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Determination of thermophysical properties of *Ficus elastica* leaves reinforced epoxy composite

Kauçuk ağacı (*Ficus elastica)* yaprağı takviyeli epoksi kompozitin termofiziksel özelliklerinin belirlenmesi

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Research Article

Abstract

In this study, *Ficus elastica* leaves have been reinforced into an epoxy composite and some physical and chemical characterization of the obtained composite is made. *Ficus elastica* leaves are ground between 297 and 149 microns. The biomass (*Ficus elastica*) prepared as a filler material is kept in sodium hydroxide (% 7 NaOH) solution for 24 hours for alkali activation. It is then washed three times with distilled water and dried in an oven at 75 °C for 3 hours. Composite production is carried out by reinforcing the prepared filler to the epoxy resin in certain proportions by mass. The effect of the biomass filler added at the rate of 0 wt.%, 1 wt.%, 3 wt.%, 5 wt.%, and 7 wt.% on the density, Shore D hardness, thermal conductivity coefficient, and activation energy of the epoxy composite is determined. According to the results obtained, the density of the epoxy composite decreases as the filler ratio in the mixture increases. Shore D hardness of epoxy composite decreases with the addition of biomass filler. The epoxy composite produced with biomass reinforcement reduces both the thermal conductivity coefficient and the activation energy. Besides, when the chemical bond structure of the obtained polyester composite is analyzed by Fourier transform infrared spectrometer (FTIR), it is seen that there is a physical interaction. According to scanning electron microscopy (SEM) images, 5 wt.% and 7 wt.% reinforcement of *Ficus elastica* leaves negatively affects the surface morphology of the epoxy composite.

Keywords: Epoxy composite, Ficus elastica, Density, Shore D hardness**,** Thermal conductivity coefficient, Activation energy

Özet

Bu çalışmada, *Ficus elastika* yaprakları takviye edilerek epoksi kompozit üretilmekte ve elde edilen kompozitin bazı fiziksel ve kimyasal özellikleri karakterize edilmektedir. *Ficus elastika* yaprakları 297 ile 149 mikron arasında öğütülmektedir. Dolgu maddesi olarak hazırlanan biyokütle (*Ficus elastika*) alkali aktivasyonu için % 7'lik sodyum hidroksit (NaOH) çözeltisinde 24 saat bekletilmektedir. Daha sonra distile su ile 3 kez yıkanmakta ve 75 °C sıcaklıkta etüvde 3 saat kurutulmaktadır. Kompozit üretimi, hazırlanan dolgu maddesinin epoksi reçineye kütlece belirli oranlarda takviye edilmesiyle gerçekleştirilmektedir. Ağırlıkça % 0, % 1, % 3, % 5 ve % 7 oranlarında eklenen biyokütle dolgu maddesinin yoğunluk, Shore D sertlik, ısıl iletkenlik katsayısı ve aktivasyon enerjisi üzerine etkisi epoksi kompozitte belirlenmektedir. Elde edilen sonuçlara göre karışımdaki dolgu oranı arttıkça epoksi kompozitin yoğunluğu azalmaktadır. Epoksi kompozitin Shore D sertliği, biyokütle dolgu maddesi ilavesiyle azalmaktadır. Biyokütle takviyesi ile üretilen epoksi kompozitin hem ısıl iletkenlik katsayısını hem de aktivasyon enerjisini düşürmektedir. Ayrıca elde edilen polyester kompozitin kimyasal bağ yapısı Fourier dönüşümlü kızılötesi spektrometre (FTIR) ile incelendiğinde fiziksel bir etkileşimin gerçekleştiği görülmektedir. Taramalı elektron mikroskobu (SEM) görüntülerine göre, *Ficus elastika* yapraklarının ağırlıkça % 5 ve % 7 takviyesi, epoksi kompozitin yüzey morfolojisini olumsuz etkilemektedir.

Anahtar kelimeler: Epoksi kompozit, Ficus elastica, Yoğunluk, Shore D sertlik, Isıl iletkenlik katsayısı, Aktivasyon enerjisi

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1. Introduction

Today, the use of bioresources in the production of pure polymers and composites is becoming more and more common. Especially environmentally friendly, economical, and some mechanical properties improved polymers are preferred. For example, inorganic industrial wastes are used to increase thermal stability in the production of polymers such as polyester and epoxy. After the boron factory components and wastes are ground very finely, they are reinforced in polymers as filler. Thus, the density, hardness, and thermal stability of the polymer composite are increased. Improvements are also observed in mechanical properties when used at optimum rates [1-4].

