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Investigation of some thermophysical properties of Asphodelus aestivus reinforced polyester composite

Çiriş otu takviyeli polyester kompozitin bazı termofiziksel özelliklerinin incelenmesi

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Abstract

In this research, both environmentally friendly and economical composites have been produced by using biomass wastes in unsaturated polyester. The use of renewable biomass wastes as a filler in unsaturated polyester is reduced the carbon footprint of the composite obtained. A low-density and flexible structure could be achieved according to the intended use of polyester composites. While the density of the pure polyester polymer not reinforced with biomass is around 1206 kg/m^3, the density of the 5 wt.% filler added composite decreases to 1167 kg/m^3. Biomass waste (*Asphodelus aestivus L.*) reduces Shore D hardness of the polyester composite and turns it into a more flexible and easy-to-process material. It has been observed that the thermal conductivity coefficient of the biomass reinforced polyester composite shows a slight increase compared to the pure polyester composite. Besides, it has been determined that as the amount of biomass in the polyester composite increases, the activation energy decreases. The specific bond structure in the polyester polymer has been determined by Fourier transform infrared (FTIR) spectroscopy. Biomass waste is not making a chemical bond with polyester, it is only used as a filler. Also, the surface morphology of the polyester composite has been investigated with the help of scanning electron microscopy (SEM). The use of 3 wt.% *Asphodelus aestivus L.* biomass as a filler does not create a negative pore structure on the composite surface.

Keywords: Polyester composite, Biomass, Density, Hardness**,** Thermal conductivity, Activation energy.

Özet

Bu araştırmada biyokütle atıkları doymamış polyester içerisinde kullanılarak hem çevre dostu hem de ekonomik kompozitler üretilmektedir. Yenilenebilir biyokütle atıklarının doymamış polyesterde dolgu maddesi olarak kullanılması, elde edilen kompozitin karbon ayak izini azaltmaktadır. Polyester kompozitlerin kullanım amacına göre düşük yoğunluklu ve esnek bir yapı elde edilebilmektedir. Biyokütle takviye edilmeyen saf polyester polimerin yoğunluğu 1206 kg/m^3 civarında iken, ağırlıkça % 5 dolgu katkılı kompozitin yoğunluğu 1167 kg/m^3'e düşmektedir. Biyokütle atığı (*Asphodelus aestivus L.*), polyester kompozitin Shore D sertliğini azaltmakta ve onu daha esnek ve işlenmesi kolay bir malzemeye dönüştürmektedir. Biyokütle takviye edilen polyester kompozitin ısıl iletkenlik katsayısının saf polyestere göre hafif bir artış gösterdiği gözlemlenmektedir. Ayrıca polyester kompozitteki biyokütle miktarı arttıkça aktivasyon enerjisinin azaldığı tespit edilmektedir. Polyester polimerdeki spesifik bağ yapısı, Fourier transform kızılötesi (FTIR) spektroskopisi ile belirlenmiştir. Biyokütle atığı polyesterde kimyasal bağ yapmamakta, sadece dolgu maddesi olarak kullanılmaktadır. Ayrıca taramalı elektron mikroskobu (SEM) yardımıyla polyester kompozitin yüzey morfolojisi incelenmiştir. Ağırlıkça % 3 *Asphodelus aestivus L.* biyokütlesinin dolgu maddesi olarak kullanılması kompozit yüzeyde negatif gözenek yapısı oluşturmamaktadır.

Anahtar kelimeler: Polyester kompozit, Yoğunluk, Sertlik, Termal iletkenlik, Aktivasyon enerjisi.

1. Introduction

In recent years, the application areas of polyester composites have become quite widespread. However, due to the petrochemical composition of such polymers, there is an increasing trend towards alternative biomass sources both in terms of environmental pollution and for economical production. Polyester composite should have some thermophysical

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properties such as density, hardness, thermal conductivity, and thermal stability suitable for its intended use. For this purpose, many additives and fillers are used in the production of polyester composites in the literature.

For example, polyester composite has been reinforced with borax as a filler. The use of borax in certain proportions by mass increases the density, hardness, mechanical strength, and thermal stability of the composite [1]. Especially sustainable resources (modified palm oil) and recycled polymers (PET) are also used in the production of polyester composites. Thanks to such additives and fillers, fewer petrochemical components are used and environmentally friendly composites can be improved [2]. Besides, many inorganic fillers are used for the thermal stability of polyester composites at high temperatures. In particular, fillers such as fumed silica (aerosil), colemanite, ulexite, and tincal increase the high-temperature performance of polyester composites [3,9].

