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Investigation of Properties of Polymer/Textile Fiber Composites

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Polymer-based composite structures have advantages over other materials. The most important advantage is the higher mechanical properties obtained from the composites when supported by fiber reinforcement. The mechanical and thermal properties of fiber-reinforced composite structures are affected by the amount of fibers in the structures, orientation of the fiber and fiber length. Silk and cotton fibers are used in many fields but especially in clothing and textiles. However, there is not enough research on their usage as reinforcement fibers in composite structures. Silk fibers as a textile material have better physical and mechanic properties than other animal fibers. It is very important that the improvement of the mechanical and physical properties of the composite structures allows them to be used in many areas. From economical, technological and environmental points of view, the improved the mechanical and physical properties of polymeric materials are receiving much attention in the recent studies.

In this study, various lengths (1 mm–2.5 mm and 5 mm) of waste silk and waste cotton fibers were added to high-density polyethylene (HDPE) and polypropylene (PP) polymer in the mixing ratios of (polymer: fiber) 100%:0%, 97%:3%, and 94%:6% to produce composite structures. On the other hand, known lengths (1–2.5–5 mm) of waste silk and waste cotton fibers were added to recycled polyamide-6 (PA₆) and polycarbonate (PC) polymers in mixing quantities of 100%:0%, 97%:3%. A twin-screw extruder was employed for the production of composites. Tensile strength, % elongation, yield strength, elasticity modulus, Izod impact strength, melt flow index (MFI), heat deflection temperature (HDT), and Vicat softening temperature properties were determined. In order to determine the materials' thermal transition and

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microstructure properties, differential scanning calorimetry (DSC) and scanning electron microscopy (SEM) were used. Results have shown that cotton and silk fibers behave differently than in the composite structure. Waste silk fiber composites give better mechanical properties than waste cotton fiber.

Keywords cotton, polyamide, polycarbonate, polyethylene, polymer composites, polypropylene, silk

INTRODUCTION

During the past 30 years, composites have pervaded almost all material domains (house furnishing, packaging, car, aerospace, transportation, sport, leisure, and so on). All synthetic polymers (thermoplastics, thermosets and elastomers) can be used as matrices. As fillers, besides inorganic powders, much use has been made of man-made fibers of inorganic (glass, silicium carbide) or organic origin (carbon, aramid) in the form of individual fibers (chopped or continuous), and mats. Except for glass fibers, these reinforcing fibers are expensive. Various fibers are also produced in nature (cellulose, wool, silk, cotton) [1]. Silk fibers are biodegradable and highly crystalline with well-aligned structure. It has been known that they also have higher tensile strength than glass fiber or synthetic organic fibers, good elasticity, and excellent resilience [2].

This kind of polymer composite (polymer/natural fiber) is used in the furniture and automotive industries. The utilization of lightweight, recyclable, ecologically conscious, and low-cost natural fibers offers the potential to replace a large segment of the glass and mineral fillers in numerous automotive interior and exterior parts. In the past decade, natural-fiber composites with thermoplastic and thermoset matrices have been embraced by European car manufacturers and suppliers for interior and exterior automotive parts. Interior parts, door panels (flax/sisal with thermoset resin), glove box (wood/cotton fibers molded, flax/sisal), seat surface/backrest (coconut fiber/natural rubber), seat coverings (leather/wool backing), trunk panel (cotton fiber), trunk floor (cotton with PP/PET fibers), insulation (cotton fiber), and exterior part floor panels (flax mat with PP) are now produced at an industrial scale. Natural fibers such as kenaf, hemp, flax, jute, and sisal are providing automobile part reinforcement due to such drivers as reductions in weight, cost, less reliance on foreign oil sources, recyclability, and the added benefit that these fiber sources are “green” or eco-friendly [3–5].

With the emphasis on environmental awareness, consciousness and legislation, academic and industrial needs for developing environmentally friendly composite materials have recently been considerably increasing, based on renewable resources like natural fibers as alternatives for glass fiber reinforcement in traditional glass fiber-reinforced polymer matrix composites [6–10].

Advantages of natural fibers over traditional reinforcing fibers such as glass and carbon fibers are low-cost with low density, high toughness, acceptable specific strength, enhanced energy recovery, recyclability, and biodegradability [11]. Therefore, natural fibers can serve as reinforcement by improving the strength and stiffness and also by reducing the weight of the resulting biocomposite materials, although the properties of natural fibers vary with their sources and treatments [12–14].