In the literature, inorganic fillers such as colemanite, ulexite, tincal, and borax are used as fillers to obtain polyester and epoxy composites. To work at optimum ratios in the production of composites, such fillers are used in the range of 0 wt.% to 15 wt.%. The addition of 10 wt.% or more inorganic filler negatively affects both the surface morphology and the pore structure of the produced composite. It also increases the coefficient of thermal conductivity, density, and Shore D hardness. Therefore, optimum ratios (usually between 3 wt.% and 6 wt.%) of inorganic filler can be used in the polymer composite [5-9].

Besides, clay, diatomite, pumice, perlite, micro-glass spheres, and nanoparticles are used in the production of composites. To reduce the density of polyester and epoxy composites, low-density inorganic fillers are used. To improve mechanical properties, fillers such as alumina, graphene, carbon nanotube, silicon carbide, and multi-walled carbon nanotubes are preferred in polymer composites. Porous fillers such as pumice and perlite are used to increase the porosity of the composite material [10-18].

There are also studies in the literature on the reuse of industrial polymer wastes in polymer composites. For example, polymer wastes such as polyethylene terephthalate, tire rubber, cable, polyethylene, mask, and polyurethane can be used in composite production. In this way, both economical composite production is realized and polymer wastes that cause environmental pollution are eliminated [19-26].

Many composites are being improved, especially by reinforcing biomass wastes into polymers. Especially plants with a fibrous structure are involved in the production of composites such as polyester and epoxy composite components. Biomass wastes such as sunflower stalk, apricot stone shell, corncob, *Cornus alba*, and *Asphodelus aestivus* have been reinforced into polymer composite [27-32]. The fibrous structure, elastic property, density, economy, workability, and compatibility of biomass wastes are very important. The leaves of rubber trees can be given as an example of the biomass wastes to be used in the study. Rubber trees, whose Latin name is *Ficus elastica*, are used as natural polymers in many sectors. *Ficus elastica* is an ornamental plant originating from tropical Asia. The plant is morphologically shiny and has a thick epicuticular vax layer on the green leaf surface [33-38].

The original direction of this research is the reinforcement of leaves of *Ficus elastica* as filler in the epoxy composite. In this study, biomass is used as a filler to obtain economical, environmentally friendly, and low-density composite materials. Epoxy composite with both low density and low thermal conductivity coefficient has been obtained. Biomass reinforcement at optimum rates (3 wt.%) does not negatively affect the porosity and surface morphology of the composite.

2. Materials and Methods

2.1. Materials

The epoxy resin components used in this study are supplied by Polisan Company. The leaves taken from the rubber ornamental plant (*Ficus elastica*) are prepared for epoxy composite production. Sodium hydroxide (Merck NaOH) is used for alkali activation. Figure 1 shows the dried leaves of *Ficus elastica*.

Figure 1. Dried Ficus elastica leaves

2.2. Methods

Biomass is reinforced in Epoxy A as filler at 0 wt.%, 1 wt.%, 3 wt.%, 5 wt.%, and 7 wt.% ratios. *Ficus elastica* leaves prepared in certain proportions are added to the resin, which is heated from room temperature to 55 °C. After providing a homogeneous mixing at 750 rpm and 5 min, Epoxy B is added to the mixture, mixed for 2 min, and cast into standard molds. After the curing process (24 hours) of the obtained epoxy composite is done, the necessary physical and chemical tests are carried out. The properties of the composite are determined by FTIR, SEM, density, Shore D hardness, thermal conductivity coefficient, and thermal stability tests [39-41]. Table 1 shows the amounts of components used in the epoxy composite production process. In Figure 2, the production scheme for the biomass reinforced epoxy composite is briefly shown.