In another research in the literature, new biocomposite materials have been developed by making a chemical bond with modified palm oil and modified castor oil. In such studies, both physical and chemical modifications are made to the polyester composite. In this way, low density, porous, flexible, economical, and environmentally friendly composites have been improved [4,5].

In particular, materials such as waste masks and waste polyurethane, which are less recyclable, are used in polyester composites after they are prepared in laboratory conditions and ground to the appropriate particle size. In this way, polyester composite materials with high porosity, low density, and thermal conductivity coefficient can be developed. [6,7].

Also, graphene (GF), silicon carbide (SiC), and multi-walled carbon nanotube (MWCNT) have been used in the production of the polyester nanocomposite. With the help of nanoparticles used in certain proportions, the mechanical strength and thermal stability of the composites increase. By using such nanoparticles, polyester composites with high density and hardness can be obtained [8].

Moreover, organic compounds and inorganic alumina particles synthesized in nano size are also reinforced into the polyester composite. In particular, alumina increases both the mechanical strength and thermal stability of the polyester composite [10]. Moreover, biomass wastes from sustainable sources are also preferred as fillers in polyester composites. Apricot stone shells reduce the density of the polyester composite, improve its mechanical properties, and increase its porosity and surface hardness [11].

The original aspect of this research is the use of a local plant species, *Asphodelus aestivus L.*, as a filler in the polyester composite. In this way, economical, environmentally friendly, and low-density composite materials are obtained with biomass wastes. The density of the produced composite has been evaluated by examining its thermal conductivity coefficient, Shore D hardness, activation energy, chemical bond structure, surface morphology, and porosity.

2. Materials and Methods

2.1. Materials

In this study, unsaturated polyester (UP), methyl ethyl ketone peroxide (MEKP), and cobalt octoate (Co Oc) were supplied from Turkuaz Polyester Company. Local plant leaves as a filler (*Asphodelus aestivus L.*) were collected and washed in Elazığ (Türkiye) province. After the leaves dried at room temperature and were ground, they were prepared for polyester composite production. The leaves of *Asphodelus aestivus L.* and its powder form after drying and grinding are shown in Figure 1.

Figure 1. *Asphodelus aestivus L.* used as a filler in the production of polyester composite

2.2. Experimental method

Biomass is mixed homogeneously into the unsaturated polyester resin as a filler at 0 wt.%, 1 wt.%, 1.5 wt.%, 3 w.t%, and 5 wt.%. Then, certain amounts of MEKP and Co Oc components are added to the mixture, respectively, mixed at 1000 rpm for 2 minutes, and poured into standard molds. After waiting 24 hours for the curing process, necessary testing and analysis processes are carried out. Density, Shore D hardness, activation energy, thermal conductivity coefficient, chemical bond structure, and surface morphology of the obtained polyester composite are investigated. In Table 1, the amounts of the components used in the experimental studies for the production of polyester composites are expressed. Also, the polyester composite production scheme is briefly shown in Figure 2.

Table 1. Polyester composite preparation plan

UP (g)	MEKP (g)	Co Oc (g)	Filler (g)
9.80	0.15	0.05	0.00
9.70	0.15	0.05	0.10
9.65	0.15	0.05	0.15
9.50	0.15	0.05	0.30
9.30	0.15	0.05	0.50

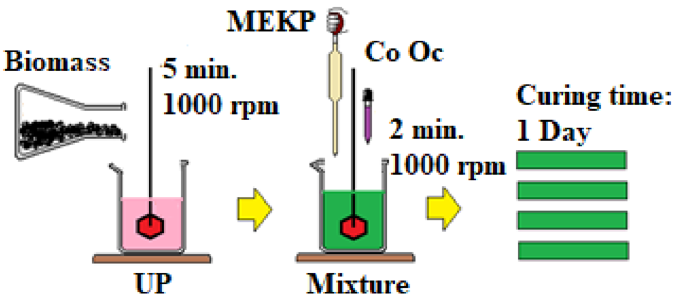

Figure 2. Polyester composite production process and flow chart

3. Results and Discussions

3.1. Density of polyester composites

After the prepared polyester composites are poured into standard molds, the volume of the matrix formed is calculated. Then, the mass is found and divided by the volume and the density values are calculated. As seen in

Figure 3, it has been determined that the density values of the polyester composite decrease with the filler reinforcement.