Natural fibers are largely divided into two categories depending on their origin: plant-based and animal-based. In general, plant-based natural fibers are lignocelluloses in nature and are composed of cellulose, hemicellulose and lignin, whereas animal-based fibers are composed of proteins. Plant-based natural fibers like flax, jute, sisal and kenaf have been more frequently utilized and studied so far, due to their natural abundance, cost effectiveness, world annual production and a wide range of properties depending on the plant source. A large number of studies have been reported on biocomposites based upon these plant-based natural fibers earlier. However, the use of animal-based natural fibers like silk and wool in a biocomposite material has been rarely reported [14].

EXPERIMENTAL

The objectives of this study are to fabricate HDPE, PP, PA₆, and PC/waste silk and cotton composites and to investigate the effect of nontreated fiber (lengths and ratios) on the mechanical, thermal and morphological properties of the polymer/natural fiber composites. In this study, we just give properties of polymer/nontreated fiber composites. In a future study, we will compare

Table 1: Mixing ratios and fiber lengths of polymer composites.

Mixture of fiber lengths (1 mm, 2.5 mm, and 5 mm)		
97% polymer/3% fiber		94% polymer/6% fiber
HDPE/Silk		HDPE/Silk
HDPE/Cotton		HDPE/Cotton
PP/Silk		PP/Silk
PP/Cotton		PP/Cotton
Fiber lengths (97% recycled polymer/3% fiber)		
1 mm	2.5 mm	5 mm
PA 6/Silk	PA 6/Silk	PA 6/Silk
PA 6/Cotton	PA 6/Cotton	PA 6/Cotton
PC/Silk	PC/Silk	PC/Silk
PC/Cotton	PC/Cotton	PC/Cotton

treated and nontreated fiber properties. In this paper, fundamental results for understanding the performance and potential of polymer/untreated natural fiber composites will be discussed in terms of tensile strength Izod impact, hardness, thermal (T_g : glass transition temperature, T_m : melting temperature, HDT: heat deflection temperature, Vicat softening point) properties, MFI (melt flow index) properties, and microscopic observations. The mixing ratios of polymer/natural fiber composites are given below in Table 1.

MATERIAL AND COMPOSITIONS

Properties of HDPE, PP, PA₆, and PC and waste silk and waste cotton fiber are given in Tables 2 and 3, respectively.

Waste silk fibers were cut with a guillotine in lengths of 1 mm, 2.5 mm, and 5 mm. Waste cotton fibers were collected from different stages of the cotton yarn production process in the lengths of 1 mm, 2.5 mm, and 5 mm. Different mixing ratios of (97% polymer/3% fiber and 94% polymer/6% fiber) HDPE

Table 2: Properties of HDPE, PP, PA₆, and PC.

Properties	HDPE ^a	PP ^b	PA ₆ ^c	PC ^d
Place of Production	Pektim Petrokimya Holding A.S. Turkey	Pektim Petrokimya Holding A.S. Turkey	Dilaplast S.p.A Italy	Sabic Innovative Plastics Asia Pacific
Type	Petilen I 668 (UV)	Petoplen MH 418	Dilamid 6 Naturel	Lexan 144 R
Density (g/cm ³)	0.968	0.910	1.14	1.20
Flexural Modulus (MPa)	–	–	2.50	–
Melt Flow Rate (MFR), (g/10 min)	5.4 (190°C/2.160 kg)	5.0 (230°C – 2.160 kg)	–	11 (300°C – 1.2 kg)
Tensile Strength (Yield) (MPa)	28.9	34.3	70	62.1
Tensile Strength (Break) (MPa)	23.5	42.2	–	68.9
Tensile Elongation (Break) (%)	1300	–	–	130
Heat Deflection Temperature (°C)(1.8MPa, Unannealed)	–	–	70	132
Izod Impact Strength (J/m) (Notched – 23°C)	49	–	–	801

^aThe data were taken from <http://prospector.ides.com/results.aspx?A=RESET&CK=77837>.

^bThe data were taken from <http://prospector.ides.com/results.aspx?A=RESET&CK=46863>.

^cThe data were taken from <http://prospector.ides.com/results.aspx?A=RESET&CK=82700>.

^dThe data were taken from <http://prospector.ides.com/results.aspx?A=RESET&CK=71180>.

(–) means not available or not applicable.

Table 3: Properties of silk and cotton fiber.

Properties	Waste silk (15)	Waste cotton (15)
Place of Production	Bursa/Turkey	Çukurova/Turkey
Type	Waste Silk	Waste Cotton
Source	Filament Waste	Blow room waste up to 5 mm Taker in waste up to 2.5 mm Tambour waste up to 1 mm
Fiber Thickness (dtex)*	1,5	1,7
% Trash Content	–	45
Density (g/cm ³)	1,3–1,37	1,54
% Moisture Absorption	11	8
Fiber Strength (g/tex)**	30–50	4–5
% Elongation	13–20	5–6
Thermal Conductivity	poor	poor

(–) means not available or not applicable.

