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An investigation on structural properties of jute particle doped polypropylene filaments

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REZUMAT – ABSTRACT

Investigații referitoare la proprietățile structurale ale firelor filamentare de polipropilenă dopate cu particule de iută

În această lucrare au fost studiate procesul de producție și caracterizarea structurală a firelor filamentare de polipropilenă dopate cu particule de iută. Înainte de compoundare, a fost efectuată modificarea suprafeței iutei, cu un agent alcalin, silan ca agent de cuplare, agent pe bază de fluorocarbon și, de asemenea, plasmă de argon. Particolele de iută modificată și polipropilena grefată cu anhidridă maleică folosită ca și catalizator au fost încorporate în polipropilenă într-un extruder cu doi melci, fiind ulterior filate prin topire în fire de polipropilenă multifilamentare, la scară de laborator. Polipropilena 100% și firele multifilamentare compozite au fost caracterizate cu ajutorul spectrofotometriei FTRI, a calorimetriei cu scanare diferențială și a difractometriei cu raze X. Au fost de asemenea efectuate teste de rupere a fibrelor și a fost analizat conținutul de umiditate al acestora. Distribuția agenților de umplere de-a lungul firelor filamentare compozite a fost analizată cu ajutorul microscopiei fluorescente. Analizele au arătat că adăugarea atât de particole de iută modificată, cât și de polipropilenă grefată cu anhidridă maleică nu au avut efecte seminficative asupra structurii cristalografice a polipropilenei, datorită conținutului scăzut de material de umplere. Pe de altă parte, prezența particolelor de dopare a produs o creștere a absobției de umiditate a firelor filamentare compozite. Analiza TGA a arătat, de asemenea, că firele filamentare de polipropilenă au atins absobția maximă de umiditate, la adausul de particole de iută tratate cu agent alcalin.

Cuvinte-cheie: polipropilenă, fir filamentar compozit, iută, caracterizare

An investigation on structural properties of jute particle doped polypropylene filaments

The production process and structural characterization of jute particle doped polypropylene filaments were considered in this paper. Prior to compounding, surface modification of jute was also carried out with alkali, silane coupling agent, fluorocarbon based agent and also argon plasma. Modified jute particles and maleic anhydride grafted polypropylene as a compatibilizer were incorporated into polypropylene in a twin-screw extruder and then master-batches were converted to polypropylene multi-filaments in lab-scale melt spinning machine. 100% polypropylene and composite multi-filaments were characterized by Fourier transform infrared spectrometer, thermo-gravimetric analyzer, differential scanning calorimeter, and X-Ray diffractometer. Fiber tensile tests and moisture contents were also studied. Distributions of fillers along the composite filaments were detected by fluorescence microscopy. The analyses showed the addition of both modified jute particles and MAPP had no significant effects on crystallographic structure of the polypropylene due to the low content fillers. On the other hand, the presence of fillers caused an increment in the moisture absorption of the composite filaments. TGA analysis also pointed out that the polypropylene filaments achieved the maximum moisture absorption with the addition of alkali treated jute particles.

Key-words: polypropylene, composite filament, jute, characterization

INTRODUCTION

Textile fibers are commonly used as apparel, upholstery, and carpets [1]. Among the textile fibers, polypropylene (PP) is a well-known and one of the most widely used man-made fibers. Recent trend towards textile products of polypropylene have been focused on to its easy processing property, low density, excellent chemical stability and also low cost. However, PP fibers have very poor water and moisture absorption properties. Thus, it is necessary to apply modifications to the PP fibers for some applications. Fibers can be modified physically or chemically. Physical modifications include changing the cross section shape of the filaments in spinning process by means of increasing surface area of the filaments. Several researches were reported on the effect of fiber cross section shape on various properties of fibers such as water absorption, compressibility, surface characteristics [2–5]. On the other hand, PP fibers can be also modified chemically by graft polymerization, finishing treatments and incorporation of a various additives or fillers (organic or inorganic) in molten PP during extrusion process [2, 6–8]. Among these techniques, composite filament production method by doping fillers to polymers became even more important recently.