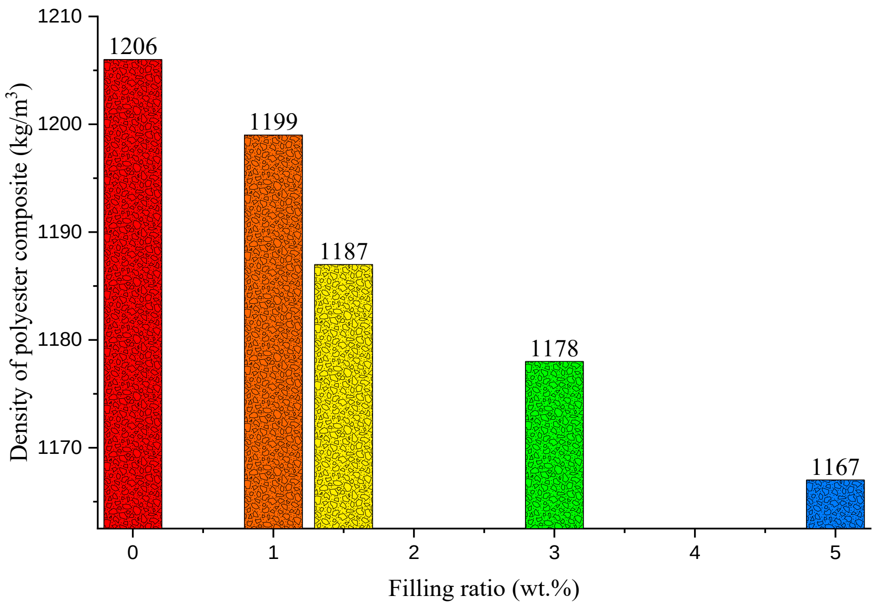

Figure 3. Effect of *Asphodelus aestivus L.* reinforcement on the density of polyester composite

3.2. Shore D hardness of polyester composites

Shore D hardness values of polyester composites produced by reinforcement *Asphodelus aestivus L.* are given in Figure 4. As seen in the graph, it has been determined that the filler added to the pure polyester reduces the hardness of the composite sample. While the hardness value for pure polyester is 78 Shore D, it is observed that 5 wt.% filler reinforcement decreases Shore D hardness of the composite to 76.1. In this case, it can be said that cellulosic biomass creates a more flexible structure than pure polyester.

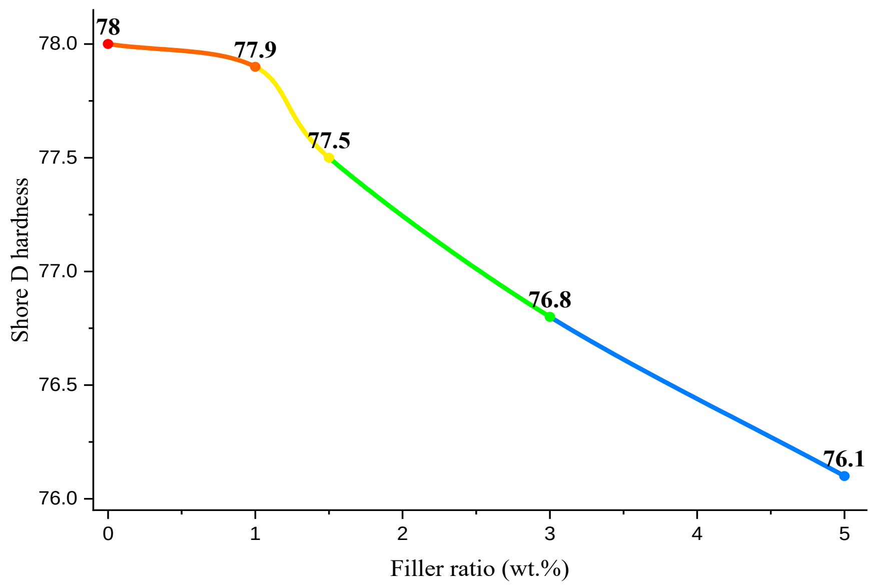

Figure 4. Effect of *Asphodelus aestivus L.* reinforcement on Shore D hardness of polyester composite

3.3. Thermal conductivity of polyester composites

In experimental studies, the thermal conductivity coefficient values of polyester composite samples produced using *Asphodelus aestivus L.* are measured using a thermal conductivity meter (Thermtest portable TLS 100). As seen in Figure 5, while the thermal conductivity coefficient of pure polyester is 0.0565 W/m·K, this value increases slightly with the addition of biomass to pure polyester.

