 Stress-strain graphs of pure and a-) 1% b-) 5% c-) 10% shell reinforced composites

The Stress-Strain graph of the composite sample containing 5% seashell powder by mass is shown in Figure

4.3 b-). According to the graph, the tensile strength was 19.2277 MPa and the percent elongation value was 6.0369%. Compared to pure epoxy and 1% waste glass reinforced composite samples, as the amount of reinforcement increases, the tensile strength decreases and the percent elongation increases.

The Stress-Strain graph of the composite sample reinforced with 10% seashell powder by mass is shown in Figure 4.3 c-). According to the graph, the tensile strength was 47.2462 MPa and the percent elongation value was 5.5016%. Compared to pure epoxy and 1% and 5% waste glass reinforced composite samples, the tensile strength of the 10% added sample increases with the increase in the amount of reinforcement. When the percentage elongation amount is examined, there is not much difference compared to pure, 1% and 5% samples [23].

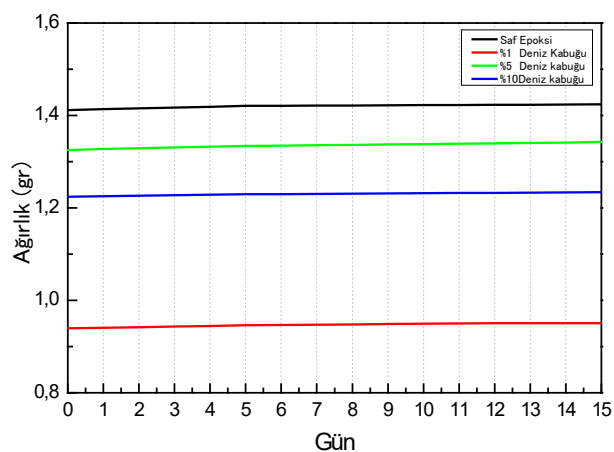


Figure 5 Water sorption values of the samples

The water sorption value of pure resins was found to be 0.87% for a period of 15 days. When the weight graphs of epoxy composites containing 1-5-10% reinforcement material by weight are examined, changing depending on the days; It is seen that the water sorption of the composites increases over time and reaches saturation.

When the water sorption of seashell composites is examined, the values of 1%, 5% and 10% seashell reinforced samples are as follows, respectively; Calculated as 1.22%, 1.29%, 0.82%. Water sorption values of all composites were found to be higher than their pure matrices [24, 25]. However, when the composites were compared among themselves, it was seen that there was a decrease in water sorption as the seashell ratio increased. The reason for this situation can be explained as follows: 95% of the seashell consists of calcium carbonate crystals. 5% is organic materials consisting of approximately 30

different proteins. These proteins hold together bricks made of calcium carbonate crystals, such as iron plates and columns, and ensure that the shell is hard and strong. Calcium carbonate is assumed to be insoluble in water, so it does not absorb water significantly. Therefore, the increase in water absorption of seashell reinforced composites is probably due to the organic compounds in the seashell. Accordingly, the water sorption of composites decreases with the increase in the amount of seashell [26].

4. Discussion

In this study, glass waste, seashell, pumpkin fiber, bird feather and palm fiber were used to produce natural raw materials and waste-reinforced composites. First of all, these natural materials were ground into different grain sizes. Then, it was brought into composite form with epoxy and the structural (physical and chemical) properties of these composites were determined by tensile, XRD, SEM, FT-IR and water sorption analyses. The results regarding all the detected features are listed below. For this purpose, it was aimed to compare the mechanical and physical properties of composite samples. The samples were subjected to tensile testing and their physical and chemical properties, such as maximum stress and percent elongation values, microstructures, bond structures, and water sorption, were compared [27].

In pure epoxy composite materials containing 1% reinforcement material by weight, the tensile strength value was determined as pumpkin fiber, glass powder, seashell, palm fiber, pure epoxy and bird feather reinforced epoxy composites, from largest to smallest.

In pure epoxy composite materials containing 5% reinforcement material by weight, the tensile strength value was determined as glass powder, pure epoxy, palm fiber, pumpkin fiber, seashell and bird feather reinforced epoxy composites, from largest to smallest.

In pure epoxy composite materials containing 10% reinforcement material by weight, the tensile strength value was determined as seashell, glass powder, pure epoxy, palm fiber, bird feather and pumpkin fiber reinforced epoxy composites, from largest to smallest [28]. Although these composite materials obtained have low strength compared to other composites, when they are evaluated as waste and natural raw materials, a material that is less costly and less harmful to natural life will be produced in applications where lower tensile strength is required.

It has been observed that the functionalization process increases the surface bonding properties of natural fiber reinforced composite materials. For this reason, it has been observed that natural fiber reinforced composites can be used instead, taking into account the costs of commonly used composite materials, depending on the desired feature in the place of use [29].

It is envisaged that different results can be obtained by using composite combinations with different ratios of reinforcement material and matrix material and by composite materials depending on the place of use. Especially considering that waste is increasing and natural raw materials are in very high quantities, research and development should be carried out on low-cost, environmentally friendly and recyclable composite materials and they should be brought into industrial use.

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changing parameters such as mixing homogeneous structure.

Test results also show that natural fiber reinforced composite materials can be preferred to classically used

Competing interests

The authors declare that they have no competing interests.

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