



Production of SBS Reinforced Polyester Composite: Characterization of Physical and Chemical Properties

Hakan Şahal¹ , Ercan Aydoğmuş^{2*} 

¹ Munzur University, Vocational School of Tunceli, Department of Food Processing, Tunceli, Türkiye.

² Fırat University, Faculty of Engineering, Department of Chemical Engineering, Elazığ, Türkiye.

Abstract: In this study, dissolved styrene butadiene styrene (SBS) copolymer is homogeneously reinforced into orthophthalic unsaturated polyester (UP) resin. Polyester composite production is carried out with the help of methyl ethyl ketone peroxide (MEKP) and cobalt octoate (Co Oc) catalysts. The density, Shore D hardness, thermal conductivity coefficient, thermal stability, morphological surface structure, and chemical bond structure of the obtained composite have been examined. According to the results, SBS reinforcement decreases the density of the composite and increases the thermal conductivity coefficient. The addition of SBS at different weight ratios (1%, 3%, 5%, 7%, and 10% w/w) reduces both the hardness and thermal stability of the polyester composite. According to the test and analysis results, 5 wt.% SBS reinforced polyester composite production is determined as the optimum ratio. 7 wt.% and above SBS reinforcement negatively affect the physical and chemical properties of the obtained composite. For example, when 10 wt.% SBS reinforced composite is examined by scanning electron microscope (SEM), and irregular pores are observed in the surface morphology. Also, it is understood by Fourier transform infrared spectroscopy (FTIR) that there is a physical interaction between SBS and polyester and that no chemical bond is formed. The thermal decomposition behavior of the composite has been determined according to the decrease in the activation energy. As SBS ratio increases, it is understood that the thermal stability of the product obtained with the decrease in the activation energy of the polyester composite weakens.

Keywords: Activation energy, density, hardness, polyester composite, SBS, thermal conductivity.

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***Corresponding author's E-mail:** ercanaydogmus@firat.edu.tr

1. INTRODUCTION

Today, composites have become widespread as an alternative to traditional products. Particularly, the properties of doped polymer composites such as insulation, thermal stability, density, hardness, and porosity have led to an increase in studies in this field. Polymer composites are widely used in many industrial applications such as automotive, aerospace, sports, household appliances, medicine, electrical-electronics, and defense (1,2). The desire to produce lighter and more efficient materials has led researchers to investigate various composite materials and their applications (3). The high density and electrical conductivity, in addition to the low corrosion and contamination resistance of conventional metals or metal-doped components, have prompted researchers to explore alternative materials for thermal applications (4).

Thermoplastic elastomers (TPEs) are an important class of materials that combine elastomeric behavior with thermoplastic properties. Usually, these are ABA-type triblock copolymers such as polystyrene-polybutadiene-polystyrene (SBS) or polystyrene-polyisoprene-polystyrene (SIS) combining a soft central block with glassy end blocks (5,6). Although these materials find widespread commercial applications, unsaturated double bonds in the middle segments make the structure susceptible to oxidation, shortening its useful life (7). These TPEs have some drawbacks such as poor chemical, heat, and UV resistance due to the unsaturated soft blocks as well as low service temperature limited by the glass transition temperature of the polystyrene block (8).

SBS, which is in the thermoplastic elastomer group, consists of hard styrene blocks and softer butadiene blocks, which are widely used in commercial applications due to their mechanical strength. Polybutadiene (PB) is the main component in the SBS block copolymer and forms the continuous matrix, while polystyrene (PS) forms the discontinuous phase (domains). Due to the rubbery character of SBS, high-performance self-supported hollow fibers are difficult to prepare by phase inversion. Therefore, membrane configurations can be obtained only by coating SBS on existing hollow fiber supports or by blending SBS with a glassy polymer in appropriate concentrations (9,10).

