

PAPER DETAILS

TITLE: Obtaining Pectin Reinforced Polyester Composite and Investigation of Thermophysical Properties

AUTHORS: Mukaddes KARATAS,Ercan AYDOGMUS

PAGES: 64-66

ORIGINAL PDF URL: <https://dergipark.org.tr/tr/download/article-file/2964426>



Obtaining Pectin Reinforced Polyester Composite and Investigation of Thermophysical Properties

Mukaddes Karataş¹ and Ercan Aydoğmuş^{1*}

¹Firat University, Faculty of Engineering, Department of Chemical Engineering, Elazığ, Türkiye, (ORCID: 0000-0001-5803-6821), mkozturk@firat.edu.tr

^{1*}Firat University, Faculty of Engineering, Department of Chemical Engineering, Elazığ, Türkiye, (ORCID: 0000-0002-1643-2487), ercanaydogmus@firat.edu.tr

(1st International Conference on Frontiers in Academic Research ICFAR 2023, February 18-21, 2023)

(DOI: 10.31590/ejosat.1254020)

ATIF/REFERENCE: Karataş, M., & Aydoğmuş, E. (2023). Obtaining Pectin Reinforced Polyester Composite and Investigation of Thermophysical Properties. *European Journal of Science and Technology*, (48), 40-44.

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 ısı iletkenlik katsayısının arttığı gözlemlenmiştir. Kompozitin ısı 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 corn cob, *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).

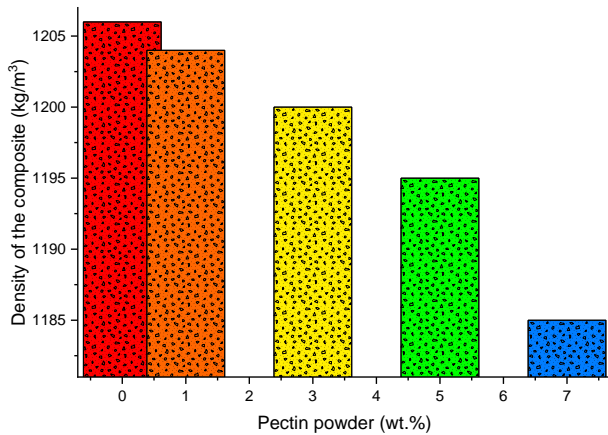


Figure 3. The effect of pectin reinforcement on the density of the composite

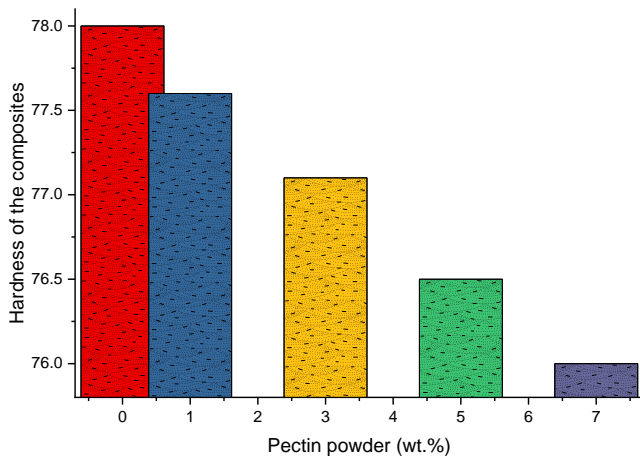


Figure 4. The effect of pectin reinforcement on the hardness of the composites

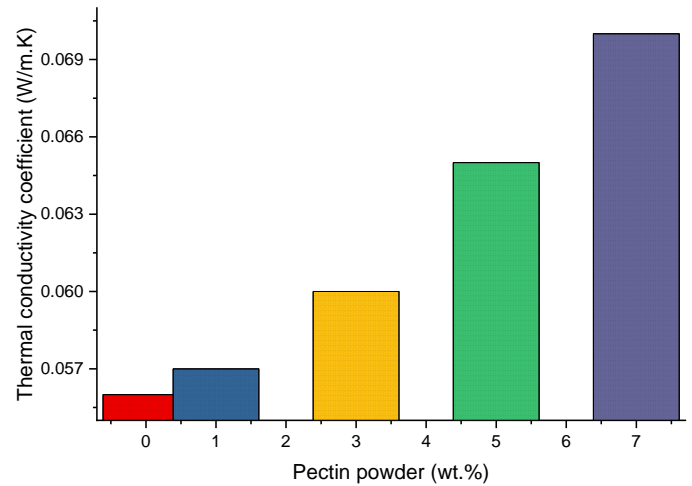


Figure 5. The effect of pectin reinforcement on the thermal conductivity of the composites

Table 2 provides activation energies calculated from thermal decomposition experiments of the polyester composites. It 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 (E_a). 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 (E_a : 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.