*(dtex): stands for decitex. Tex is the international unit for the fineness of textile fibers, and expresses the weight in grams per 1,000 m length; i.e., 1 tex is 1 g/km. Decitex or dtex stands for the weight in grams of a fiber 10,000 m long. The higher the dtex figure, the coarser the fiber.

** (g/tex): Tenacity of the fibers.

and PP polymer with waste silk and cotton fibers were prepared at mixed lengths of 1 mm to up to 5 mm. However, recycled PA₆ and PC polymer were mixed with waste silk and waste cotton fiber at the ratios of 97% polymers/3% fibers with different fibers lengths of 1 mm, 2.5 mm, and 5 mm.

Samples were mixed with a twin-screw extruder (Maris TM40MW – Maris America Corporation Baltimore, USA). Extrusion conditions are given in Table 4.

The injection conditions are given in Table 5. Test samples were prepared in an Arburg brand injection machine (Arburg GmbH Co., Lossburg, Germany), according to ISO 294. Tensile tests were carried out with a Zwick 1120 machine (Zwick GmbH, Ulm, Germany); with test speed 50 mm/min according to ISO 572.2. Consequently, the mechanical properties, like tensile strength, elasticity modulus, yield strength and % elongation, are tested in the same machine. The Izod impact test was done according to ASTM D256 standard with a Ceast impact test device (Ceast Spa, Pianezza, Italy). MFI values were determined in a Zwick 4100 brand testing machine, according

Table 4: Extrusion conditions of the polymer composites.

Process	HDPE silk and cotton	PP silk and cotton	PA ₆ silk and cotton	PC silk and cotton
Temperature (°C)	85–190	85–190	85–230	85–230
Pressure (bar)	26–30	20–24	15–27	8–9
Screw Turning Rate (rpm)	305–357	193–355	246–355	240–249
Cooling Temperature (°C)	85	85	85	85

Table 5: Injection conditions for the polymer composite.

Process	HDPE silk and cotton	PP silk and cotton	PA ₆ silk and cotton	PC silk and cotton
Temperature (°C)	210–230	210–230	220–250	230–260
Pressure (bar)	40	40	40	40
Waiting Time in Mold (s)	10	10	10	10
Cooling Temperature (°C)	40	40	40	40

to ASTM D 1238. Thermal transition temperatures of each polymer composite were determined by using a Universal V2.6D Differential Scanning Calorimeter (DSC) (TA Instruments, New Castle, NH, USA; starting point: 40–50°C, ending point: 180–300°C, test rate: 20°C/min). To investigate their microstructures, samples were covered with 40 Å thickness carbon with Polaron SC 502 machine (Gala Instrument GmbH, Bad Schwalbach, Germany) and SEM photographs were taken under 10 kV current with a JSM-5410 LV JOEL SEM machine (Jeol, Peabody, MA).

RESULTS AND DISCUSSION

Figure 1 shows the elasticity modulus, yield and tensile strength, % elongation, Izod impact strength and hardness values of HDPE/silk and HDPE/cotton polymer composites.

Figure 1 outlines the effects of 3% and 6% waste silk and waste cotton fiber addition to the HDPE polymer on the mechanical properties of the composites. As is shown in Figure 1, the elasticity modulus of the HDPE composite has

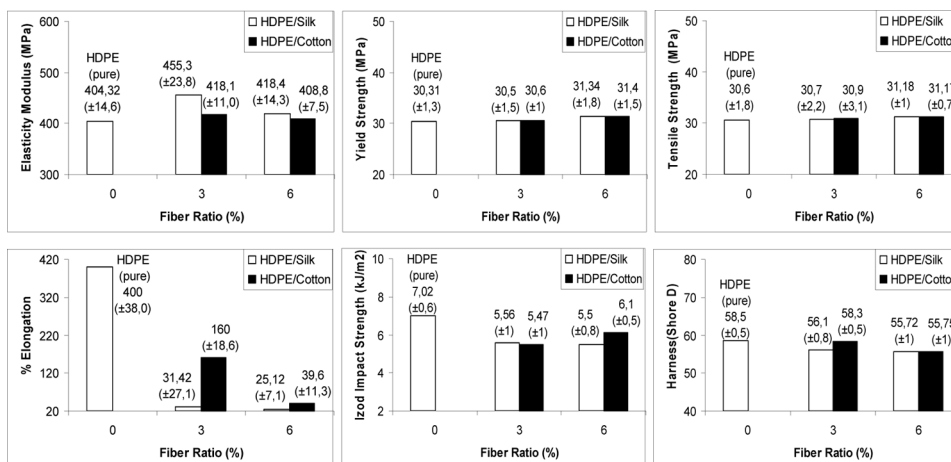


Figure 1: Mechanical properties of HDPE/Silk and HDPE/Cotton polymer composites.