For this reason, there are studies developed by adding many physical and chemical additives according to the purpose of the use of polymers in the literature. Many organic or inorganic reinforcing materials are used in composite construction with unsaturated polyesters. In a study, it has been reported that improvements are observed in both the mechanical and thermal properties of the composite obtained with waste crumb rubber added to unsaturated polyester (11). Thermal decomposition kinetics of thermoplastic polyesters and optimized polypropylene/poly(lactic acid) mixture and thermal degradation kinetics of fumed silica reinforced polyester composites were conducted (12,13).

In studies using synthetic materials, the response of a polyester matrix composite with the addition of marble waste as filler under vacuum and vibro-compression was investigated (14). In another research, the thermal, mechanical, and morphological properties of synthetic graphite and carbon fiber-filled polybutylene terephthalate polyester composites were investigated (15).

In this study, the production and some physical and chemical properties of SBS reinforced polyester composites have been investigated. It is aimed to use SBS, which has thermoplastic properties, as a reinforcement material in the production of polyester composites. The use of certain proportions of SBS in polyester reduces the density and hardness of the obtained composite, facilitates its workability, and increases its elastic properties. Another unique aspect of this study is that since SBS is mixed with unsaturated polyester as a dissolved gel, not as a powder, a more homogeneous composite matrix is obtained. SBS reinforcement is made to improve the thermoplastic properties of polyester composites. In this way, it

can be processed easily and becomes more flexible than brittle structure. Also, this study will be an example for the development of composite materials with low density and low stiffness.

2. MATERIALS AND METHODS

2.1. Methods Used in the Experimental Study

Methyl ethyl ketone peroxide (MEKP), cobalt octoate (Co Oc), and orthophthalic unsaturated polyester resin (UP) used in experimental studies were supplied from Turkuaz Polyester Company (Türkiye). Also, chemicals used for the synthesis and analysis were purchased from the following: Toluene (Sigma-Aldrich), and SBS-Kumho KTR 101 (Kumho Petrochemical).

2.2. Methods Used in the Experimental Study

The matrix density of the samples obtained is calculated by proportioning the weight of the composites to the volume since they have a uniform geometry in standard molds. Shore D hardness is measured with the LX-D-2 double-needle durometer. Thermal conductivity coefficient measurement was made with TLS-100 Thermtest device. Chemical bond structures of polyester polymer are examined with Fourier transform infrared spectrometer (FTIR). A Shimadzu IRSpirit (QATR-S) branded device is used for FTIR spectral measurements. Thermal decomposition experiments are performed in an isolated PID-controlled reactor system. The thermal degradation behavior of the samples was studied at a heating rate of 10 °C/min from about room temperature to 600 °C in an inert environment. Besides, the surface morphology of the produced polyester composite was examined with Zeiss Evo/MA10 SEM device (16-18). SBS block copolymer used as a supplement in our study was dissolved by using 20 mL of toluene for each 10 g to gel it. The resulting solution was allowed to remove from the toluene in a vacuum oven for 4 hours. Afterward, a homogeneous mixture was obtained by mixing orthophthalic unsaturated polyester (UP) resin with SBS at room temperature. Experimental studies were carried out at approximately 25 °C and atmospheric pressure. At the last stage, MEKP and Co Oc were added to the mixture and the gelling mixture was poured into standard molds. After waiting for 24 hours for curing, the necessary physical tests and chemical analyses were carried out (22-25). In Figure 1, a brief schematic of polyester composite production is given.

