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## Recycled Polyamide-6/Waste Silk & Cotton Fibre Polymer Composites:Effect of Fibre length

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### Abstract

Among textile fibres today, cotton is the most commonly used natural fibre. Cotton is being used in almost all areas of textiles. Waste cotton which is produced during the yarn production process is then re-used in yarn production again or as a waste in other uses. Besides this, silk fibres have been used for nearly 5000 years. Silk is one of the most precious fibres among all textile fibres and it has a very wide range of uses like: sewing threads, clothes, home textiles, technical textiles and ornamental uses. The cocoons which are damaged during the cocoon-forming process are called waste silk, and because of the damage their trading value is decreased. These wastes can then be re-used in textiles.

In this study, besides the classic textile applications, a different application area was chosen to evaluate silk and cotton wastes. Silk and cotton waste and recycled PA<sub>6</sub> polymer was mixed and a composite structure was obtained. Silk and cotton wastes were in fibre lengths of 1mm, 2.5 mm and 5mm. The recycled PA<sub>6</sub>/Silk and cotton wastes were mixed in the rates of 97% / 3% respectively. The mixture was made by twin screw extruder. The samples were tested in terms of tensile strength, % elongation, yield strength, elasticity module, Izod Impact Strength, Melt Flow Index (MFI), Heat Deflection Temperature (HDT), and Vicat Softening Point Temperature. Thermal transitions of the materials were determined using the Differential Scanning Calorimeter (DSC) and micro-structure properties were investigated with Scanning Electron Microscope (SEM).

**Keywords:** Mechanical properties, polyamide 6, fiber length, polymer composites, silk, cotton.

## 1. Introduction

The most commonly used matrix materials in fibre-reinforced composites are polymers. Composites with plastic base and reinforced with fibres have very good properties. Today, there are many fibres used as a reinforcement material in composites. The fibres that are used as a reinforcement material especially in the polymeric mixtures are chosen to have very short lengths. Therefore, the fibre wastes which can occur during the yarn production processes can be used as fibre reinforcement in a composite structure. In the literature, natural fibres are used as a reinforcement material as well as mineral and glass fibres in composite structures. Studies were concentrated especially on: high density polyethylene, polypropylene, polyamide-6 and polyamide-12/wood flour, cellulose flour and cellulose fibres[1], dicumyl peroxide-modified cellulose/LLDPE composites[2], low density polyethylene (LDPE)/cellulose fibers[3], wood fibers as reinforcing fillers for polyolefins[4], polypropylene/wood fiber composites[5], jute fiber reinforced thermoplastic polymers (LDPE, HDPE, PE copolymer, and PP)[6], polypropylene (PP) and high density polyethylene (HDPE) filled with wood and cellulose flour[7], polypropylene/flax[8], polypropylene/sisal fibers[9], short sisal fiber-reinforced polyethylene composites[10], LDPE/short sisal fiber[11], PS/short sisal Fiber [12,18], polyester/Jute fiber[13], methacrylamide/silk[14-17]. In this study, 1, 2.5 and 5mm in length of cotton and silk wastes in 97% (recycled PA<sub>6</sub>)/3%(waste fibre) mixture rate with recycled PA<sub>6</sub>/ waste silk and recycled PA<sub>6</sub>/ waste cotton were mixed and the composite structures produced were examined.

## 2. Experimental

### Materials

Recycled PA<sub>6</sub>/ waste silk and cotton polymer composites mixture rates and fibre lengths in groups are given in Table 1. Mechanical, thermal and physical properties of the materials used in these polymer composites are given in Table 2.