increased with the addition of 3% and 6% cotton and silk fibers compared to 100% HDPE polymer. However, a decrease was observed as the fiber concentration in the composite increased when compared with all other composites. There were no considerable changes in the yield and tensile strength of the composites when compared with 100% HDPE polymer. However, elongation values of the composites decreased with the addition of fibers. Izod impact strength values of the polymer composites decreased with the addition of fibers. Addition of fibers to HDPE polymer did not considerably affect the hardness.

Figure 2 shows the elasticity modulus, yield and tensile strength, % elongation, Izod impact strength and hardness values of PP/silk and PP/cotton polymer composites.

Figure 2 outlines the effects of 3% and 6% waste silk and waste cotton fiber addition to the PP polymer on the mechanical properties of the composites. Figure 2 shows that the addition of 3% and 6% cotton and silk to PP decreased the elastic modulus of the composites compared with 100% PP. But an increase was observed as the fiber content increased in the case of silk but a decrease was observed in the case of cotton composites. There were no considerable changes in the yield and tensile strength of the composites when compared with 100% PP polymer. Elongation of the composites decreased drastically when compared with 100% PP, but there were no significant changes within the composites themselves. The addition of fibers to PP polymer did not change the hardness values but increased the Izod impact strength. However, as the silk fiber concentration increased, Izod impact strength decreased, but in the case of cotton increases were observed.

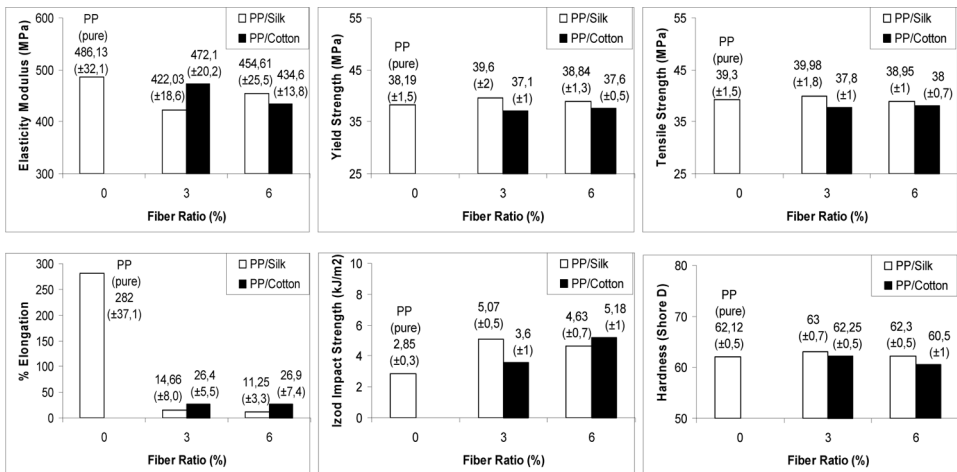


Figure 2: Mechanical properties of PP/Silk and PP/Cotton polymer composites.

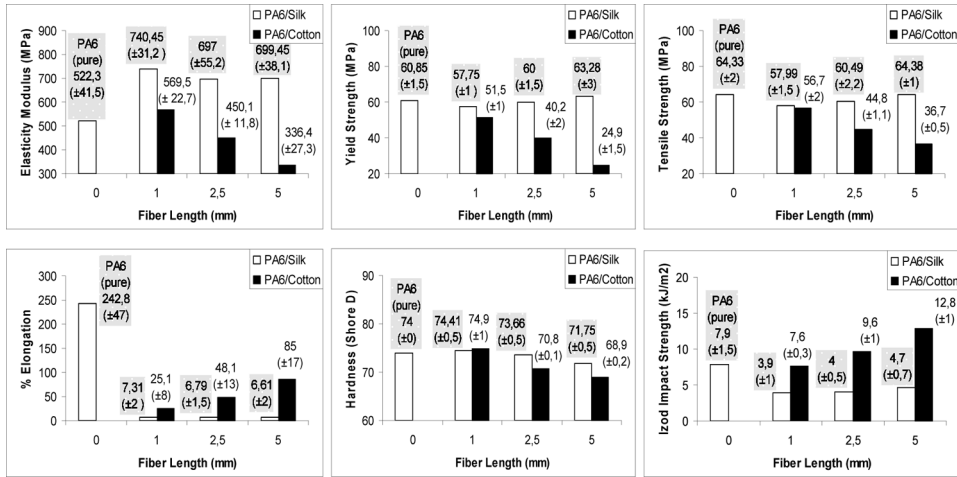


Figure 3: Mechanical properties of PA₆/Silk and PA₆/Cotton polymer composites.