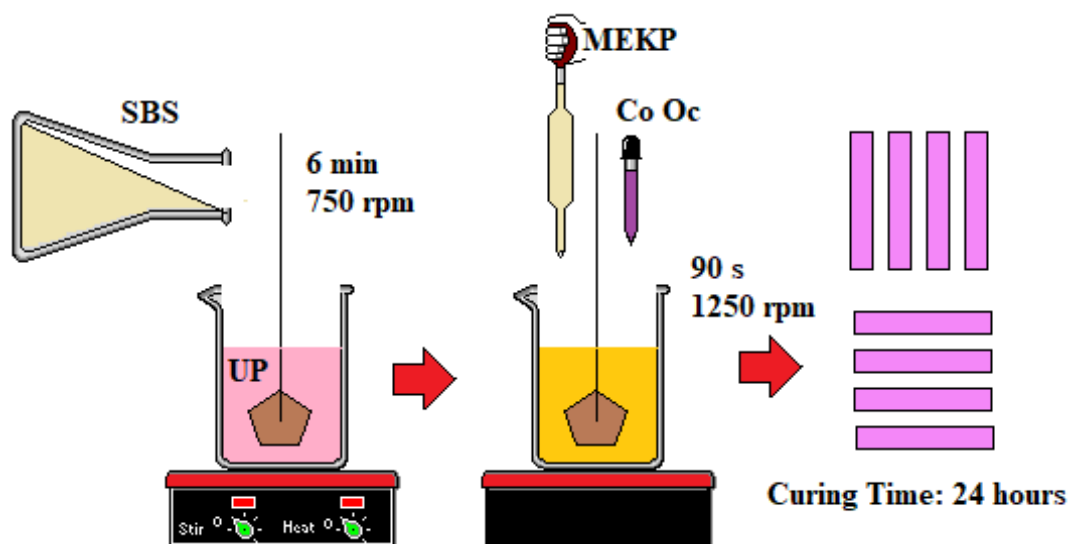


Figure 1: Production scheme of SBS reinforced polyester composite.

The experimental study plan and chemical composition ratios are given in Table 1. In this table, it is seen that polyester composites are produced by

keeping Co Oc and MEKP ratios constant and SBS reinforcement at different weight ratios.

Table 1: Components and quantities used to obtain composites.

Experiment No	SBS (wt.%)	UP (wt.%)	Co Oc (wt.%)	MEKP (wt.%)
1	0	98	0.6	1.4
2	1	97	0.6	1.4
3	3	95	0.6	1.4
4	5	93	0.6	1.4
5	7	91	0.6	1.4
6	10	88	0.6	1.4

3. RESULTS AND DISCUSSIONS

3.1. The Density of SBS Reinforced Polyester Composite

In this research, some physical and chemical properties of SBS reinforced polyester composite have been evaluated. It is seen in Figure 2 that the

density of the produced composite decreases with SBS reinforcement. While the density of pure polyester polymer is approximately 1206 kg/m³, the density of 10 wt.% SBS reinforced polyester composites decrease to 1174 kg/m³.

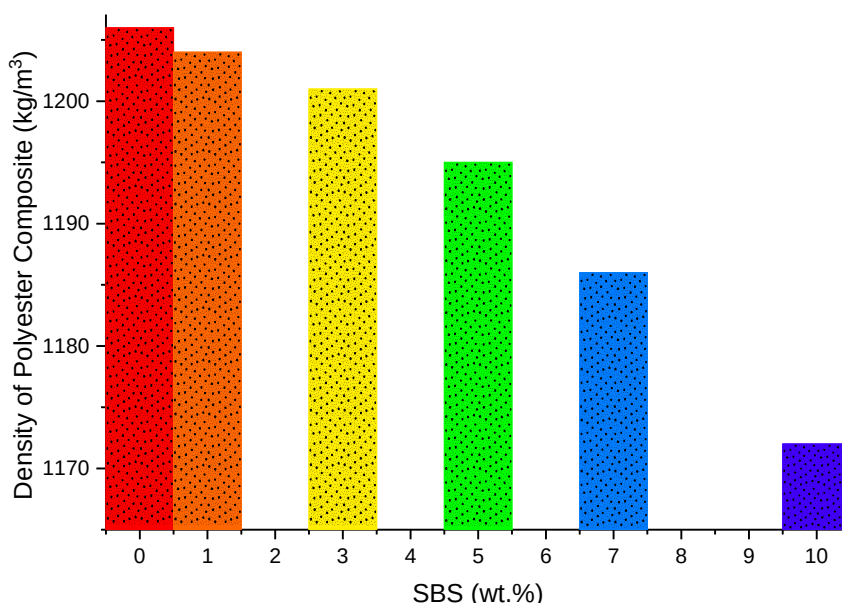


Figure 2: Effect of SBS reinforcement on the density of polyester composite.