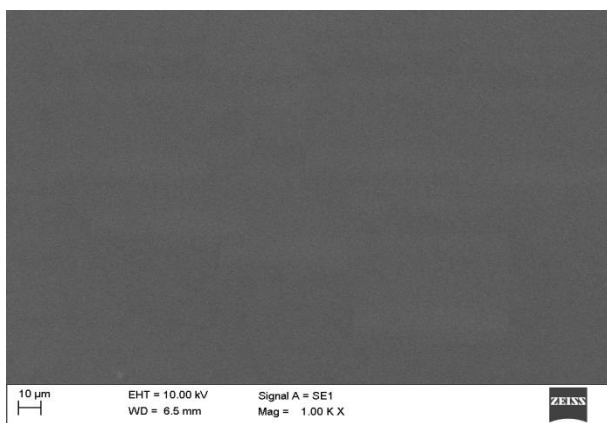


Figure 6. SEM image of pure polyester polymer

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.

References

- [1] Shibata, M., Takachiyo, K.I., Ozawa, K., Yosomiya, R., and Takeishi, H. Biodegradable polyester composites reinforced with short abaca fiber, *J Appl Polym Sci*, 85, 129–138, 2002.
- [2] Jagadeesh, P., Puttegowda, M., Girijappa, Y.G.T., Rangappa, S.M., and Siengchin S. Effect of natural filler materials on fiber reinforced hybrid polymer composites: An Overview, *Journal of Natural Fibers*, 19(11), 4132-4147, 2022.
- [3] Bagherpour, S. Fibre Reinforced Polyester Composites, *Intech, Rijeka, Croatia*, 56-385, 2012.
- [4] Nesic, A., Meseldzija, S., Cabrera-Barjas, G., and Onjia, A. Novel Biocomposite Films Based on High Methoxyl Pectin Reinforced with Zeolite Y for Food Packaging Applications, *Foods*, 11, 360, 2022.
- [5] Valdés, A., Burgos, N., Jiménez, A., and Garrigós, M.C. Natural Pectin Polysaccharides as Edible Coatings, *Coatings*, 5(4), 865-886, 2015.
- [6] Mohnen, D. Pectin structure and biosynthesis, *Current Opinion in Plant Biology*, 11(3), 266-277, 2008.
- [7] Lara-Espinoza, C., Carvajal-Millán, E., Balandrán-Quintana, R., López-Franco, Y., and Rascón-Chu, A. Pectin and Pectin-Based Composite Materials: Beyond Food Texture, *Molecules*, 23(4), 942, 2018.
- [8] Sriamornsak, P. Application of pectin in oral drug delivery, *Expert Opinion on Drug Delivery*, 8(8), 1009-1023, 2011.
- [9] Marangoni Júnior, L., Fozzatti, C.R., Jamróz, E., Vieira R.P., and Alves, R.M.V. Biopolymer-Based Films from Sodium Alginate and Citrus Pectin Reinforced with SiO₂, *Materials*, 15(11), 3881, 2022.
- [10] Picot-Allain, M.C.N., Ramasawmy, B., and Emmambux, M.N. Extraction, Characterisation, and Application of Pectin from Tropical and Sub-Tropical Fruits: A Review, *Food Reviews International*, 38(3), 282-312, 2022.
- [11] Pothan, L.A., Oommen, Z., and Thomas, S. Dynamic mechanical analysis of banana fiber reinforced polyester composites, *Compos Sci Technol*, 63, 283–293, 2003.
- [12] Baiardo, M., Zini, E., and Scandola, M. Flax fibre–polyester composites, *Composites Part A: Applied Science and Manufacturing*, 35(6), 703-710, 2004.
- [13] Monteiro, S.N., Terrones, L.A.H., and D’Almeida, J.R.M. Mechanical performance of coir fiber/polyester composites, *Polymer Testing*, 27(5), 591-595, 2008.
- [14] Senthilkumar, K., Rajini, N., Saba, N., and et al. Effect of Alkali Treatment on Mechanical and Morphological Properties of Pineapple Leaf Fibre/Polyester Composites, *J Polym Environ*, 27, 1191–1201, 2019.
- [15] Dağ, M., Yanen, C., and Aydoğmuş, E. Effect of Boron Factory Components on Thermophysical Properties of Epoxy Composite, *European Journal of Science and Technology*, 36, 151–154, 2022.
- [16] Orhan, R., Aydoğmuş, E., Topuz, S., and Arslanoğlu, H. Investigation of thermo-mechanical characteristics of borax reinforced polyester composites, *Journal of Building Engineering*, 42, 103051, 2021.
- [17] Yanen, C., Dağ, M., and Aydoğmuş, E. Investigation of Thermophysical Properties of Colemanite, Ulexite, and Tincal Reinforced Polyester Composites, *European Journal of Science and Technology*, 36, 155–159, 2022.
- [18] Yilmaz, E., Aydoğmuş, E., and Demir, A. Life Cycle Assessment and Characterization of Tincal Ore Reinforced Polyester and Vinylester Composites, *Journal of the Turkish Chemical Society Section B: Chemical Engineering*, 5(2), 183-194, 2022.
- [19] Aydoğmuş, E., and Arslanoğlu, H. Kinetics of thermal decomposition of the polyester nanocomposites, *Petroleum Science and Technology*, 39(13–14), 484–500, 2021.
- [20] Şahal, H., and Aydoğmuş, E. Investigation of Thermophysical Properties of Polyester Composites Produced with Synthesized MSG and Nano-Alumina, *European Journal of Science and Technology*, 34, 95-99, 2022.
- [21] Şahal, H., Aydoğmuş, E., and Arslanoğlu, H. Investigation of thermophysical properties of synthesized SA and nano-alumina reinforced polyester composites, *Petroleum Science and Technology*, 1–17, 2022.
- [22] Pekdemir, M. E., Aydoğmuş, E., and Arslanoğlu, H. (2023). Thermal decomposition kinetics of synthesized poly(N-isopropylacrylamide) and Fe₃O₄ coated nanocomposite: Evaluation of calculated activation energy by RSM. *Petroleum Science and Technology*, 1-19, 2023.
- [23] Aydoğmuş, E., Aydın, M., and Arslanoğlu, H. Production and characterization of microsphere reinforced polyester composite: Modeling of thermal decomposition with ANN and optimization studies by RSM. *Petroleum Science and Technology*, 1-17, 2022.
- [24] Yanen, C., and Aydoğmuş, E. Characterization of Thermo-Physical Properties of Nanoparticle Reinforced the Polyester Nanocomposite, *Dicle University Journal of the Institute of Natural and Applied Science*, 10(2), 121–132, 2021.