Table 1 Recycled PA<sub>6</sub>/ waste silk and cotton polymer composite mixture ratio and fibre length

Groups	PA <sub>6</sub> (%)	Silk (%)	Cotton (%)
1	100	-	-
2	97	3 (Silk length: 1 mm)	3 (Cotton length: 1 mm)
3	97	3 (Silk length: 2.5 mm)	3 (Cotton length: 2.5 mm)
4	97	3 (Silk length: 5 mm)	3 (Cotton length: 5 mm)

Table 2 Mechanical, thermal and physical properties of the materials used in this polymer composites

Properties	Silk *	Cotton *	PA6 **
Place of Production	Bursa / Turkey	Cukurova / Turkey	Dilaplast /Italy
Type	Waste silk	Waste cotton	Dilamid 6
Source	Filament waste	Blowroom waste up to 5mm, Taker-in waste up to 2.5mm, Tambour waste up to 1mm	-
Fibre Thickness	1.5 (dtex)	4.2 (micronaire)	-
Trash (%)		45	-
Density (g/cm <sup>3</sup> )	1.3-1.37	-	1.13
Moisture (%)	11	-	-
Tenacity (g/tex)	30-50	-	-
Elongation (%)	13-20	-	-
Tensile Strength (MPa)	-	-	75
Water absorption (24 Hours - %)	-	-	1.5-2.0
Izod Impact Strength 23 °C (J/m <sup>2</sup> )	-	-	75
Tensile Module(MPa)	-	-	2500

(-) means Not Available or Not Applicable

\* A. Pervin, *Textile Pre-treatment*, Alfa Pub, Turkey, 1998, 124.

\*\* Petkim Production Catalogue(HDPE ),2008.

### Instruments

Silk fibres used in the textile industry are called Bombyx Mori type silk. Waste silk fibers which are obtained during the cocoon forming or the silk filaments production are cut with guillotine in to 1mm, 2.5mm and 5mm fiber lengths. Different lengths of cotton fibres which had fallen down from grids during the opening and carding process were used in this work. The cotton wastes obtain from this process is gathered into fibre lengths of 1, 2.5 and 5mm. The mixture of recycled PA<sub>6</sub> with silk and cotton waste were obtained under 85-230 °C temperatures, 25 Bar pressure and mix rate of 335 t/min; using twin screw extruder in Maris brand (Maris TM40MW–Maris America Corporation Baltimore, USA). After the mixing process test samples were prepared on injection moulding machine. The extrusion and injection conditions are given in Table 3.

Table 3 The extrusion and injection conditions used in preparing the polymer composite

Process	Extrusion	Injection
Temperature (°C)	85-230	220-250
Pressure (bar)	25	40
Waiting Time in Mould (s)	-	10
Screw Turning Rate (rpm)	355	-
Cooling Temperature (water) (°C)	85	40

Samples were prepared in an Arburg brand injection machine (Arburg GmbH Co., Lossburg-Germany), according to ISO 294. Tensile strength tests were carried out with a Zwick 1120 machine (Zwick GmbH, Ulm-Germany); with test speed 50mm/min and according to ISO 572.2. Consequently, the mechanical properties like tensile strength, elasticity modulus, and yield strength and % elongation were tested in the same machine. Izod impact strength tests were made with a Zwick brand test machine, according to ISO 180. MFI values were made in Zwick 4100 brand testing machine, according to ASTM D 1238. DSC studies and the thermal transitions were determined with a Seteram DSC 131 machine (Scientex Pty., Ltd., Victoria, Australia). HDT and Vicat Softening Point was determined with a Ceast 6505 testing machine (Ceast SPA, Pianezza, Italy). To investigate their microstructure, samples were covered with 40 A° thickness carbon with a Polaron SC 502 machine (Gala Instrument GmbH, Bad Schwalbach, Germany) and SEM photographs were taken under 10kV current with a JSM-5410 LV JOEL SEM (Jeol, Peabody, MA) machine.

### **3. Result and discussion**

Mechanical properties of the PA<sub>6</sub>/waste silk and PA<sub>6</sub>/waste cotton polymer composites are given in Figure 1. As seen in Figure 1, the addition of 1mm length silk waste to recycled PA<sub>6</sub> increases the elasticity modulus of polymer composites, and increasing the length of silk decreased elasticity modules. Addition of 1mm fibre length silk waste gave the highest elasticity modulus value. The values of yield strength and tensile strength were decreased with in the first group which is 1mm fibre lengths but increased with increasing fibre lengths. With the addition of waste silk, elongation (%) values were decreased considerably. Hardness values were decreased slightly and Izod impact strength values of the composites were also decreased compared to 100% recycled PA<sub>6</sub>. The addition of waste cotton to recycled PA<sub>6</sub> increases the elasticity modulus of the polymer composite. However, the increase in fibre length causes a decrease in the elasticity values. The values of yield strength and tensile strength values were decreased with increasing fibre lengths. Considerable decreases were observed on the values of elongation (%) with the addition of cotton waste to recycled PA<sub>6</sub>. However, the increase in fibre lengths causes an increase in elongation values. Hardness values were also decrease with addition of cotton waste to recycled PA<sub>6</sub> after an initial increase in Group 2. The addition of cotton waste to recycled PA<sub>6</sub> to form polymer composite increases the Izod impact strength of the resultant composites with increasing fibre lengths.