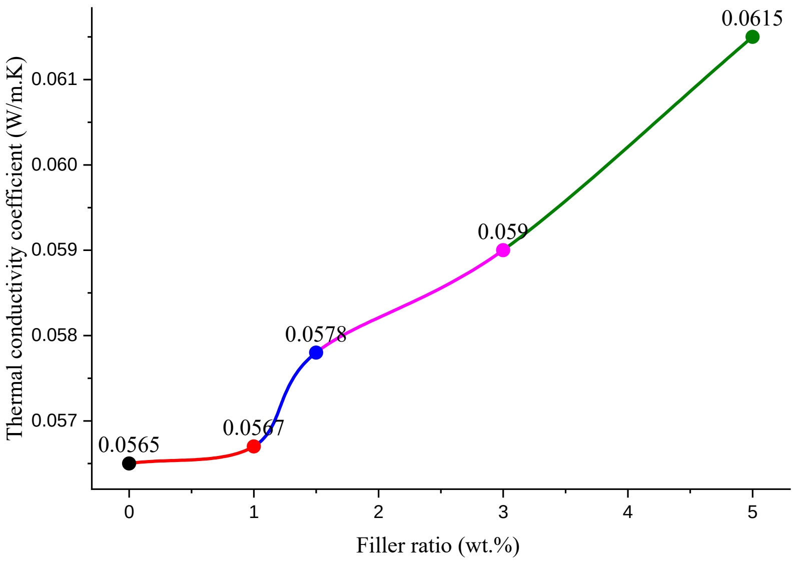

Figure 5. Effect of *Asphodelus aestivus L.* reinforcement on thermal conductivity of polyester composite

The thermal stability of polyester composites has been evaluated by calculating the activation energies. For this purpose, the weight loss of the prepared samples has been found by measuring the temperature increase and the weight loss in an inert environment in the PID-controlled experiment system. In this study, it has been determined that biomass wastes decrease the activation energy values of the polyester composite. Activation energy *(Ea)* values are calculated according to Coats-Redfern method. In this method, the highest correlation coefficients are obtained with the three-dimensional diffusion equation. Activation energies (conversion ratio: 0.2-0.8) of pure polyester and polyester composites reinforced with the filler are calculated in thermal decomposition experiments that have been carried out at a heating rate of about 10 °C/min to 600 °C. Calculated activation energy values are given in Table 2.

Table 2. Calculated *Ea* of biomass reinforced composites

Filler ratio (wt.%)	*Ea* (kJ/mol)
0	114.53
1	108.25
3	104.70
5	102.56

3.4. FTIR spectra of polyester composite

In this study, there is no chemical bonding as the leaves of *Asphodelus aestivus L.* plant are used as filler in the polyester composite. Since there is only a physical interaction, there is no difference in FTIR spectra of pure polyester polymer and composite. Figure 6 shows 1240-1100 cm^{-1} C-O ester groups, 3000 cm^{-1} C-H bonds, 2900-2850 cm^{-1} CH_2 groups vibration stresses in FTIR spectra. A peak is observed in the polyester composite at 1720 cm-1 due to the stretching vibrations of the C=O group. The peaks at 1400 and 1450 cm-1 observed in the polyester composite spectrum indicate the aromatic ring. The peak at 1260 cm^{-1} seen in the polyester spectra is due to the twisting vibration of the CH_2 groups [6-10].