- [25]Aydoğmuş, E., Arslanoğlu, H., and Dağ, M. Production of waste polyethylene terephthalate reinforced biocomposite with RSM design and evaluation of thermophysical properties by ANN, Journal of Building Engineering, 44, 103337, 2021.
- [26]Aydoğmuş, E., Dağ, M., Yalçın, Z. G., and Arslanoğlu, H. Synthesis and characterization of EPS reinforced modified castor oil-based epoxy biocomposite, Journal of Building Engineering, 47, 103897, 2022.
- [27]Demirel, M. H., and Aydoğmuş, E. Production and Characterization of Waste Mask Reinforced Polyester Composite, Journal of Inonu University Health Services Vocational School. 10(1), 41-49, 2022.
- [28]Demirel, M. H., and Aydoğmuş, E. Waste Polyurethane Reinforced Polyester Composite, Production and Characterization, Journal of the Turkish Chemical Society Section A: Chemistry, 9(1), 443–452, 2022.
- [29]Aydoğmuş, E. Biohybrid nanocomposite production and characterization by RSM investigation of thermal decomposition kinetics with ANN, Biomass Conversion and Biorefinery, 12, 4799-4816, 2022.
- [30]Şahal, H., and Aydoğmuş, E. Production and Characterization of Palm Oil Based Epoxy Biocomposite by RSM Design, Hittite Journal of Science and Engineering, 8(4), 287-297, 2021.
- [31]Aydoğmuş, E., Dağ, M., Yalçın, Z. G., and Arslanoğlu, H. Synthesis and characterization of waste polyethylene reinforced modified castor oil-based polyester biocomposite, Journal of Applied Polymer Science, 139, e525256, 2022.
- [32]Orhan, R., and Aydoğmuş, E. Investigation of some thermophysical properties of *Asphodelus aestivus* reinforced polyester composite, Firat University Journal of Experimental and Computational Engineering 1(3), 103-109, 2022.
- [33]Orhan, R., and Aydoğmuş, E. Production and Characterization of Waste Corncob Reinforced Polyester Composite, European Journal of Science and Technology Special Issue. 42, 176-179, 2022.
- [34]Buran, A., Durğun, M. E., and Aydoğmuş, E. *Cornus alba* Reinforced Polyester-Epoxy Hybrid Composite Production and Characterization, European Journal of Science and Technology, 43, 99-103, 2022.
- [35]Buran, A., Durğun, M. E., Aydoğmuş, E., and Arslanoğlu, H. Determination of thermophysical properties of *Ficus elastica* leaves reinforced epoxy composite, Firat University Journal of Experimental and Computational Engineering, 2(1), 12-22, 2023.