3.2. The Hardness of SBS Reinforced the Composite

In Figure 3, it is stated that SBS reinforcement reduces the density of the polyester composite. While inorganic fillers generally increase the surface

hardness of polyester composites, thermoplastic polymers and wastes can reduce it (28-31). Shore D hardness of pure polyester polymer is around 78 and this value decreases to about 71 with 10 wt.% SBS reinforcement.

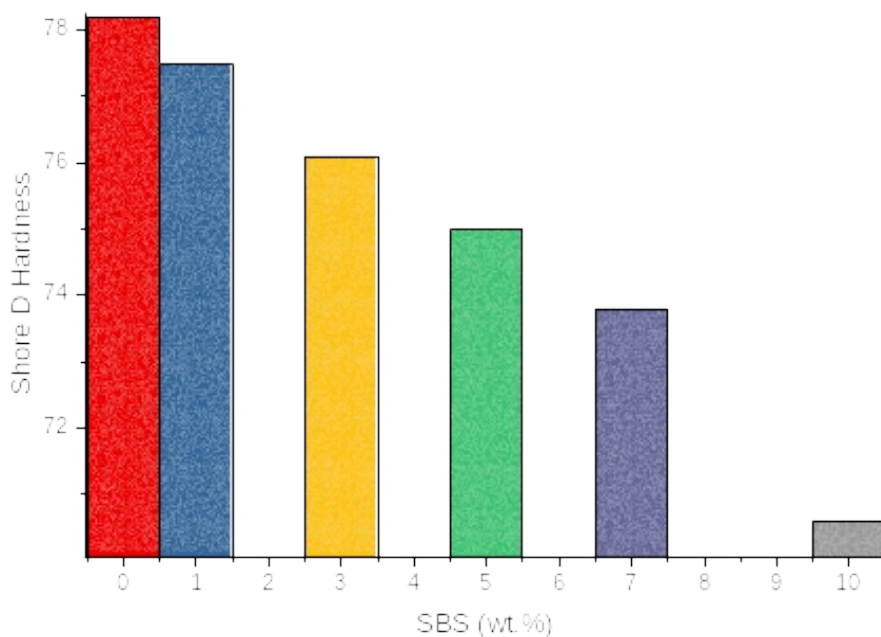


Figure 3: Effect of SBS reinforcement on Shore D hardness of the composite.

3.3. The Thermal Conductivity of SBS Reinforced the Polyester Composite

It is seen in Figure 4 that the addition of SBS slightly increases the thermal conductivity coefficient of the polyester composite. While the thermal conductivity

coefficient of pure polyester polymer is around 0.056 W/m·K, this coefficient increases up to approximately 0.062 W/m·K with 10 wt.% SBS supplementation.