In this study, polypropylene composite filaments doped with modified jute particles were manufactured and structural characterization of these filaments was studied. Prior to compounding, jute particles were

modified with several surface treatments to enhance compatibility with PP and to provide homogeneous distribution of jute particles along the filament. It is well known that PP has no side groups that cellulose based particles could be attached to. Previous related scientific literature confirmed that surface treatments of cellulose based fibers enhanced the interaction with non-polar polymer [9–11]. Therefore, jute fibers as in particle form were treated with alkali (as a pretreatment process) (AJ), silane coupling agent (ASJ), fluorocarbon based agent (AFJ) and also exposed to argon plasma modification (APJ). PP master-batches containing 0.5% wt modified jute particles and 5% wt maleic anhydride grafted polypropylene (MAPP) as a compatibilizer agent were manufactured and then spinning process of multi-filaments were carried out. 100% PP and composite multi-filaments were characterized by Fourier transform infrared spectrometer (FTIR), thermo-gravimetric analyzer (TGA), differential scanning calorimeter (DSC), and X-Ray diffractometer (XRD). In addition to these analyses, tensile tests, color measurements, and determination of moisture absorption of the multi-filaments were also studied. Moreover, distributions of fillers along the composite filaments were detected by fluorescence microscopy.

EXPERIMENTAL

Materials

Waste jute yarns supplied from Atlantik Halı A.Ş. were used as filler materials. The contents of cellulose, hemicellulose, lignin and the others constituents of the jute yarns were determined as 69.6%, 10.2%, 12.5% and 7.7%, respectively [12]. Sodium hydroxide and ethanol were purchased from Merck Corp. 3-aminopropyl triethoxysilane was supplied from ChemCruz Biochemicals. Fluorocarbon chemical agent (Rucostar EEE) was provided from Rudolf Group. Polypropylene was supplied from Hellenic Petroleum with the trade number of HZ40S for fiber spinning. Maleic anhydride grafted polypropylene (MAPP) used as a compatibilizer was purchased from Sigma Aldrich.

Methods

Preparation of jute particles

Waste jute yarns were grinded in Retsch Cutting Mill SM 100 grinder by using a sieve having holes in 250 μm. In order to reduce the size of the jute particles, they were exposed to grinding process again in Fritsch Pulverisette 7 grinder at the speed of 850 rpm for 20 min.

Surface treatments of jute particles

Jutes were treated with 5% NaOH aqueous solution for 2 h at ambient temperature. The jutes were rinsed out with distilled water several times to remove the chemical residues and then neutralized with distilled water with few drops of acetic acid. After treatment, jutes were oven-dried at 60°C until it dried and then kept in a desiccator [13, 14]. It should be mentioned that alkali treatment was performed as a pretreatment process for surface cleaning before plasma modification, silane and fluorocarbon treatments.

Subsequent to alkali pretreatment, silane treatment was carried out to modify the jutes to achieve less hydrophilic surface character. Before treatment, silane coupling agent was hydrolyzed in water/ethanol solution (60/40 v/v) with the addition of concentrated acetic acid to adjust pH 4 and continuously stirred for 1 h. Then, jutes were immersed in 5% w/w silane hydrolyzed solution for 3 h at ambient temperature. The jutes were washed with distilled water several times to remove the chemical residues. The treated jutes were dried in ambient temperature for 3 days and then oven dried at 80ºC [15].

After alkali pretreatment, 50 g/L fluorocarbon based compound was added in the padding liquor for fluorocarbon treatment of jutes. The jutes were immersed in the padding liquor for 3 min. After washing several times, the jutes were oven-dried at 170°C for 2 h.

After alkali pretreatment, the jutes were modified by plasma in PICO RF (radio frequency) plasma polymerization system (Diener electronic GmbH+Co.KG, Germany). Argon was used as plasma gas for 15 min and in 90 W discharge power.

Preparation of polypropylene composite masterbatches

PP, MAPP and jute masterbatches were compounded at various ratios (table 1) by using a laboratory scale twin screw melt extruder. Prior to extrusion process, all compounds in powder form were mechanically mixed, dried and then melt blended in the extruder at a 170 rpm screw speed. The temperatures from feeder to spinneret hole including six extrusion zones were set at approximately 190°C. After extrusion, the compounded material was cooled down and pelletized as granules with the aforementioned concentrations.

Spinning of polypropylene composite multi-filaments

The polypropylene composite master batches were fed into a lab scale melt spinning machine having single screw extruder with two spinning nozzles in circular cross sections in order to spin multi-filaments. The temperatures from feeder to spinneret hole were including six extrusion zones in the range of 205–215°C. The extrusion speed and the extrusion pressure were set as 14 dpf and 80 bar, respectively. A draw ratio of 3.25 was applied to all samples by setting the speeds of the first and the second godet rolls at 200 rpm and 650 rpm, respectively. The temperatures of the first and the second godet rolls were set as 105°C and 115°C, respectively. Components of the composite filaments are detailed in table 1.

