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Research Article

Obtaining Pectin Reinforced Polyester Composite and Investigation of Thermophysical Properties

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Abstract

In this study, pectin powder is mixed homogeneously in unsaturated polyester (UP). For the production of polyester composite, methyl ethyl ketone peroxide (MEKP) and cobalt octoate (Co Oc) are added to the mixture. The obtained composite is poured into standard molds as a gel and allowed to cure for 24 hours. Some physical and chemical properties of the composite are determined and characterization processes are carried out. Density, Shore D hardness, thermal conductivity coefficient, and thermal stability of polyester composite are examined. According to the results obtained, pectin is used as a filler in the polyester composite. There is no change in the chemical structure of the polyester polymer with Fourier transform infrared (FTIR) spectroscopy. The density of polyester composites decreases as the reinforcement of 0 wt.%, 1 wt.%, 3 wt.%, 5 wt.%, and 7 wt.% pectin powder increases. Besides, the reinforcement of pectin powder as filler reduces Shore D hardness of the polyester composite. However, it has been observed that the thermal conductivity coefficient of the polyester composite increases as the filler ratio rises. In thermal decomposition experiments of the composite, the activation energy decreases slightly as the filler ratio increases. According to the optimization results, 3 wt.% pectin powder supplementation does not adversely affect both the surface morphology and thermophysical properties of the polyester composite.

Keywords: Pectin powder, polyester composite, density, Shore D, activation energy.

Pektin Takviyeli Polyester Kompozit Elde Edilmesi ve Termofiziksel Özelliklerinin İncelenmesi

Öz

Bu çalışmada, pektin tozu doymamış polyester (UP) içinde homojen bir şekilde karıştırılmıştır. Polyester kompozit üretimi için karışıma metil etil keton peroksit (MEKP) ve kobalt oktoat (Co Oc) eklenir. Elde edilen kompozit standart kalıplara jel olarak dökülür ve 24 saat kürlenmeye bırakılır. Kompozitin bazı fiziksel ve kimyasal özellikleri belirlenir ve karakterizasyon işlemleri yapılır. Polyester kompozitin yoğunluğu, Shore D sertliği, termal iletkenlik katsayısı ve termal kararlılığı incelenir. Elde edilen sonuçlara göre polyester kompozitte dolgu maddesi olarak pektin kullanılmaktadır. Fourier dönüşümü kızılötesi (FTIR) spektroskopisi ile polyester polimerin kimyasal yapısında herhangi bir değişiklik olmamıştır. Polyester kompozitlerin yoğunluğu, ağırlıkça % 0, % 1, % 3, % 5 ve % 7 pektin tozu takviyesi arttıkça azalır. Ayrıca dolgu maddesi olarak pektin tozunun takviyesi, polyester kompozitin Shore D sertliğini azaltır. Ancak dolgu oranı arttıkça polyester kompozitin ısıl iletkenlik katsayısının arttığı gözlemlenmiştir. Kompozitin ısıl ayrışma deneylerinde dolgu oranı arttıkça aktivasyon enerjisi bir miktar azalmaktadır. Optimizasyon sonuçlarına göre ağırlıkça % 3 pektin tozu ilavesi polyester kompozitin hem yüzey morfolojisini hem de termofiziksel özelliklerini olumsuz etkilememiştir.

Anahtar Kelimeler: Pektin tozu, polyester kompozit, yoğunluk, Shore D, aktivasyon enerjisi.

1. Introduction

Composites based on polymers have become increasingly in demand because they are relatively inexpensive and offer excellent physicochemical properties in terms of thermal, mechanical, and moisture resistance. Natural fiber-reinforced polymer composites, which will not cause environmental problems after use due to their high biodegradability, are a class of sustainable materials that exhibit good mechanical properties for many applications [1-3].

Polysaccharides, the main component of biomass, are the most abundant renewable polymer sources in nature. Pectin, one of the most important among them, has potential uses in many industries. Pectin, which is composed mainly of galacturonic acid units, differs in composition, structure, and molecular weight, and is the most structurally and functionally complex polysaccharide found in plant cell walls [4-7]. Pectin, a homopolymer consisting mainly of galacturonic acid linked by α-1,4-glycosidic bonds, is used in the food industry, cosmetics and personal care products, and the pharmaceutical industry. There are studies in the literature on polyester composites reinforced with natural fibers [8-14].

Composite materials produced by using various fillers in recent years are becoming more common day by day. In particular, composites are strengthened with organic and inorganic reinforcing materials to improve them thermally and mechanically. For example, boron factory components (such as colemanite, ulexite, and tincal) are used to improve the thermophysical properties of polyester composite materials. Besides, as aerosil, alumina, Fe_3O_4, and microsphere reinforcement, it provides mechanical and thermal improvements to the polyester composite [15-23]. Nanoparticles such as graphene (GN), silicon carbide (SiC), and carbon nanotube (CNT) are also preferred in polyester composite production [24].