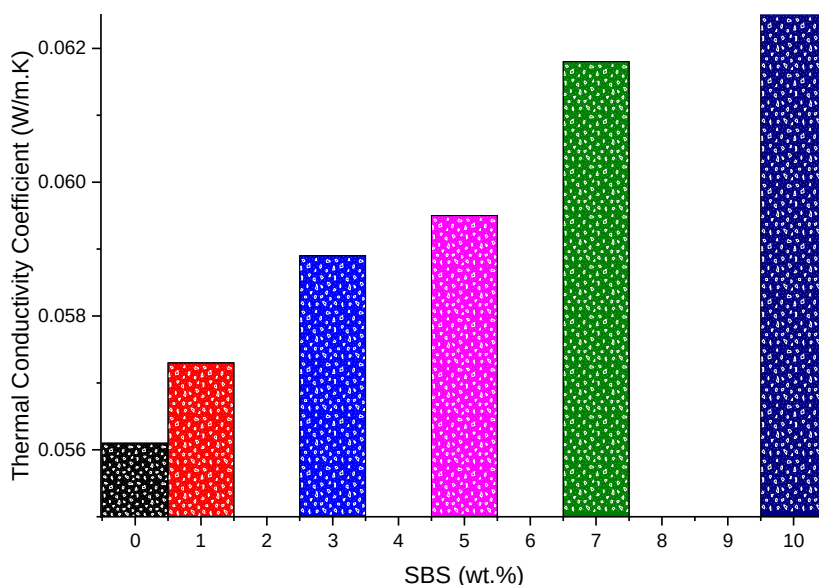


Figure 4: Effect of SBS reinforcement on thermal conductivity of composite.

3.4. The Activation Energy of SBS Reinforced the Composite

The thermal decomposition behavior of the composites was carried out in an inert environment (nitrogen gas) with a temperature increase rate of 10 °C/min from 25 °C to 600 °C. Activation energies

of composites were calculated by Coats-Redfern using data from thermal degradation experiments (26,27). In this method, calculations were made by choosing the function (three-dimensional diffusion) with the highest correlation ($R^2 \geq 0.9830$) coefficient. In Figure 5, it is determined that the addition of SBS

reduces the activation energy of the polyester composite.

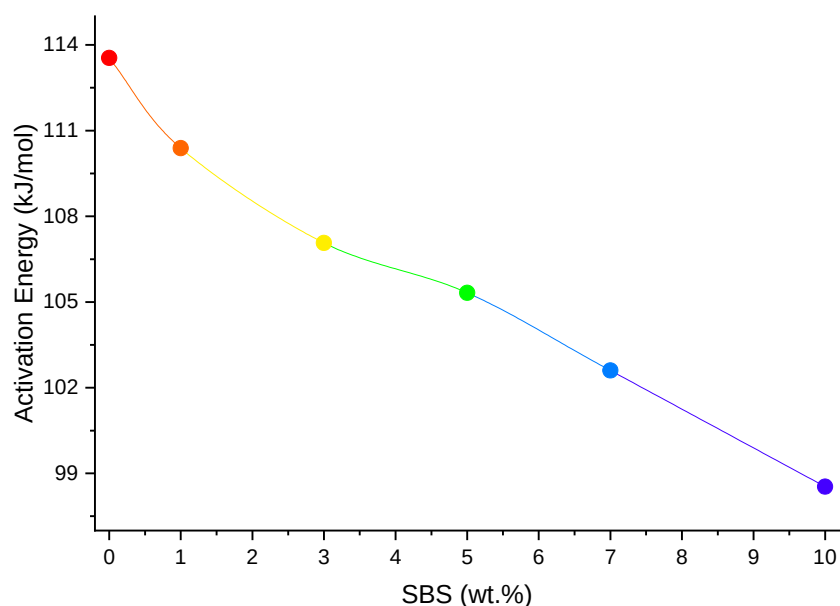


Figure 5: Effect of SBS reinforcement on the activation energy of the composite.

3.5. FTIR Spectra of the Polyester Composite

Figure 6 shows the FTIR spectrum of the pure polyester polymer. The band seen at a wavelength of about $3450\text{--}3500\text{ cm}^{-1}$ expresses the stretching vibrations of the hydroxyl groups. The bands in the wavelength range of $2800\text{--}2950\text{ cm}^{-1}$ show the stretching vibrations of the CH groups. The vibrations of the carbonyl group in the polyester composite are evident at a wavelength of 1718 cm^{-1} . When the peaks of SBS are examined, C=C stretching vibrations at a wavelength of

approximately 1640 cm^{-1} are striking. Besides, cis HC=, vinyl $\text{H}_2\text{C=}$, and trans HC= deformation peaks are observed at approximately 700 , 910 , and 960 cm^{-1} wavelengths. In the wavelength range of 1400 and 1460 cm^{-1} , it expresses the vibrations of the aromatic ring. Besides, the band seen at a wavelength of 1255 cm^{-1} represents the torsional vibration of CH_2 groups (19-21). There is no chemical bond between SBS and polyester polymer and only a physical interaction can be said by looking at Figure 7.