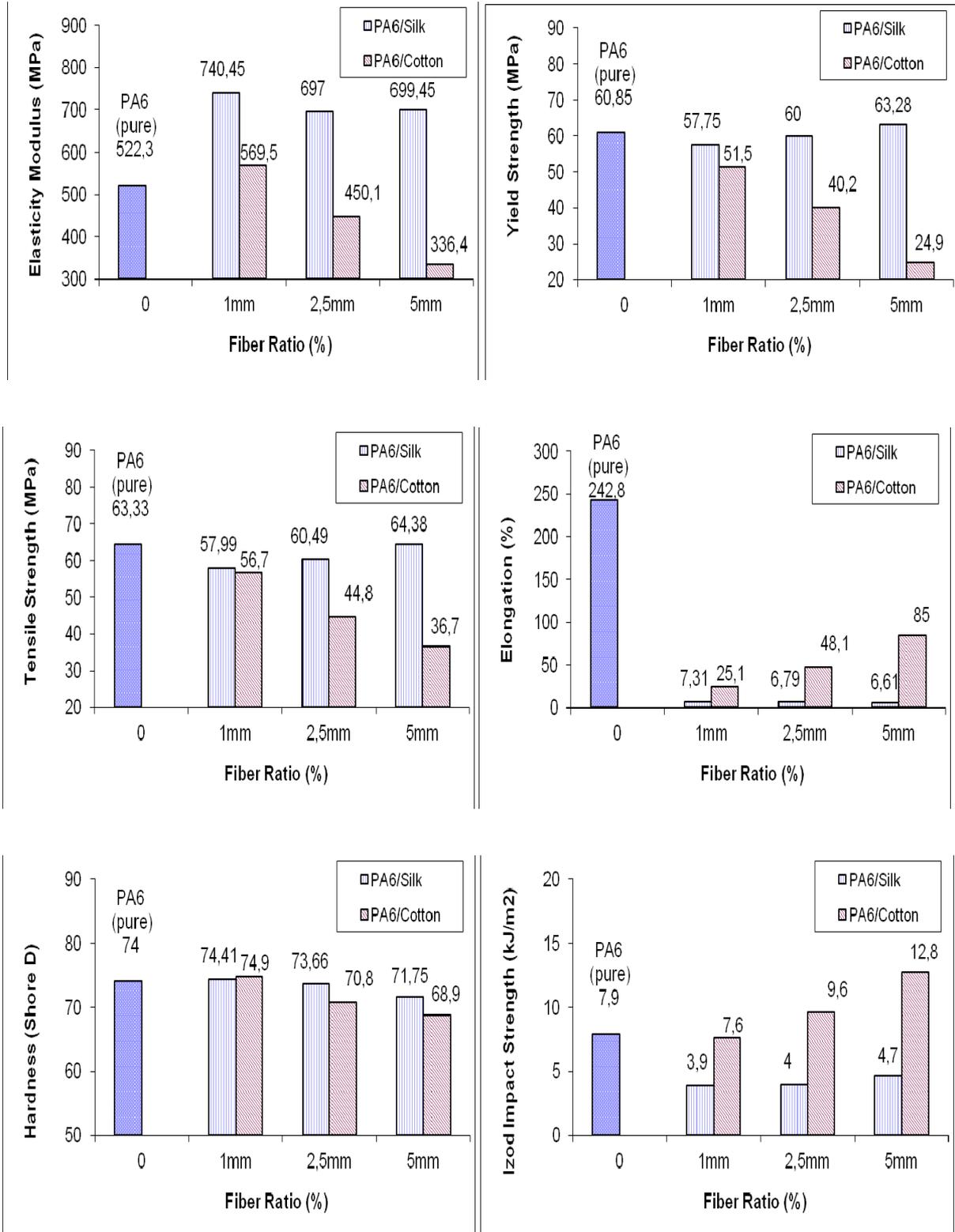


Figure 1 Mechanical properties of the PA<sub>6</sub>/waste silk and PA<sub>6</sub>/waste cotton polymer composites