Table 1. Epoxy composite preparation plan

Epoxy A (g)	Epoxy B (g)	Filler (g)
6.6	3.4	0.0
6.5	3.4	0.1
6.3	3.4	0.3
6.1	3.4	0.5
5.9	3.4	0.7

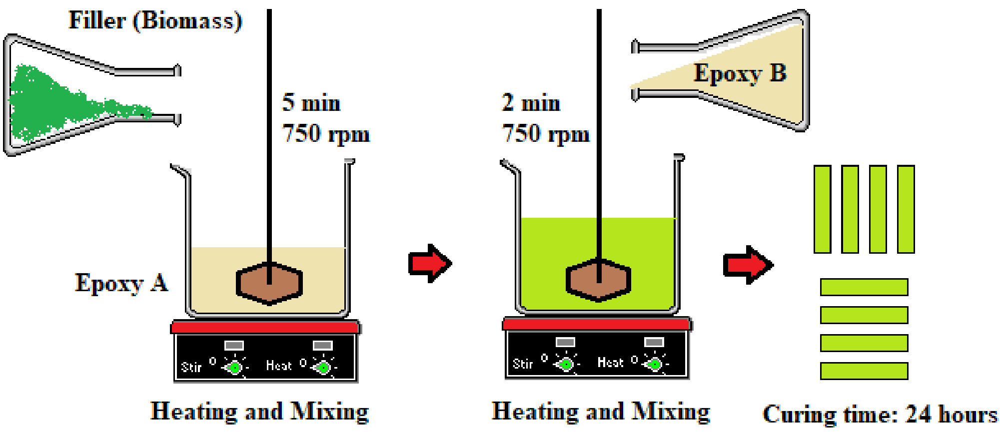

Figure 2. Biomass reinforced epoxy composite production scheme

3. Results and Discussions

3.1. Densities of the epoxy composites

Since the volume occupied by the obtained epoxy composites in standard molds is known, the matrix density is calculated from the mass/volume ratio. As expressed in Figure 3, the density of the epoxy composite decreases with biomass reinforcement. Biomass reinforcement reduces the density of the epoxy composite from 1134 kg/m^3 to 1106 kg/m^3.

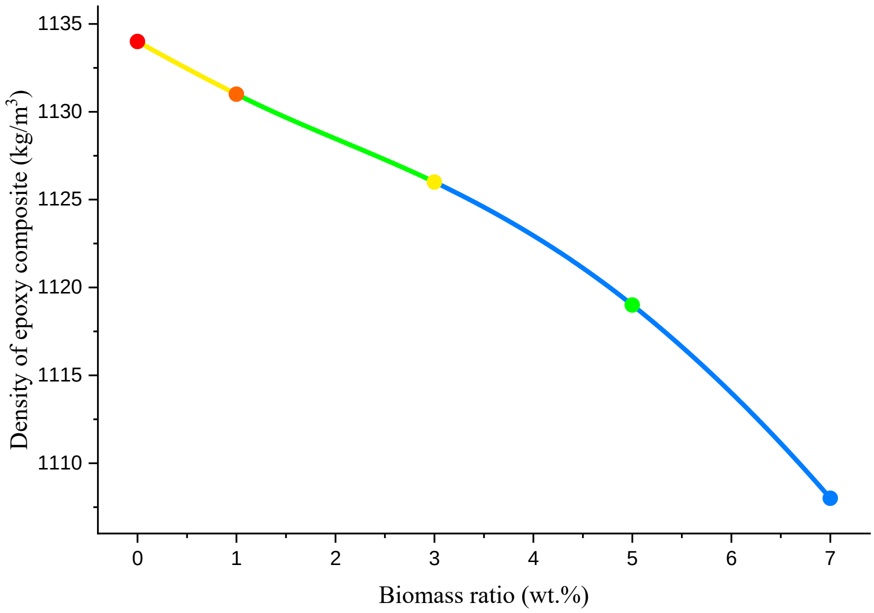

Figure 3. Effect of biomass reinforcement on the density of epoxy composite

3.2. Shore D hardness of the composites

The variation of Shore D hardness values of biomass reinforced epoxy composite with filler ratio is expressed in Figure 4. As seen in this figure, the biomass filler reinforced with epoxy reduces Shore D hardness of the composites. While the hardness value measured in the pure epoxy polymer is 77.5 Shore D, the hardness of 7 wt.% filler reinforced composite decreases to 75.6. *Ficus elastica* leaves can be said to reduce the hardness of the epoxy composite and increase its workability.