Figure 3 shows the elasticity modulus, yield and tensile strength, % elongation, Izod impact strength and hardness values of PA₆/silk and PA₆/cotton polymer composites.

Figure 3 outlines the effects of constant 3% waste silk and waste cotton fiber addition to the recycled PA₆ polymer with increasing fiber length of 1 mm, 2.5 mm and 5 mm on the mechanical properties of the composites. As can be seen in Figure 3 on the PA₆ composite, the addition of constant 3% fiber content with a fiber length increase did not significantly effect the elasticity modulus in the case of silk fiber, but a decrease was observed in the case of cotton except 1 mm length, where an increase was observed for both fibers. Yield and tensile strength values of the recycled PA₆ composites were decreased when compared with 100% recycled PA₆. But as the cotton and silk fiber lengths increased, yield and tensile strength values of cotton composites decreased and silk composites increased. Elongation values of the composites decreased when compared with 100% recycled PA₆ but increasing fiber length increased the elongation values of the cotton composites. No apparent changes were observed for silk composites. The addition of 1 mm fiber lengths of cotton and silk to the 100% recycled PA₆ had no significant effect on the composites' hardness values, but a decrease was observed as the fiber lengths were increased to 2.5 mm and 5 mm. Izod impact strength values of the composites increased in the case of cotton fiber and decreased in the case of silk fiber addition.

Figure 4 shows the elasticity modulus, yield and tensile strength, % elongation, Izod impact strength and hardness values of PC/silk and PC/cotton polymer composites.

Figure 4 outlines the effects of constant 3% waste cotton and waste silk fiber addition to the recycled PC polymer with increasing fiber length of

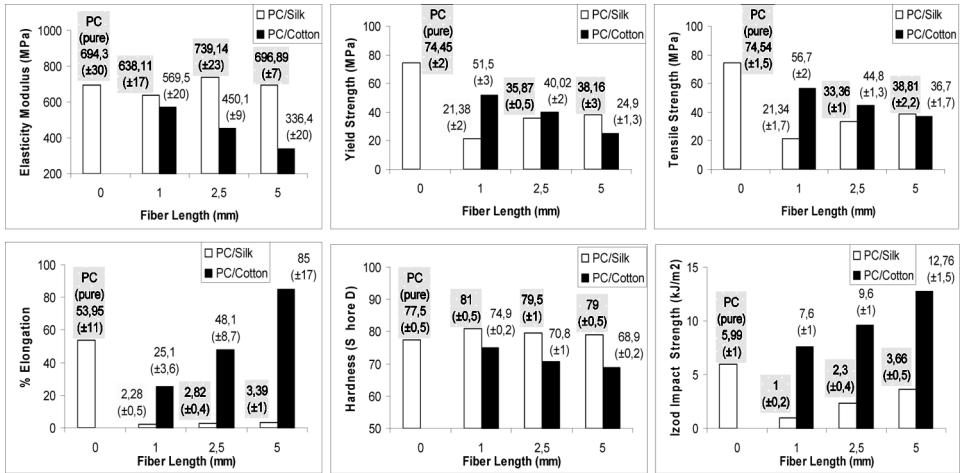


Figure 4: Mechanical properties of PC/Silk and PC/Cotton polymer composites.

1 mm, 2.5 mm and 5 mm on the mechanical properties of the composites. Elasticity modulus and strength values of the cotton composites decreased as compared with 100% recycled PC polymer and also decreased with increasing fiber length. However, in the case of silk there was an overall increase after an initial decrease by 1 mm fiber length. With the fiber length increase, yield strength values increased with silk composites and decreased with cotton composites. But yield strength values of different fiber lengths of silk and cotton composites were decreased relative to 100% recycled PC polymer. Tensile strength values of the silk and cotton-added composites were decreased as compared with 100% recycled PC polymer. On the other hand, with the fiber length increase, tensile strength values increased in silk-added composites and decreased in cotton-added composites.