As seen in Figure 2, the addition of waste silk to recycled PA<sub>6</sub> polymer increases the MFI value. There is no significant change in heat deflection temperature and Vicat softening point with the addition of silk waste. With the addition of waste cotton to recycled PA<sub>6</sub> polymer the MFI value of the composite decreased considerably. Also, there is no significant change in heat deflection temperature and Vicat softening point with the addition of cotton waste in different lengths. The DSC curves of polymer composites produced with recycled PA<sub>6</sub>/waste silk and recycled PA<sub>6</sub>/ waste cotton are given in Figure 3.

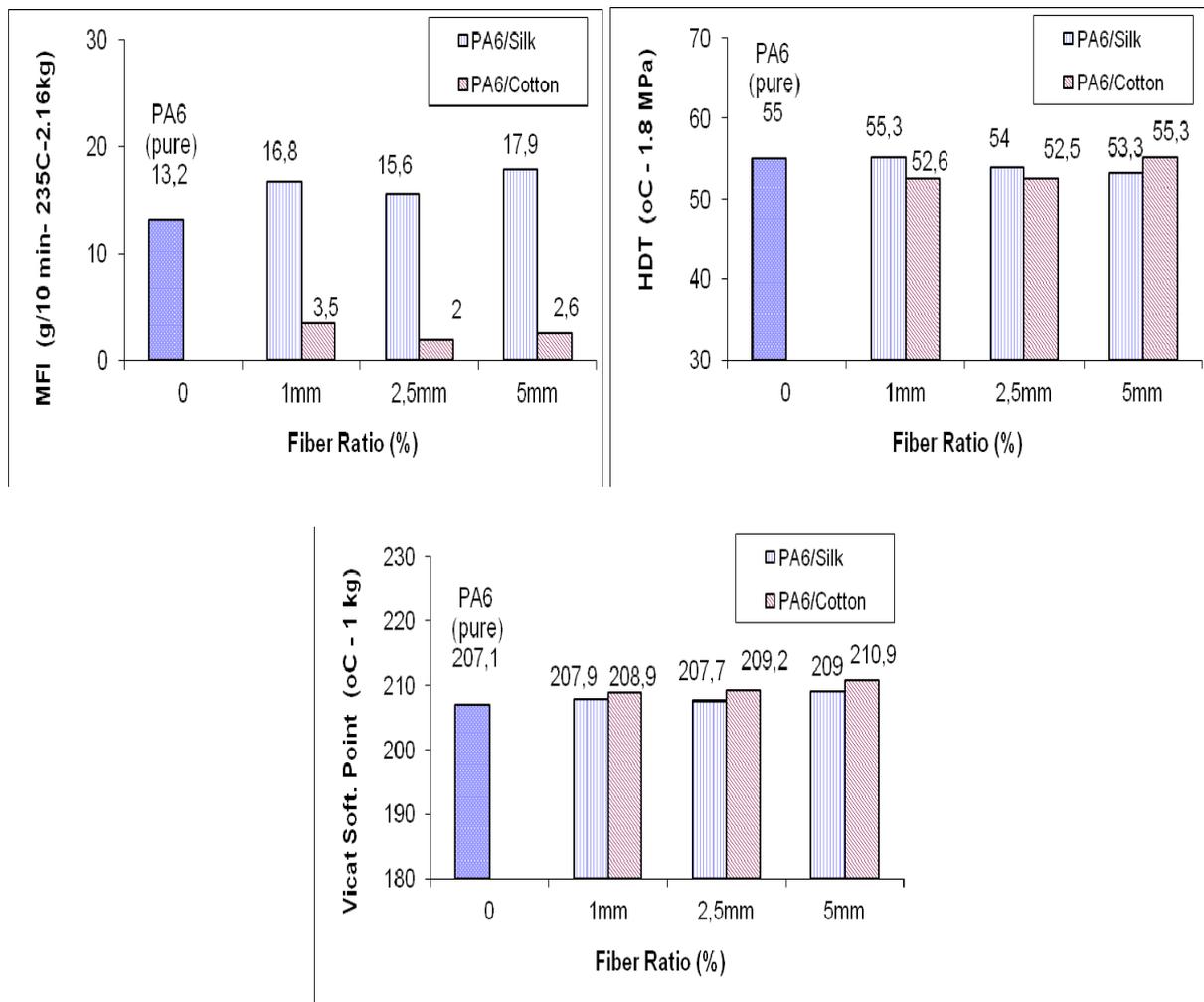


Figure 2 Thermal properties of the PA<sub>6</sub>/waste silk and PA<sub>6</sub>/waste cotton polymer composites