Characterization of the polypropylene composite filaments

The percentage crystallization of the polypropylene filaments was analyzed by using Perkin Elmer/Pyris 1

Table 1

COMPONENTS OF THE COMPOSITE PP GRANULES AND MULTI-FILAMENTS				
	Jute particle, wt %	**Type of jute particle**	**MAPP, wt %**	**Pure PP, wt %**
PP0	0	None	0	100
PP1	0	None	5	95
PP2	0.5	Alkali treated (AJ)	5	94.5
PP3	0.5	Alkali+silane treated (ASJ)	5	94.5
PP4	0.5	Alkali+fluorocarbon treated (AFJ)	5	94.5
PP5	0.5	Alkali+plasma modified /APJ)	5	94.5

Differential Scanning Calorimeter (DSC) under nitrogen atmosphere. The temperature raised from 0°C to 200°C at heating and cooling rates of 20°C/min. The percentage of crystallinity was calculated using the melting enthalpy by following formula:

$$\% \text{ Crystallinity} = (\Delta H_m / \Delta H_m^\circ) \times 100 \quad (1)$$

where ΔH_m is the melting enthalpy of the PP analyzed in this experimental study, ΔH_m° is assigned to the melting enthalpy of 100% crystalline PP, 207J/g [16].

The fine structure of polypropylene filaments were investigated by X-Ray diffractometer (XRD) using XRD Rigaku D/Max 2200 PC using Cu-Kα radiation and operating at 40 kV and 36 mA. The diffraction angles (2θ) of each sample were measured from 3° to 90° at a scan speed of 4°/min. The thermal decomposition of polypropylene filaments was studied by Thermogravimetric analysis (TGA) using Perkin Elmer Diamond TG/DTA instrument under nitrogen atmosphere (10 $mLmin^{-1}$) from 25°C to 600°C at a rate of 10°C min^{-1}. The polypropylene filaments were also characterized by Fourier transform infrared spectrometer (FTIR) analysis using Perkin Elmer Spectrum BX. Each spectrum was recorded in the range of 400–4000 cm^{-1} with a resolution of 2 cm^{-1}. Longitudinal views of composite filaments were taken by using Olympus BX 43 Fluorescence microscope for tracking the jute particles. The breaking strength, elongation at break and tenacity of the polypropylene filaments were determined by single fiber tensile test using Instron Tensile Testing Machine according to ASTM D 3822-07. The cross head speed and the gauge length was kept as 60 mm/min and 25 mm, respectively. Moisture absorption performances were tested in order to investigate the effect of jute particles and MAPP on moisture absorption behavior of the polypropylene filaments. Before testing, each sample for all types of the PP filaments having a mass of 0.5 g was dried at 60°C for 1 h in an oven. Then, each sample which was put in a closed weighing bottle, was taken to the constant temperature and humidity room (the temperature is 20±2 °C, and the humidity is 65±2 %). The weighing bottles were opened and the samples were weighed for every 5 minutes until the filaments were reached to moisture absorption balance [17]. The colour measurements of the polypropylene filaments were performed by Minolta 3600D CM spectrophotometer (D65 illuminant, specular included, 10° observer angle). The spectrophotometer having a software that could calculate CIEL*$a^*b^*C^*h^0$. The software also gives data about color strength (K/S) values from the reflectance values at the appropriate λ_{max} for each filament sample.

RESULTS AND DISCUSSIONS

Morphology of the membrane

DSC analysis

Table 2 lists the results of thermal analyses obtained by DSC and the crystallinity percentage values of the polypropylene filaments. DSC analysis resulted that melting temperature of PP0 is higher than that of PP1 which may be due to deterioration in polymer chain orientation as a result of the compatibilizer [16]. However, the melting temperatures of the PP filaments incorporated with the modified jute particles are higher than that of PP1. This may be due to the nucleating agent effect of the jute particles. The changes in the melting ($\Delta H_{(melting)}$) and crystallization ($\Delta H_{(crystallization)}$) enthalpies of the PP filaments confirm the thermal demonstration of the components during crystallization, as well as the interaction between PP, the compatibilizer and the jute particles in creating the supermolecular structure of the polypropylene filaments [18].

XRD analysis

Figure 1 illustrates XRD patterns of the polypropylene filaments. It is determined that the diffraction peaks of the crystals which ranged from 10° to 30°, indicated a typical form of α PP crystals [7]. The diffraction peaks of the PP filaments observed at around 14°, 17° and 25° correspond to (110), (040) and (060) crystallographic planes, respectively [19]. It is noticed that the PP filaments exhibited similar diffraction patterns. This may be due to the low content of jute particles and the compatibilizer agent.