The addition of silk to the recycled PC polymer reduced the elongation values of silk composites drastically, but in the case of cotton the reduction of elongation values was not so drastic and for both silk and cotton composites elongation values increased with the fiber length increase. The addition of silk to the recycled PC polymer increased a little the hardness, and hardness values were a little increased compared with 100% recycled PC as well. But in the case of added cotton recycled PC composites' hardness values decreased with increasing fiber lengths, and the hardness values decreased from those of 100% recycled PC.

Izod impact values of cotton composites were increased compared with 100% recycled PC. The increase continued with increasing fiber length but in the case of silk composites the values reduced drastically. But with increasing fiber lengths, Izod impact strength values did increase.

Table 6: MFI, HDT and Vicat softening point values of polymer composites.

**MFI values (g/10 min) HDPE (190°C-2.16 kg), PP (230°C-2.16 kg)
PA₆ (235°C-2.16 kg), PC (300°C-1.2 kg)HDT (°C- 1.80 M Pa)
Vicat softening point (°C-1 kg)
Mixing fiber lengths (1 mm, 2.5 mm, and 5 mm)**

	97% polymer/3% fiber			94% polymer/6% fiber		
	MFI	HDT	Vicat	MFI	HDT	Vicat
HDPE (pure)	:4.24	107,1	110,4			
HDPE/Silk	:2.51	53,3	122,9	HDPE/Silk	65,5	132,0
HDPE/Cotton	:1.50	111,0	130,3	HDPE/Cotton	114,2	140,8
PP (pure)	:4.28	125,8	153,7			
PP/Silk	:4.29	130,4	159,0	PP/Silk	135,3	155,2
PP/Cotton	:2.21	150,3	154,4	PP/Cotton	133,3	154,1

	(97% Recycled polymer/3% fiber)			Fiber length: 5 mm		
	MFI	HDT	Vicat	MFI	HDT	Vicat
PA ₆ (pure)	:13,22	55,0	207,1			
PA ₆ /Silk	:16,87	55,3	207,9	PA ₆ /Silk	54,0	207,7
PA ₆ /Cotton	:3,52	52,6	208,9	PA ₆ /Cotton	52,5	209,2
PC (pure)	:26,20	110,0	142,0			
PC/Silk	:50,74	110,0	142,9	PC/Silk	112,3	142,7
PC/Cotton	:71,02	117,4	143,5	PC/Cotton	117,9	154,3

	Fiber length: 1 mm			Fiber length: 2.5 mm		
	MFI	HDT	Vicat	MFI	HDT	Vicat
PA ₆ (pure)	:13,22	55,0	207,1			
PA ₆ /Silk	:16,87	55,3	207,9	PA ₆ /Silk	54,0	207,7
PA ₆ /Cotton	:3,52	52,6	208,9	PA ₆ /Cotton	52,5	209,2
PC (pure)	:26,20	110,0	142,0			
PC/Silk	:50,74	110,0	142,9	PC/Silk	112,3	142,7
PC/Cotton	:71,02	117,4	143,5	PC/Cotton	117,9	154,3

Table 7: DSC values of polymer composites.

DSC values (starting point: 40–50 °C, ending point: 180–300 °C, test rate: 20 °C/min)			
97% polymer/3% fiber	94% polymer/6% fiber		
		T_m (°C)	T_m (°C)
HDPE (pure)		:138,90	
HDPE/Silk	HDPE/Silk	:138,95	:167,88
HDPE/Cotton	HDPE/Cotton	:148,60	:152,22
PP (pure)		:161,7	
PP/Silk	PP/Silk	:162,17	:168,25
PP/Cotton	PP/Cotton	:169,47	:171,28

DSC values (starting point: 40–50 °C, ending point: 180–300 °C, test rate: 20 °C/min)					
Fiber length: 1 mm		Fiber length: 2.5 mm		Fiber length: 5 mm	
	T_g (°C)	T_m (°C)	T_g (°C)	T_m (°C)	T_g (°C)
PA ₆ (pure)		220,01			
PA ₆ /Silk		220,37	PA ₆ /Silk	219,69	PA ₆ /Silk
PA ₆ /Cotton		219,88	PA ₆ /Cotton	225,96	PA ₆ /Cotton
PC (pure)	145,60				
PC/Silk	140,48		PC/Silk	147,88	PC/Silk
PC/Cotton	138,18		PC/Cotton	141,60	PC/Cotton

T_g: Glass Transition Temperature. T_m: Melting Temperature.

Table 6 shows all the MFI, HDT and Vicat softening point values of all composites made with different quantities of cotton and silk waste in PP, HDPE, PA₆, and PC polymers.