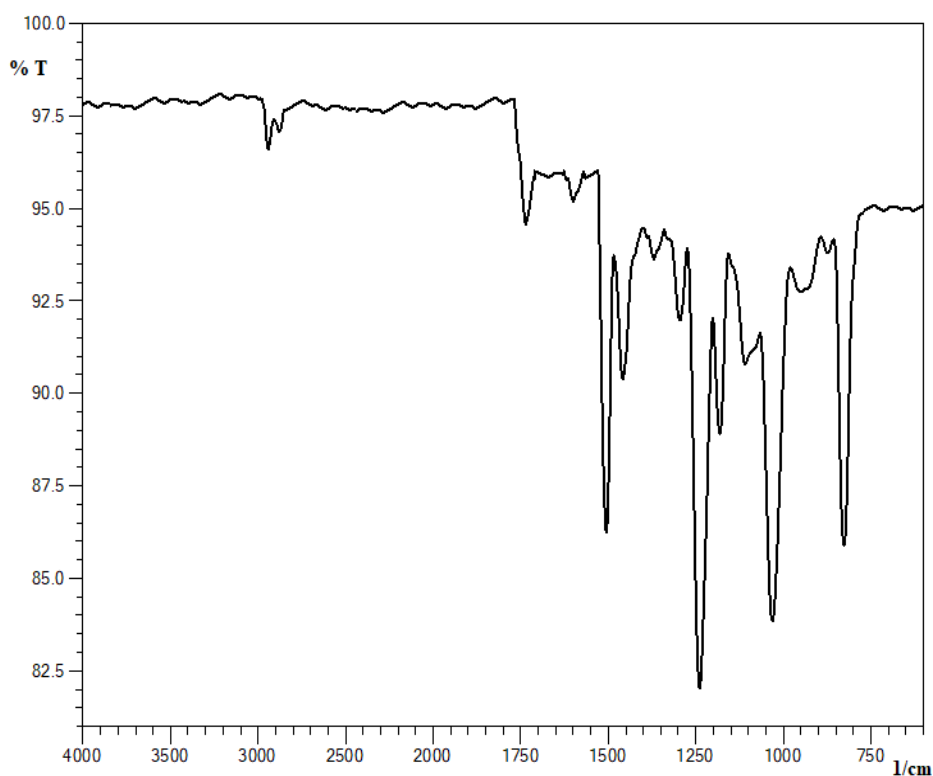


Figure 6: FTIR spectrum of pure polyester polymer.