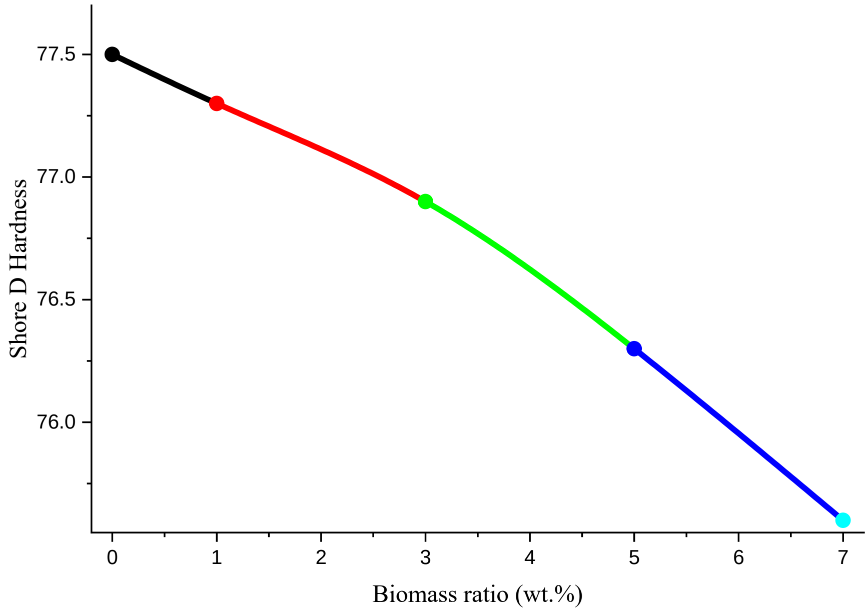

Figure 4. Change of hardness of epoxy composite with reinforcement of *Ficus elastica* leaves

3.3. Thermal conductivity coefficient of the epoxy composites

It has been determined that the thermal conductivity coefficient of the epoxy composite obtained by reinforcing *Ficus elastica* leaves decreases. In Figure 5, the thermal conductivity coefficient of the epoxy polymer is about 0.112 W/m·K, while it drops to 0.092 W/m·K with biomass supplementation.

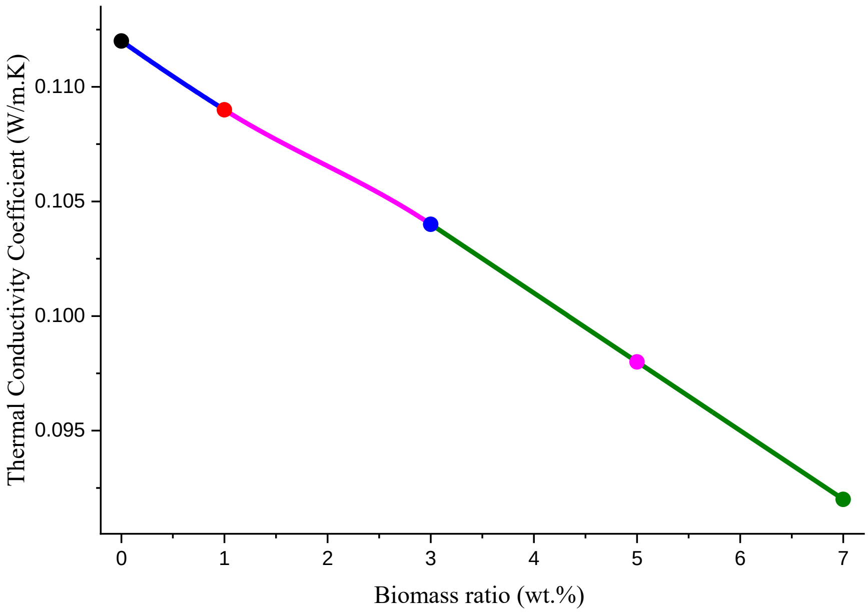

Figure 5. Effect of *Ficus elastica* leaves reinforcement on thermal conductivity of epoxy composite