From the values in Table 6, it is seen that the addition of 3% and 6% silk and cotton to HDPE and PP reduced the MFI values, except for the case of PP/silk where the values increased. The addition of silk to PA₆ and PC have increased the MFI values except in PC polymer with a 5 mm fiber length. In contrast, the addition of cotton increased the MFI values for the PC composite

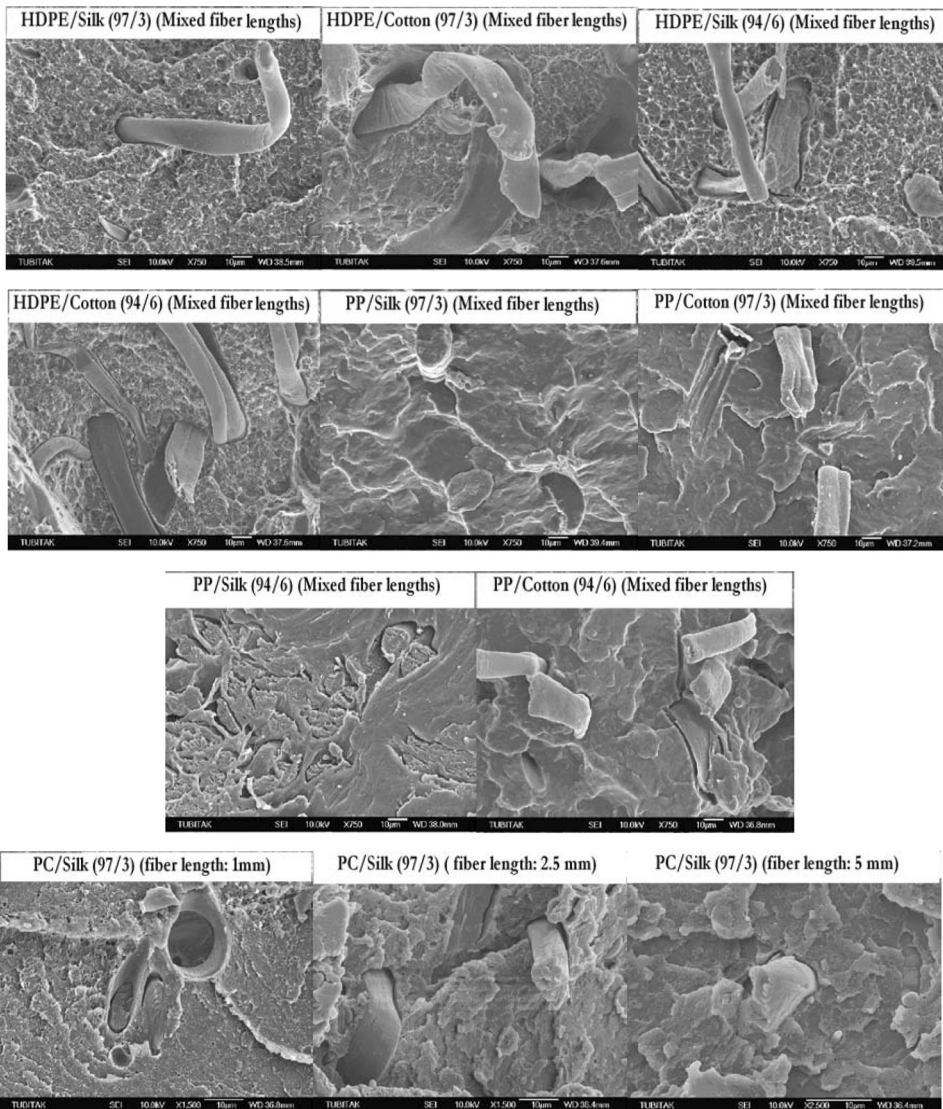


Figure 5: SEM photos of Polymer Composites.