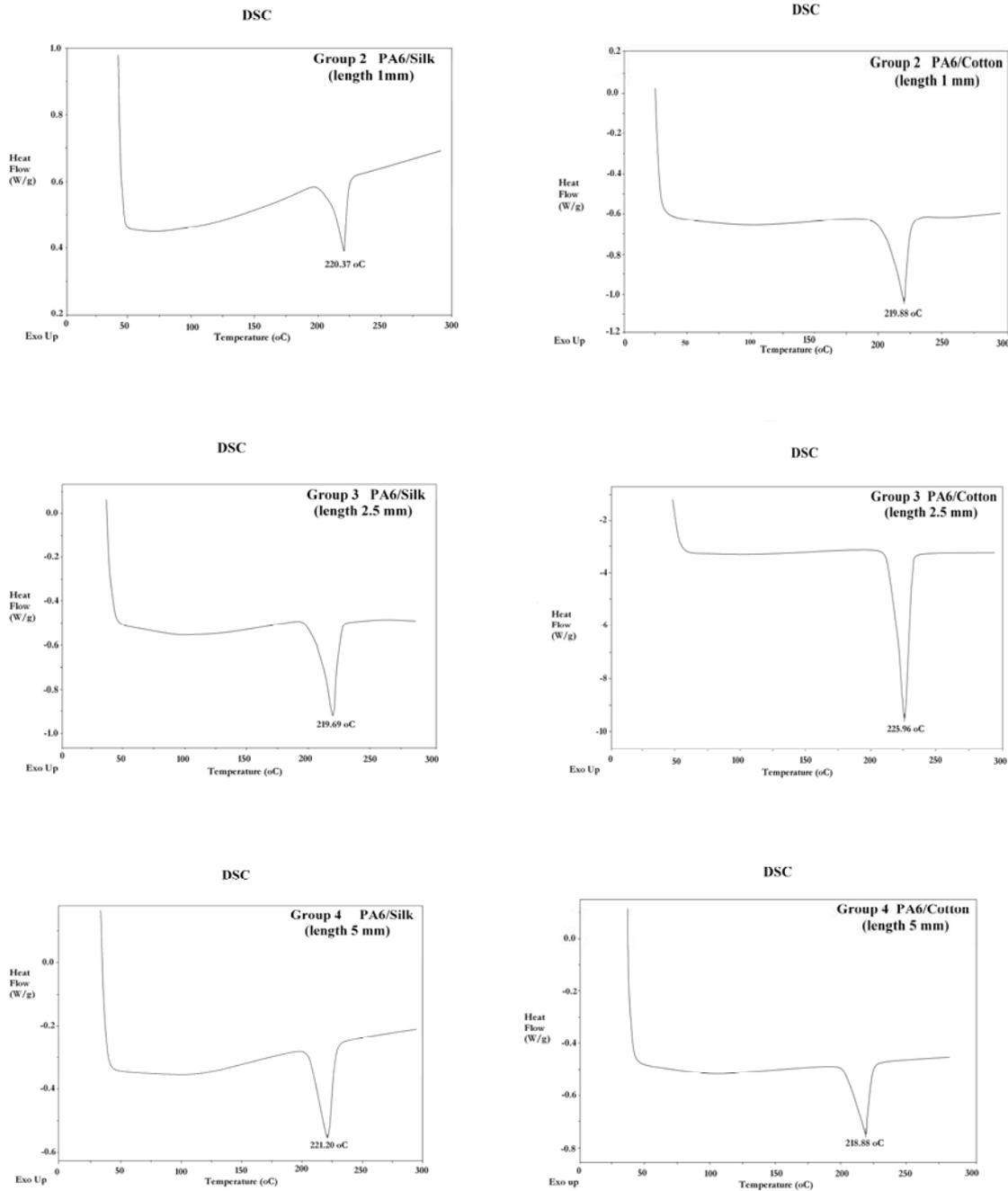


Figure 3 DSC curves of the recycled PA<sub>6</sub>/waste silk and PA<sub>6</sub>/waste cotton polymer composites

There is no significant change in melting temperature ( $T_m$ ) for PA<sub>6</sub> with the addition of silk waste in different lengths. On the other hand, the addition of 2.5 mm length cotton waste (group 3) to PA<sub>6</sub> increases the  $T_m$  value of the composites. Figure 4 shows the SEM photographs of the recycled PA<sub>6</sub>/ waste silk and recycled PA<sub>6</sub>/waste cotton polymer composites. When SEM pictures taken from the breaking surfaces are checked, it can be

seen that the fibres come out of the matrix as they hold lightly between non-polar matrix polymers and polar fibres. It was also seen that the fibres were not well dispersed over the matrix. Presence of cotton and silk fibres are obvious in the composites, and fibres are not fully adhering into the polymer. This was due to the absence of compatibilizers which were not used in this study. As a result there was no or little adhesion with matrix polymer and the fibre, especially in the case of the cotton waste. Silk fibre shows better adhesion into the matrix in the composite structure than the cotton fibre. It is very interesting that in this work raw cotton fibre that carries natural waxes on the surface of the fibre were used. With heat the surface waxes were affected by the heat and provided skin for the cotton fibre which is very obvious in the SEM photographs.

