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The effects of silane coupling agents on the mechanical properties of basalt fiber reinforced poly(butylene terephthalate) composites

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ABSTRACT

In the current study, the effects of three different silane coupling agents, namely (3-aminopropyl) triethoxysilane (AP), (3-Glycidyloxypropyl) trimethoxysilane (GP) and (3-trimethoxysilyl) propylmethacrylate (MA) are investigated on the mechanical properties of the basalt fiber (BF) reinforced poly (butyleneterefthalate) (PBT) composites. The tensile, flexural, impact, thermomechanical and morphological properties of the composites are investigated. According to the test results, the remarkable increase in tensile strenght and elastic modulus is observed, whereas slight improvement in flexural strenght and no change in impact properties is observed. According to flexural strength and elastic modulus values, the effectiveness of the silane coupling agents can be ranked as follow: GP > AP > MA. It is clearly shown that the increase in mechanical properties arises from improvement in interfacial adhesion between BF and PBT. It is concluded that the covalent bond formation causes the highest improvement in mechanical properties including tensile and flexural strenght and elastic modulus.

1. Introduction

Poly (butyleneterephthalate) (PBT), a kind of polyester based thermoplastic engineering polymer, has great importance owing to the exclusive features including high thermal resistance, excellent electrical insulation, easy processability and good surface apperance. These properties provide wide applications in automative, electrical and packing applications [1,2]. In the literature, PBT is reinforced with various chopped fibers including carbon [3–5], glass [6–14] and natural fibers [15] for improving its mechanical performance. As seen from the literature, the glass fiber is the most widely used reinforcing material.

Basalt fiber (BF), known as non-pollution green material of 20th century, is produced from volcanic basalt rocks [16]. It has superior properties such as high tenacity, high fire performance, heat and sound insulation, alkaline resistance and dimensional stability than E-glass fiber [17–21]. These exclusive characteristics provide widely use in such areas including aviation, civil application, defense industry and agriculture. BF is seemed to be the best alternative of E-glass fiber in terms of cost and mechanical properties. In the literature, BF in various form is used as reinforcing material in various polymers [20–34].

Glass fiber and BF have hydroxyl groups on their surface. Accordingly, the modification with silane coupling agents is the most preferred method for improving the dispersion and the interfacial adhesion between polymer and the fiber [6,8,13,22–30,35,36]. In the literature, different silane coupling agents are used in glass fiber reinforced PBT composites [6,8,13,36]. Ishak et al. investigated the mechanical properties of (3-Aminopropyl) triethoxysilane (AP) modified glass fiber reinforced PBT composites manufactured by injection molding technique. It was concluded that the used silane coupling agent improved the tensile properties of the composites significantly [6]. Bergeret et al. examined the mechanical and the interphase properties of the AP modified glass fiber reinforced PBT composites. The use of silane coupling agent improved the interfacial adhesion and increased the impact strength of the composites [8]. Yang et al. synthesized a novel phosphorus-containing coupling agent. They investigated the effect of synthesized coupling agent on the mechanical and flame retardant properties of the glass fiber reinforced PBT composites. It was found that the use of synthesized silane coupling agent significantly enhanced the fire retardant properties and improved the tensile properties [13]. Zhou et al. studied the mechanical properties of the (3-Glycidyloxypropyl) trimethoxysilane (GP) modified long glass fiber reinforced PBT composites. The use of the silane coupling agent improved the tensile, flexural and impact properties of the composites [36].

In the literature, only two studies that investigated the mechanical performance of the BF reinforced PBT composites were found. Baets

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et al. investigated the effect of the non-isothermal process conditions on the crystallization characteristics and the mechanical properties of basalt roving and woven basalt fabric reinforced PBT composites [37]. Hao et al. examined the effects of the various process temperatures on the mechanical properties of the woven basalt fabric reinforced PBT composites [38]. In these studies, BF is either roving or woven fabric form and there is no compatibilizer or coupling agent is used. To our best knowledge, this is the first study that investigates the mechanical performances of the chopped BF reinforced PBT composites. In the current study, the effects of three different silane coupling agents on the mechanical and thermo-mechanical properties of the chopped BF reinforced PBT composites. The properties of the composites are characterized by tensile, flexural, impact tests and dynamic mechanical analysis (DMA). The tensile and impact fractured surfaces of the composites are also inspected by scanning electron microscopy (SEM) analysis.

2. Experimental

2.1. Materials

PBT under the trade name of Tecodur® PB70 NL 1.31 g/cm^3, was purchased from Interplast. Chopped BF with length and diameter of 6 mm and 13–20 μm was supplied from Tila Kompozit. The specifications of the raw materials given by the suppliers are given in Table 1. Three kinds of silane coupling agents, (3-Aminopropyl) triethoxysilane (AP) (0.946 g/cm^3 and 221.37 g/mol), (3-Glycidyloxypropyl) trimethoxysilane (GP) (1.07 g/cm^{3}and236.34 g/mol) and 3-(Trimethoxysilyl) propylmethacrylate (MA) (1.045 g/cm^{3}and 248.35 g/mol) were purchased from Sigma Aldrich. The chemical structures of the silane coupling agents are given in Fig. 1.