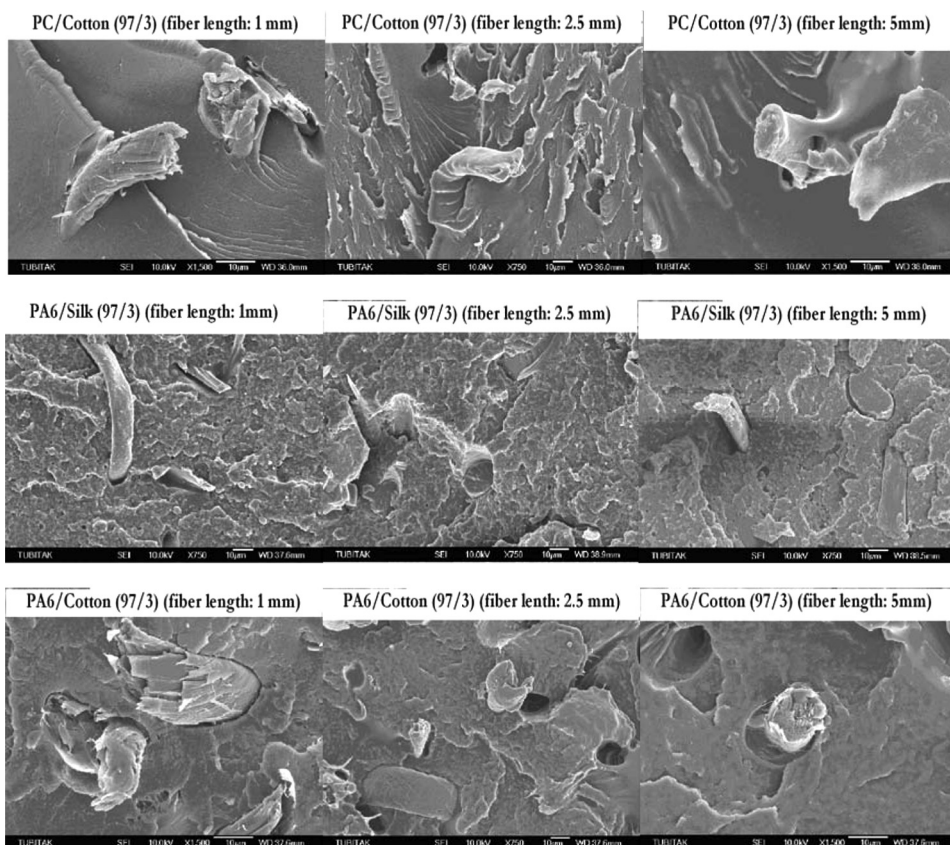


Figure 5. Continued.

relative to the PC polymer, but a decrease was observed in the case of the recycled PA₆ composite compared with pure recycled PA₆ polymer. With the addition of silk to HDPE, the Vicat softening temperature increased but HDT decreased. Increasing the amount of silk increased these values gradually. With the addition of cotton to HDPE, the Vicat softening temperature and HDT increased. Increasing the amount of cotton increased these values gradually. With the addition of silk to PP, HDT increased in parallel to the waste silk ratio. On the other hand, the Vicat softening point increased with the addition of 3% cotton to PP, but it decreased with the addition of 6% cotton to PP.

With the addition of silk and cotton to PA₆, there no considerable changes in the Vicat softening point and HDT. With the addition of silk to PC, there is no considerable change in the Vicat softening point, but with the addition of cotton to PC, HDT increased.

Table 7 shows all the DSC values of all composites with different quantities of silk and cotton waste in PP, HDPE, PA₆, and PC.

The results of DSC studies show that there is no considerable change in the T_m value of HDPE, with the addition of 3% silk waste but an increase to 6% increases the T_m value. However, the addition of 3% cotton increased the T_m of HDPE. There was no significant change in the melting temperature for PP with 3% addition of silk. Increasing the rate to 6% cotton increased the T_m value. As shown in Table 7, the addition of 3% cotton to PP, increased the T_m value.

There is no significant change in melting temperature for PA₆ with the addition of silk waste in different lengths. On the other hand, the addition of 2.5 mm length cotton waste to PA₆ increased the T_m value of the composites. With the addition of waste silk to PC the T_g value decreased, but in silk fiber of 2.5 mm length there was an increase in the T_g value of the composite. On the other hand, with the addition of cotton waste to PC the T_g value decreased.

Figure 5 shows the SEM photos of all polymer composites made with silk and cotton waste added to PP, HDPE, PA₆, and PC polymers.

When the silk and cotton polymer composites' microstructures were examined it was clearly seen that the silk and cotton fibers did not orientate in a clear direction and there was little no or adhesion with the matrix polymer. This may be due to the absence of bonding between the matrix and the fiber.

CONCLUSIONS

Results have shown that cotton and silk fibers behave differently in the composite structure. Much information is available in the literature concerning the global mechanical properties of thermoplastic/natural fiber composites: tensile strength, flexion and breaking stresses, elongation at break. The lack of cohesion between nontreated fibers and matrix, and fiber orientation, are well evidenced by the polymer/fibers rupture profiles where the fibers separate from the matrix when the rupture occurs. Consequently, when the fiber ratio was increased in HDPE-PP/silk-cotton polymer composites, mechanical properties did not change significantly. On the other hand, short silk and cotton fiber-reinforced PA₆ and PC polymer composites were successfully fabricated and their mechanical properties are generally better.

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