In studies in the literature, polymer wastes such as polyethylene terephthalate (PET), expanded polystyrene (EPS), mask, and polyurethane are used in polyester composites. Such polymer wastes are both recovered and used in the production of an economical composite material. Hence, the obtained polyester composite has been reinforced with polymer wastes that cause environmental pollution [25-28].

There are also studies in the literature on polyesters synthesized by obtaining raw materials from renewable sources. Both chemical and physical interactions can occur in polyester composites produced using modified palm and castor oil. As a filler, the leaves of some fibrous plants can be reinforced into the polyester composite. In the studies, low density, easy-to-process, economical, and environmentally friendly biocomposites are produced by using the leaves of *Asphodelus aestivus*, waste corncob, *Cornus alba*, and *Ficus elastic*. Optimum ratios should be determined in the production of biomass reinforced polyester composites. The addition of high biomass weakens both the surface morphology and mechanical properties of the composite. Composite materials with irregular pore structures are not preferred. Moreover, high biomass reinforcement weakens the thermal decomposition behavior of the polyester composite. The thermal stability of the composite is evaluated by calculating the activation energies found in the thermal degradation kinetics [29-35].

No report investigating the thermophysical properties of pectin-reinforced polyester composites has been found. This report aims to synthesize pectin reinforced polyester composites and determine their thermophysical properties such as density, hardness, and thermal conductivity to develop highly biodegradable composites with superior thermophysical properties. In this study, it is understood that polyester composite materials can be produced by reinforcing pectin powder. By using renewable resources (biomass), low-density, economical, and easy-to-process biocomposites are being improved. Also, environmentally friendly composites are produced by reducing the petrochemical composition and carbon footprint of the polyester composite.

2. Materials and Methods

The pectin powder used in this study is under 149 microns in particle size. The bulk density of pectin powder is determined as 0.113 g/cm^3.

Unsaturated polyester (UP), methyl ethyl ketone peroxide (MEKP), and cobalt octoate (Co Oc) used in the experiments are supplied by Turkuaz Polyester Company.

In this study, pectin powder is supplemented into UP and homogenization is provided for 5 minutes at a mixing speed of 750 rpm. Then, the chemical reaction is carried out by adding MEKP and Co Oc components to the mixture in certain proportions. The mixture obtained is mixed at 750 rpm for 2 minutes and then poured into standard molds. After waiting 24 hours for the obtained polyester composite to cure, necessary physical tests and chemical analyzes are performed [32,33].

In Figure 1, ground and dried pectin powders are prepared for polyester composite production.

Figure 1. Pectin powder

Figure 2 shows the production of the polyester composite and its cured form in the standard mold.

Figure 2. Polyester composite production scheme

In Table 1, UP, MEKP, and Co Oc components used in the experiments and their weight ratios are expressed.

Table 1. Composite production plan

UP (wt.%)	MEKP (wt.%)	Co Oc (wt.%)	Pectin (wt.%)
98.0	1.4	0.6	0
97.0	1.4	0.6	1
95.0	1.4	0.6	3
93.0	1.4	0.6	5
91.0	1.4	0.6	7

3. Results and Discussion

In this research, the density, Shore D hardness, thermal conductivity coefficient, and surface morphology of the produced polyester composite have been investigated. As seen in Figure 3, pectin powder supplementation slightly reduces the density of the polyester composite. In Figure 4, it has been determined that Shore D hardness of the polyester composite decreases with increasing filler content. However, pectin powder supplementation slightly raises the thermal conductivity coefficient of the produced polyester composite (Figure 5).