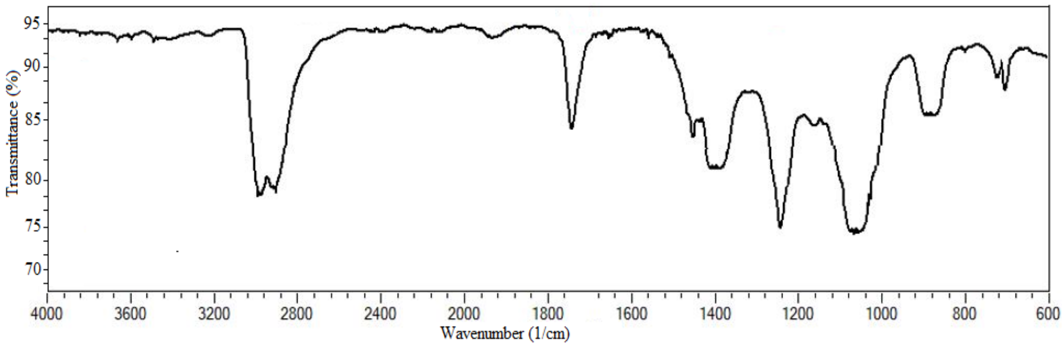

Figure 6. FTIR spectra of polyester composite

3.5. SEM image of polyurethane

In Figure 7, an SEM image of the composite is given when the leaves of *Asphodelus aestivus L.* plant are reinforced with polyester at a rate of 3 wt.%. Since the high rate of biomass reinforcement creates an irregular pore structure in the polyester composite, a rate of 3 wt.% has been preferred.

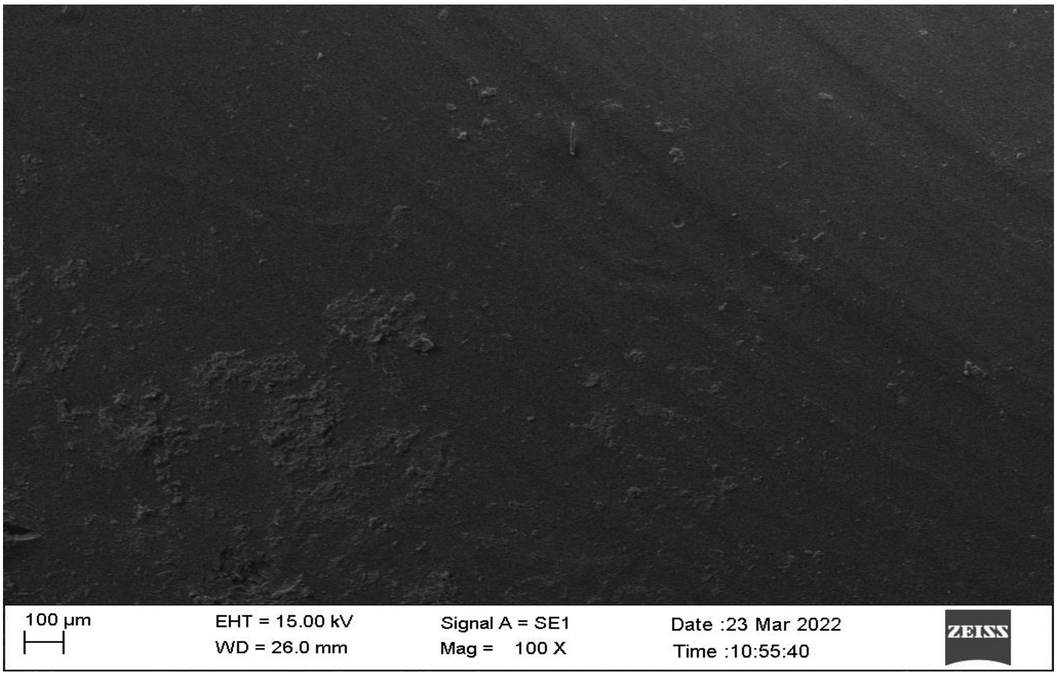

Figure 7. SEM image of *Asphodelus aestivus L.* (3 wt.%) reinforced polyester composite

4. Conclusions

In this study, biomass wastes (*Asphodelus aestivus L.*) have been evaluated and both renewable resources are used and economical biomaterials are produced. 1 wt.%, 1.5 wt.%, 3 wt.%, and 5 wt.% biomass waste is reinforced with unsaturated polyester as a filler. Polyester composite materials have been obtained by using the hand casting method under laboratory conditions. Some physical and chemical properties of the produced polyester composite materials are characterized. For example, thermal conductivity coefficient, activation energy, density and hardness, surface morphology, and chemical bond structure of the composites have been evaluated.

According to the results obtained, it has been determined that the density of the biomass reinforced composites decreases. It has been observed that the thermal conductivity coefficient of the polyester composite raises with the increase in biomass in the mixture, albeit slightly. The activation energy of the biomass reinforced polyester composite decreases as the filler increases. Besides, it is determined that Shore D hardness decreases as the amount of filler in the mixture raises.

The use of biomass wastes in the production of composite materials will enable economical composites to be obtained and the development of environmentally friendly biomaterials with a low carbon footprint.

5. Acknowledgments

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6. Author Contribution Statement

Author 1 contributed to making the design, and the literature review contributed to forming the idea, and the analysis of the results. Author 2 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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