3.4. Activation energy of the epoxy composites

In this section, the thermal stability of epoxy composites has been evaluated by calculating their activation energies. Weight loss of composites has been measured with temperature increase in an inert environment in a PID (proportional integral derivative) controlled experiment system. Activation energy (*Ea*) values are calculated

according to Coats-Redfern method. In this method, the highest correlation coefficients ($R^2 > 0.9895$) are found with the three-dimensional diffusion equation. The activation energies (conversion rate (α): 0.15-0.85) of biomass reinforced epoxy composites are calculated from room temperature to 600 ℃ with a temperature rise rate of about 10 ℃/min. *Ficus elastica* leaves are found to decrease the activation energy values of the epoxy composite.
In thermal decomposition experiments, physical impurities are removed from the samples in the range of about 25 ℃ to 150 ℃. After a temperature of about 200 ℃, the chemical decomposition region of the epoxy composite begins. It refers to the region where chemical decomposition occurs rapidly in the temperature range of 300 ℃ to 400 ℃. Chemical decomposition appears to slow down in the temperature range of 450 ℃ to 600 ℃. Figure 6 shows the variation of the activation energy of the epoxy composite with the biomass reinforcement ratio [42,43].

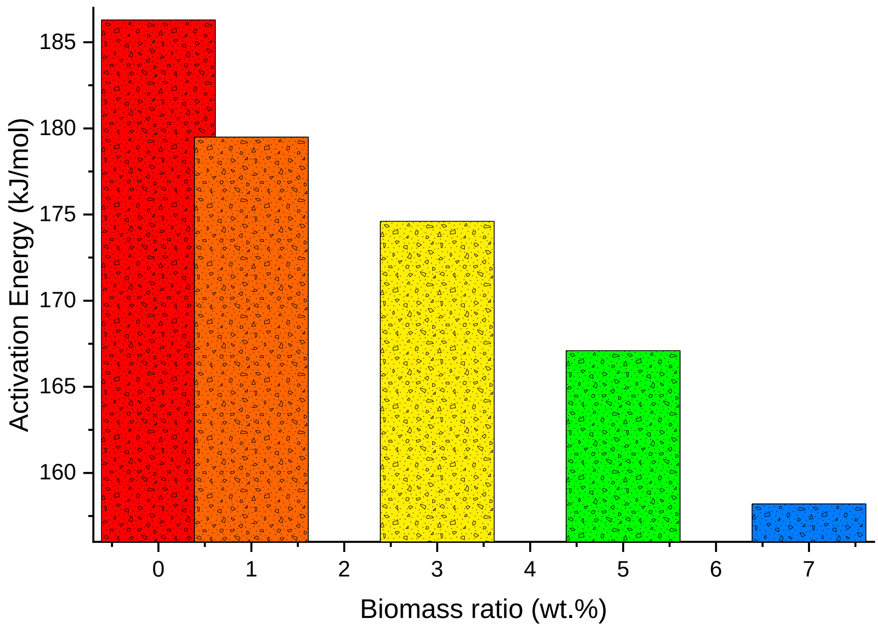

Figure 6. Change of activation energy of epoxy composite with biomass reinforcement

3.4. FTIR spectra of the epoxy composite

In this research, *Ficus elastica* leaves are used as filler in the epoxy composite. According to FTIR spectra results, there is no chemical bond between the biomass and the epoxy resin, but there is a physical interaction. When the FTIR spectra in Figure 7 are examined, C=O stretching vibrations of the ester groups are seen at a wavelength of 1715-1730 cm^{-1}, and a wavelength of 2850-3000 cm^{-1} attributed to the aliphatic C-H stretching band. It indicates the presence of the peak hydroxyl (OH) group seen at a wavelength of 3450 – 3550 cm^{-1}.