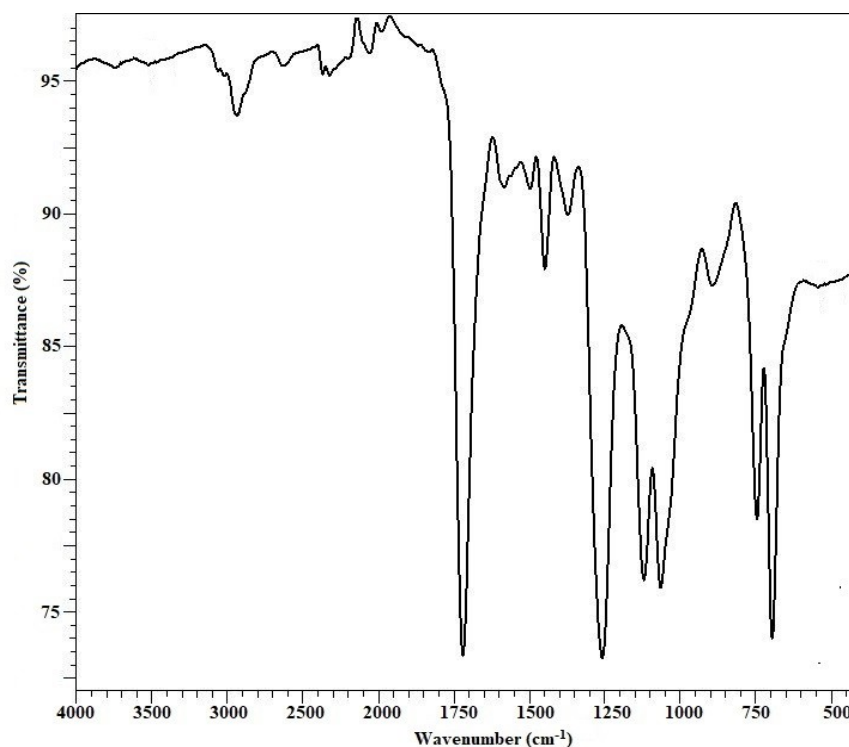


Figure 7: FTIR spectrum of 5 wt.% SBS reinforced polyester composite.

3.6. SEM Image of the Polyester Composite

Figure 8 shows the surface image of the pure polyester polymer. As can be understood from the surface morphology, there is no complex formation in the pore structure and distribution. In Figure 9,

the surface structure of 10 wt.% SBS reinforced polyester composite is negatively affected due to excessive use of filler. According to the results, the use of filler below 7 wt.% is important for both a homogeneous and smoother surface morphology.

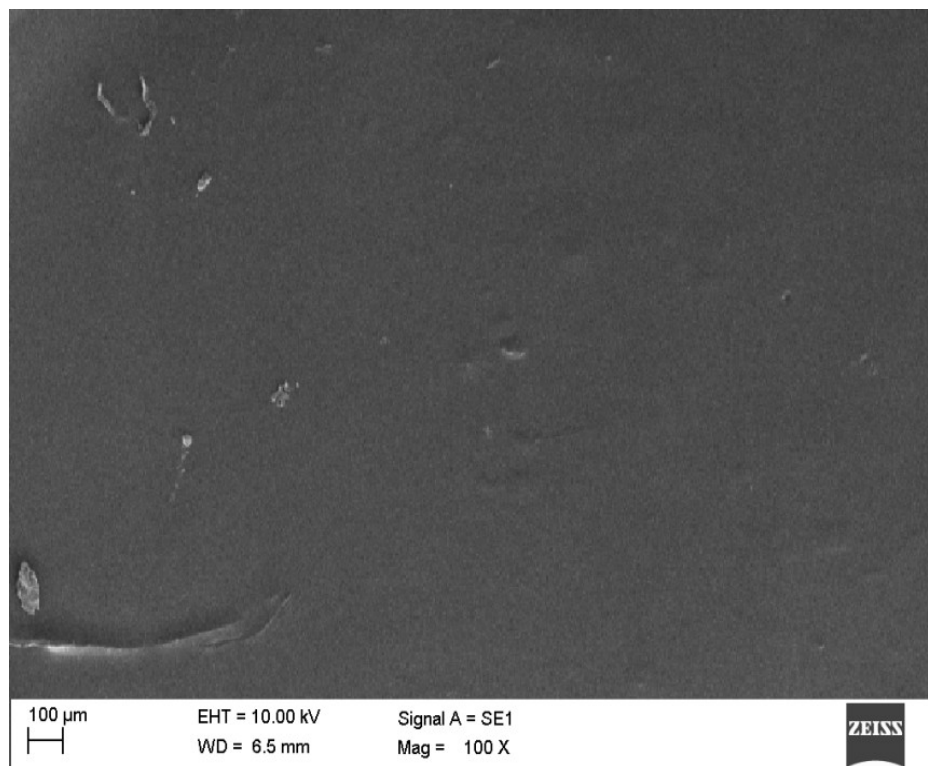


Figure 8: SEM image of pure polyester polymer.