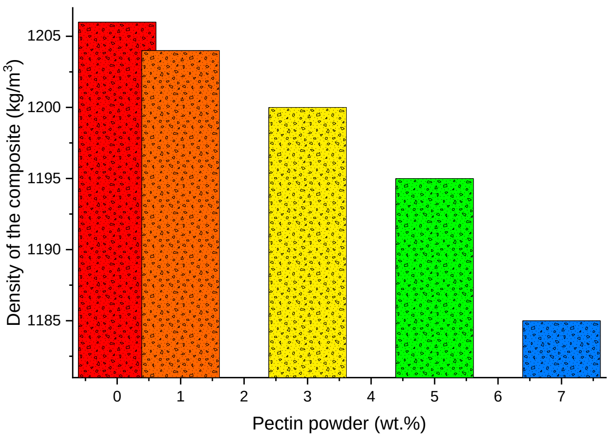

Figure 3. The effect of pectin reinforcement on the density of the composite

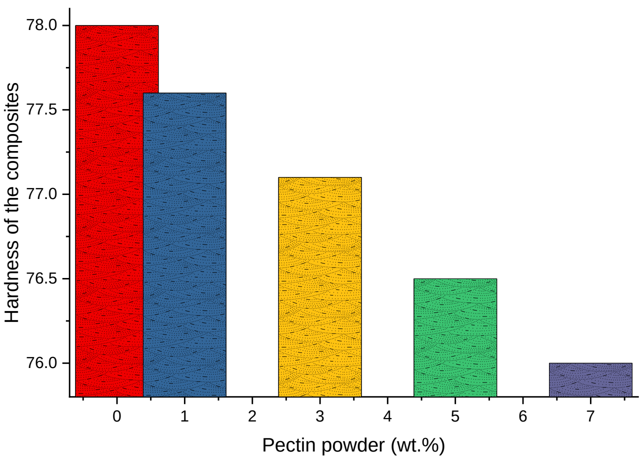

Figure 4. The effect of pectin reinforcement on the hardness of the composites

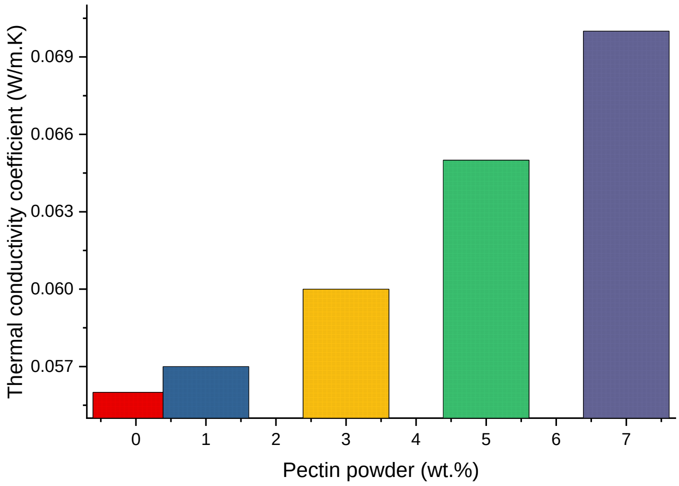

Figure 5. The effect of pectin reinforcement on the thermal conductivity of the composites

Table 2 provides activation energies calculated from thermal decomposition experiments of the polyester compositesIt has been determined that the activation energy of the produced composite decreases slightly as the pectin powder ratio in the mixture increases. Coats Redfern method has been used to calculate activation energy (*Ea*). The highest coefficient of determination is calculated by the three-dimensional diffusion equation. Activation energies of pectin reinforced the composites at a certain conversion ratio (range of 0.20 to 0.80) found. Thermal decomposition experiments have been carried out from 20 °C to 600 °C at a heating rate of 10 °C/min [34,35].

Table 2. Activation energies of the polyester composites

Filler ratios (wt.%)	Activation energy (*Ea*: kJ/mol)
0	121.56
1	118.79
3	115.68
5	112.93
7	109.85

In this study, 5 wt.% and higher pectin powder reinforcement negatively affect the surface morphology of the polyester composite. Figure 6 shows a scanning electron microscope (SEM) image of the pure polyester polymer. When pectin powder is reinforced as filler at optimum ratios (3 wt.%), both the pore structure and thermophysical properties of the polyester composite are not negatively affected.

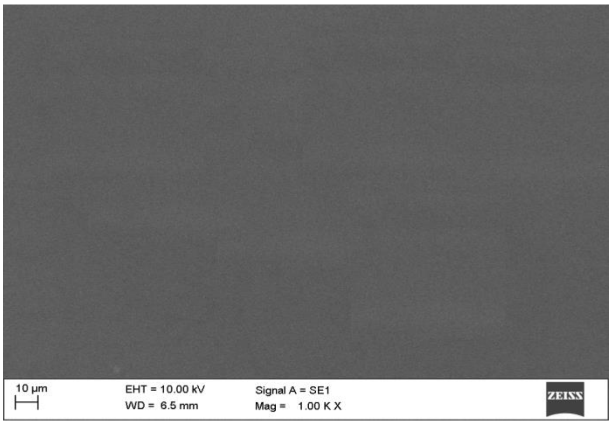

Figure 6. SEM image of pure polyester polymer

4. Conclusions and Recommendations

In this study, pectin powder is used as a reinforcing agent to produce an environmentally friendly polyester composite with low density and is easy to process. The use of such fillers improves some thermophysical properties of the composite. According to the results obtained, pectin supplementation slightly reduces the density and Shore D hardness of the polyester composite. However, a slight increase in the thermal conductivity coefficient of the composites occurs with the filler reinforcement. Reinforcing the pectin powder into the composite reduces the use of petrochemical raw materials and encourages the production of economical, easy-to-process, and environmentally friendly biocomposites.

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