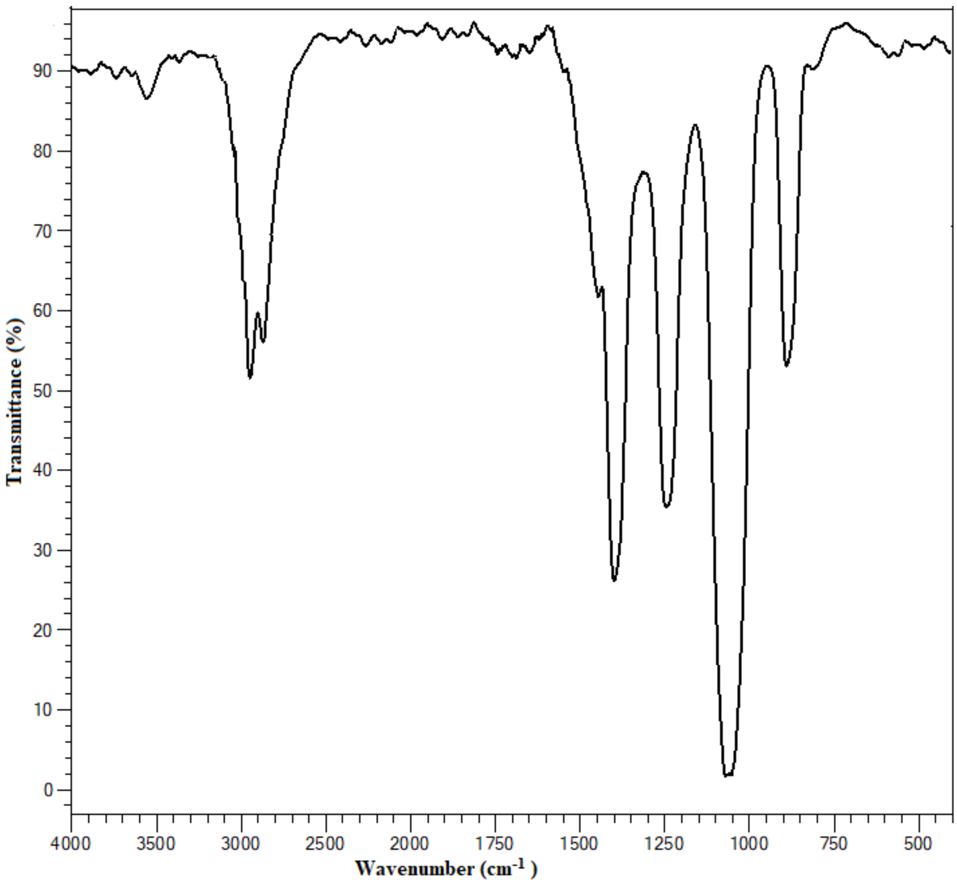

Figure 7. FTIR spectra of biomass (3 wt.%) reinforced epoxy composite

3.5. SEM image of the epoxy composite

In Figure 8, SEM image of the composite is given when the leaves of *Ficus elastica* plant are reinforced into epoxy resin at a rate of 1 wt.%. In Figure 9, it is seen that the high ratio of biomass reinforcement (7 wt.%) has a negative effect on the surface morphology of the epoxy composite and creates an irregular pore structure.

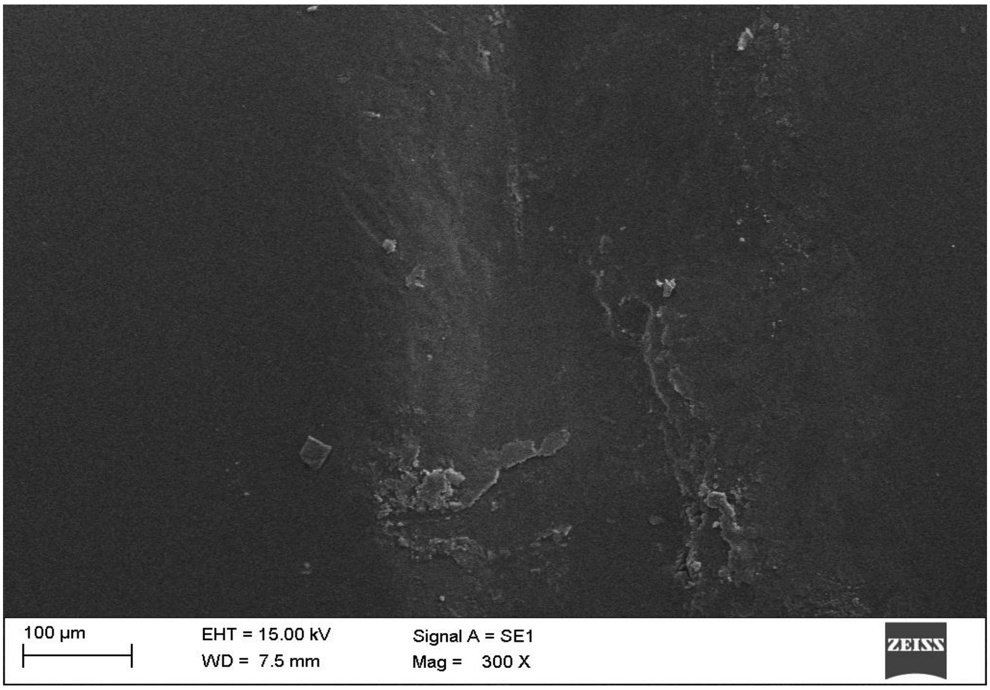

Figure 8. SEM image of epoxy composite reinforced with leaves of *Ficus elastica* (1 wt.%)