Table 2

CALORIMETRIC DATA OF THE PP FILAMENTS					
	Melting temperature (°C)	**Crystallization temperature (°C)**	**Crystallinity (%)**	**$\Delta H_{(melting)}$ (J/g)**	**$\Delta H_{(crystallization)}$ (J/g)**
PP0	163.77	112.94	38.46	79.61	–85.10
PP1	161.40	111.60	38.83	90.38	–86.87
PP2	163.08	111.93	38.32	79.33	–86.00
PP3	164.07	114.33	39.50	81.77	–88.48
PP4	163.07	112.62	37.31	77.24	–81.78
PP5	162.07	112.29	38.08	78.83	–82.71

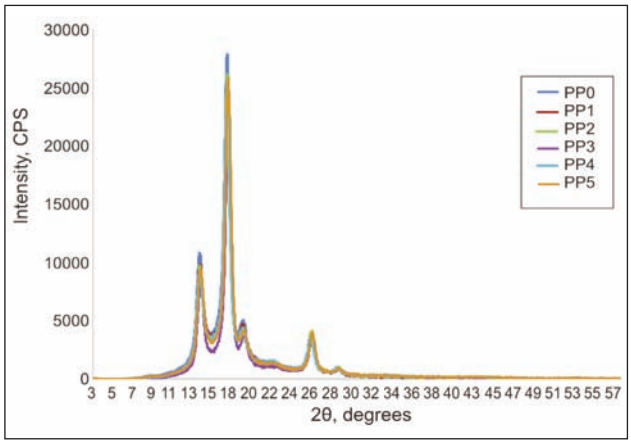

Fig. 1. XRD pattern of the PP filaments

Table 3

PERCENTAGE MASS LOSSES UNTIL SEVERAL TEMPERATURES ACCORDING TO TGA ANALYSIS OF THE PP FILAMENTS						
	Temperature (°C)					
	120	250	375	450	500	600
PP0	0	0	1.8	36.5	97.81	100
PP1	0.22	0.56	4.26	57.01	98.88	100
PP2	0.7	0.98	7.59	66.53	99.3	100
PP3	0.57	1.37	9.43	82.07	100	100
PP4	0	0.23	1.4	33.84	97.56	100
PP5	0	0.64	10.90	87.99	91.86	91.49

TGA was carried out to evaluate thermal stability of the polypropylene filaments as well as the effects of the jute particles and the compatibilizer on the PP. Figure 2 shows TGA thermograms and table 3 lists mass losses of the PP filaments until several temperatures. The single degradation step for both of the PP filaments confirms that the polymers are composed of the carbon-carbon bonds in the main chain, thereby allowing a temperature increase to promote random scission, with associated thermal degradation and thermal de-polymerization taking a place at a weak part of the polymer main chain [20].

As easily figured out from table 3 that no mass loss was recoded until 120°C for PP0 due to their highly non-polar character. But PP1 (having 5% MAPP) was observed to lose 0.22% of its mass until 120°C which can indicate its moisture content. Further, the addition of AJ particle increased the weight loss of PP2 until this temperature due to the presence of highly polar alkali treated jute particles. But PP4 and PP5 are noticed to have no moisture content. These may be due to the application of fluorine groups by fluorocarbon treatment and reduction in hydrophilic groups of jute particles by argon plasma modification [11, 21]. Close examination of the thermogravimetric results that the temperatures which PP0, PP1, PP2, PP3, PP4 and PP5 filaments lose 50% of their masses are 455°C, 448°C, 441°C, 432°C, 458°C and 427°C, respectively. It is figured out that the addition of MAPP and AJ, ASJ and APJ particles negatively influenced the thermal stability of the PP filaments. The jute particles may agglomerate and act as impurities in the PP. But the improved thermal stability of PP4 might be due to enhanced interaction occurred between the jute particles and the PP.