2.2. Silane treatment of basalt fiber

In order to remove the coated sizing on the fiber surface applied by the producer, BF was heated in a furnace at 500 °C for an hour prior to the silane treatment process. The silane treatment of the BF (100 g) was made with 5 wt% three different silanes in ethanol/water (1:1 by weight) mixture at 85 °C for 5 h. After the silane treatment, BF was washed with ethanol several times in order to remove unreacted silane coupling agent. The characterization of the fiber samples were made with attenuated total reflectance – Fourier transform infrared spectroscopy (ATR-FTIR) analysis. The modified BF samples are coded as AP-BF, GP-BF and MA-BF for (3-Aminopropyl) triethoxysilane, (3-Glycidyloxypropyl) trimethoxysilane and 3-(Trimethoxysilyl) propylmethacrylate modified BF, respectively.

2.3. Production of the composites

PBT and BF and were dried at 85 °C for 8 h prior to the compounding process. The constant amount of neat and modified BF (20 wt %) containing composites were prepared via laboratory type co-rotating twin screw extruder (L/D:40; Φ:16 mm), (GULNAR MAKINA, Istanbul, Turkey) with a temperature profile of 50-210-215-220-215-210 °C at 150 rpm. The extrudate was chopped into small pellets and stored in sealed plastic bag until the injection molding process. The specimens for mechanical tests were molded by a laboratory scale injection-molding machine (DSM Xplore 12 ml Micro-injection Molder, Netherlands) at a barrel temperature of 240 °C and the mold temperature of 28 °C.

Table 1
Mechanical properties of raw materials.

Material	Tensile Strength (MPa)	Percentage Strain (%)	Tensile Modulus (GPa)	Density (g/cm^3)
PBT	45–55	≤70	2.5	1.31
BF	2825	3.15	89	2.8

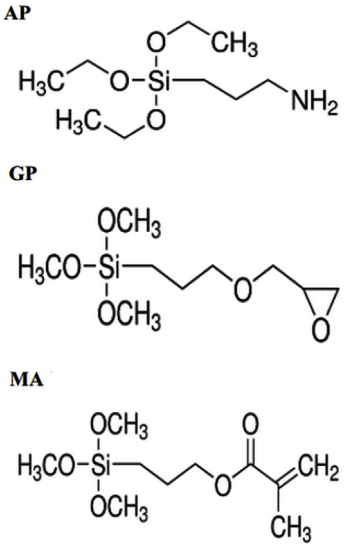

Fig. 1. Chemical structures of the silane coupling agents.

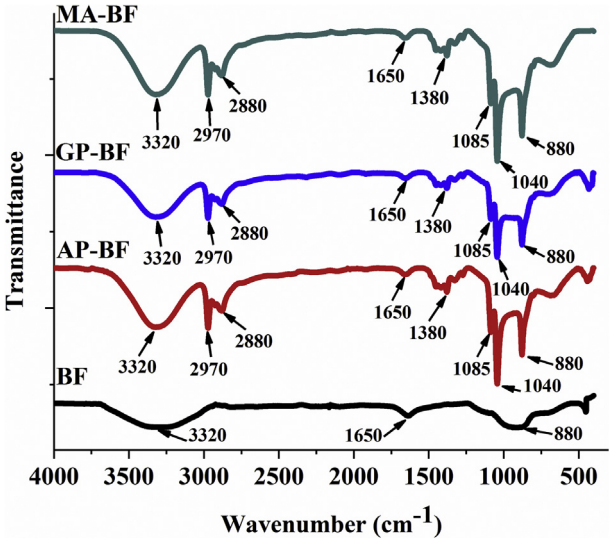

Fig. 2. FTIR spectrum of neat and modified BFs.

2.4. Characterization methods

ATR-FTIR was used to characterize the silane modified BF at an optical resolution of 4 cm^{-1} with 32 scans. Tensile and flexural tests were carried out on Shimadzu AG-X universal testing machine equipped with 50 kN load cell, according to ASTM D 638 and ASTM D-790 standards at room temperature. Tensile tests were conducted on the dog-bone shaped samples (7.4 × 2.1 × 80 mm^3) at a crosshead speed of 5 mm/min. Tensile strength and percentage elongation at break values were recorded. The flexural tests were performed on the specimens with the nominal dimensions of 13 × 125 × 3.2 mm^3, setting the span length of 55 mm and a cross-head speed of 1 mm/min. Charpy Impact tests were performed on unnotched samples with the dimensions of 3.2 × 6.5 × 130 mm^3 using Coesfeld-Material impact tester at room temperature according to ASTM D256. All mechanical test results represented an average value of five samples with standard deviations. DMA experiments were carried out using Perkin Elmer DMA 8000 in