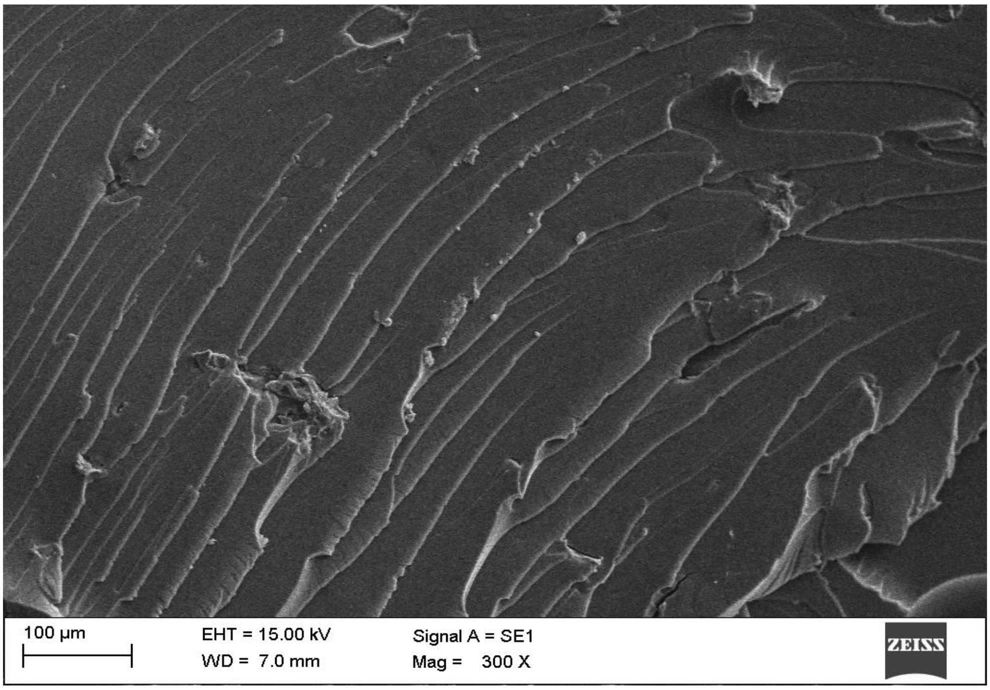

Figure 9. SEM image of epoxy composite reinforced with leaves of *Ficus elastica* (7 wt.%)

4. Conclusions

In this research, economical epoxy composites are produced from renewable resources by using biomass (*Ficus elastica* leaves). Both environmentally friendly and low carbon footprint composites are being improved. In this study, 0 wt.% 1 wt.%, 3 wt.%, 5 wt.%, and 7 wt.% biomass is reinforced into epoxy resin as filler. The thermal conductivity coefficient, activation energy, density, Shore D hardness, surface morphology, and chemical bond structure of the produced epoxy composites have been evaluated. According to the results obtained, biomass reinforcement reduces the density of the epoxy composite. It is seen that the thermal conductivity coefficient of the composite decreases as the filler ratio increases in the epoxy resin. Since biomass reinforcement reduces the activation energy of the epoxy composite, it also reduces its thermal stability. Also, biomass reinforcement reduces Shore D hardness of the epoxy composite. The surface morphology and pore structure of the epoxy composite obtained with the optimum rate (3 wt.%) biomass reinforcement are not negatively affected.

5. Acknowledgments

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6. Author Contribution Statement

Murat Ersin DURĞUN and Abayhan BURAN contributed to making the design, and the literature review contributed to forming the idea, and the analysis of the results. Hasan ARSLANOĞLU and Ercan AYDOĞMUŞ contributed to conducting experimental studies, writing the article checking the spelling, and checking in terms of content.

7. Ethics Committee Approval and Conflict of Interest

There is no need for an ethics committee approval in the prepared article. There is no conflict of interest with any person/institution in the prepared article.

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