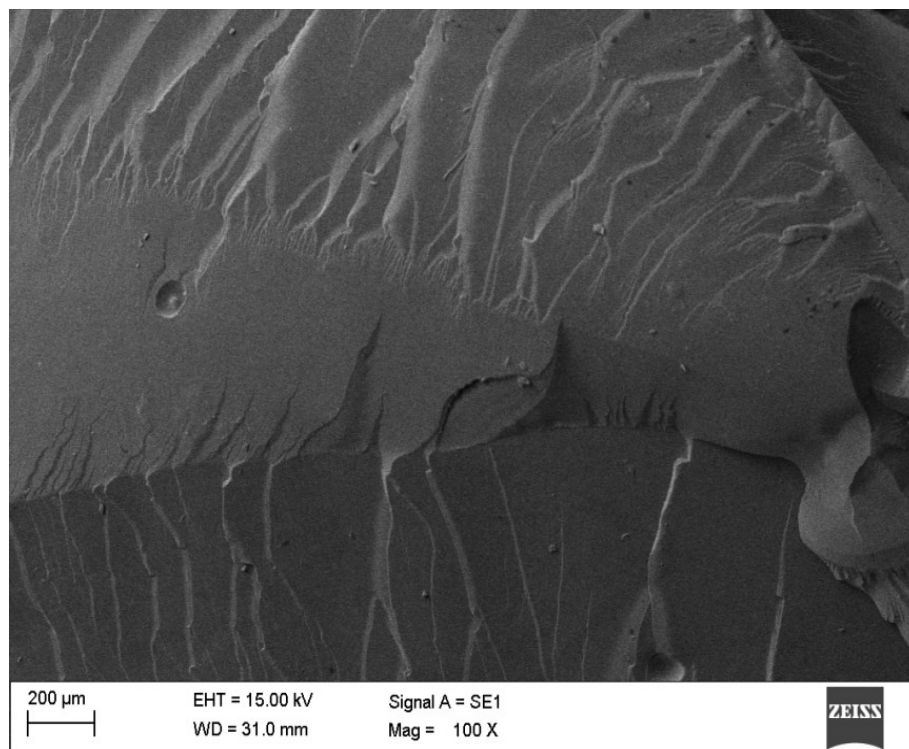


Figure 9: SEM image of 10 wt.% SBS reinforced polyester composite.

4. CONCLUSIONS

In this research, easy-to-process, flexible, low hardness and density SBS reinforced polyester composites have been produced. To provide a homogeneous mixture, SBS is mixed into UP in gel form. According to the results obtained, SBS reinforcement reduces both the density and hardness of the polyester composite. Besides, SBS slightly increases the thermal conductivity coefficient of the composite, while decreasing its thermal stability slightly. As SBS reinforcement ratio increases, the calculated activation energy decreases, indicating that the thermal stability of the composite also decreases. When the chemical bond structure of SBS reinforced composite is examined in FTIR spectrums, polyester polymer formation can be seen. Here, it is understood that the addition of SBS does not make a chemical bond with which it interacts physically in the composite. Also, when the surface morphology of the obtained polyester structures is examined, SBS reinforcement of 7 wt.% and above is not recommended for composite production.

As a result, it has been determined as the optimum ratio in the production of 5 wt.% SBS reinforced polyester composite. At this ratio, the homogeneity, surface structure, flexibility, workability, physical properties, and thermoplastic behaviors of the composite have been improved. Therefore, both the type and amount of filler are effective to produce polyester composites at the desired standards according to the intended use (32-35).

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