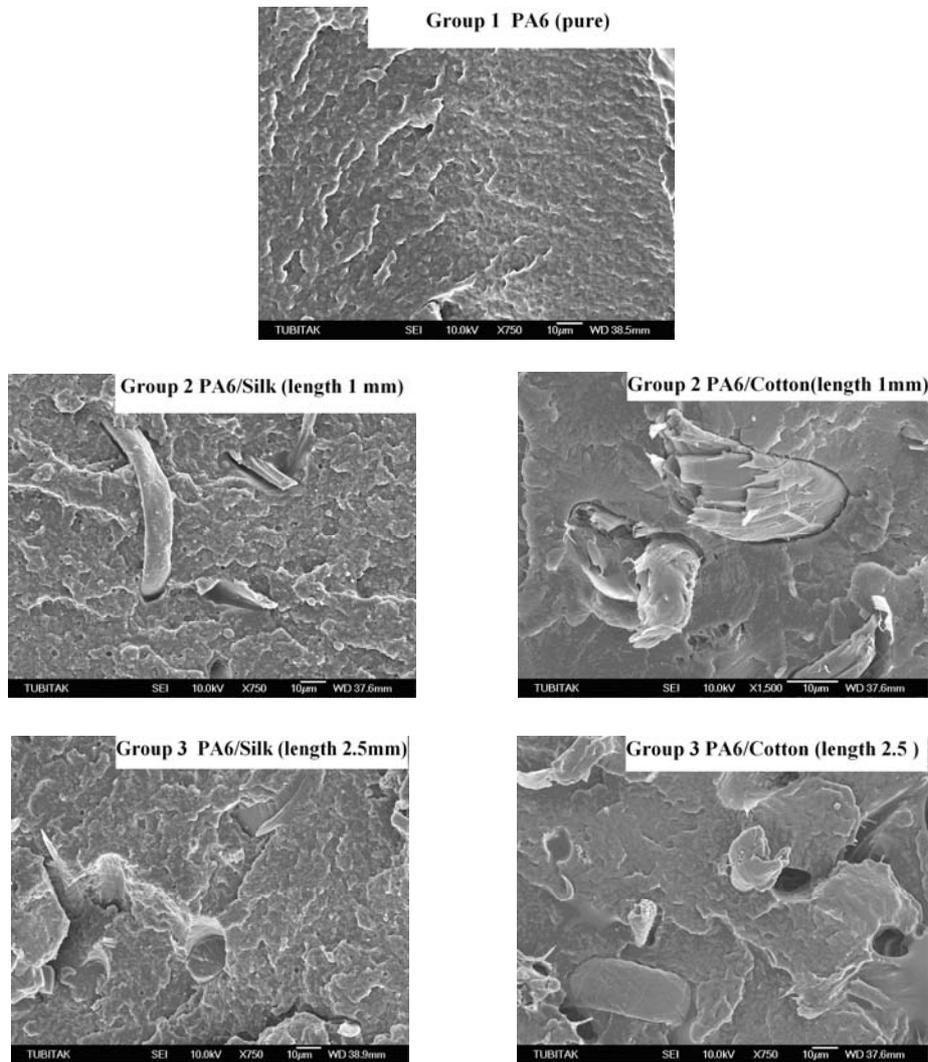


Figure 4 SEM photographs of recycled PA<sub>6</sub>/waste silk & cotton polymer composites

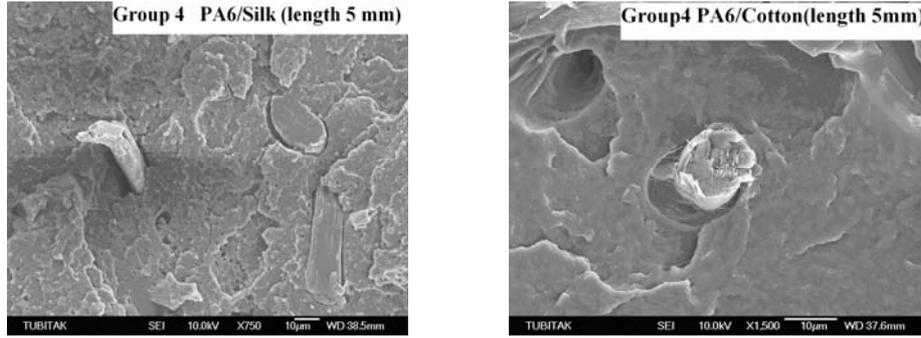


Figure 4 (continue)

#### 4. Conclusion

There is an important increase in the elasticity modulus of the composites made up of PA<sub>6</sub> polymer/waste silk fibres. The amount of the increase for the composite made up with the addition of 1mm length waste silk fibres especially can be explained in terms of attachment achieved by the dimensional volume increase. Improvement in elasticity modulus of the polymer composite with the addition of waste cotton fibres of 1 mm length was achieved. But, elasticity modulus decrease was observed in composites made with the addition of 2.5mm and 5mm waste cotton fibres. This can be explained in relation to the waste cotton fibre dimensional volume. When flow resistance values of the composed composites are considered, because of circular segment of the silk fibre, there is no flow problem, whereas cotton fibre show flow disturbance because of non-circular segment. When tensile strength values are evaluated, there is no strength increase in either fibre added to PA<sub>6</sub> polymer. As silk fibre is stronger than cotton fibre, tensile values were protected as before. It is known that tensile strength of a composition mainly depends on the intermediate structure. As there is no connective used in the intermediate structure formed here, it was not enough that fibres held on the structure. Furthermore, it can be seen that depending on the structure cotton fibres affect the structure negatively. Elongation values of the composites seemed to decrease in waste silk and cotton fibres with PA<sub>6</sub> polymer. The reason for that is that polymer structure is weaker than fibres and the waste fibres are in more fragile structure. When hardness values and Izod impact strength values are considered, SEM photos taken from breaking surfaces show interestingly big gaps. Here the increase in Izod impact strength of the composite formed with the addition of waste cotton, there is fewer gaps between the non-circular segment of cotton fibre and increasing dimensional percentage. As a result, when mechanical, thermal and morphological characteristics are considered, with the addition of waste cotton fibres to the PA<sub>6</sub> polymer and elasticity modulus values of composite formed with silk fibres

increase and the composite formed in both fibres meet the demanded characteristics in themselves.

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