FTIR analysis

Fourier transform infrared spectroscopy was utilized to identify various functional groups that emerged in the samples, as well as the disappearance of determinate groups [16]. Figure 3 shows FTIR spectra of the PP filaments. It is determined that the spectrums of the PP filaments are very similar to each other. A peak was obtained at the 2,951 cm^{-1}absorption band assigned to asymmetric CH_3 and the other peaks at 2,918 cm^{-1} and 2,838–2,839 cm^{-1} concern with asymmetric and symmetric CH_2 stretching vibrations, respectively [22]. The C=C bending vibrations which are in the region of about 1,300–1,400 cm^{-1} are overlapped with the symmetrical and asymmetrical bending vibrations of CH_3 in the PP at 1,374 cm^{-1} and 1,456 cm^{-1}, respectively [23]. The band at 1,102 cm^{-1} may be attributed to C-C and C-H deformations. The absorption band at 998 cm^{-1} is assigned to the characteristic crystalline band of PP [24].

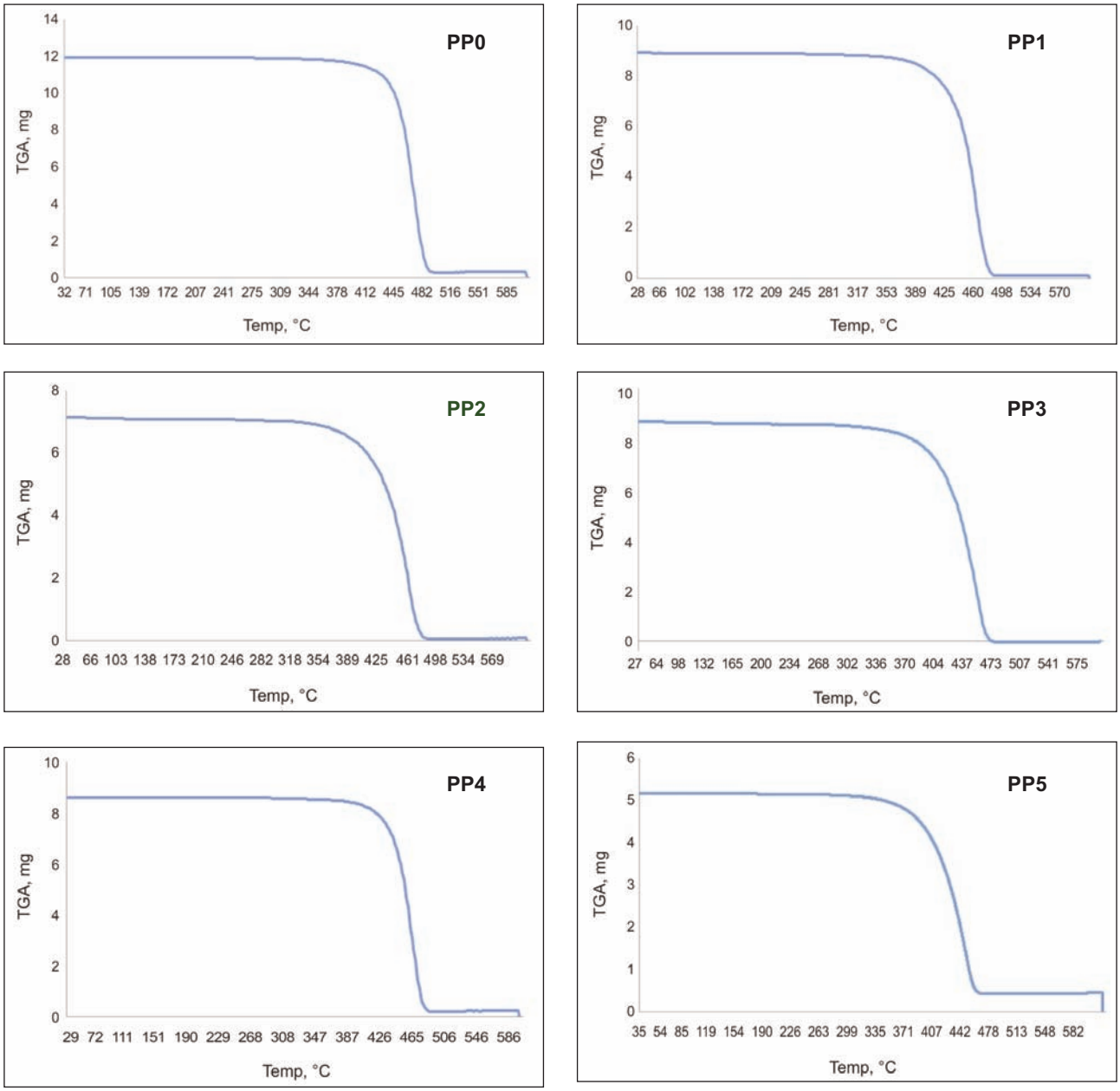

Fig. 2. TGA curves of the PP filaments

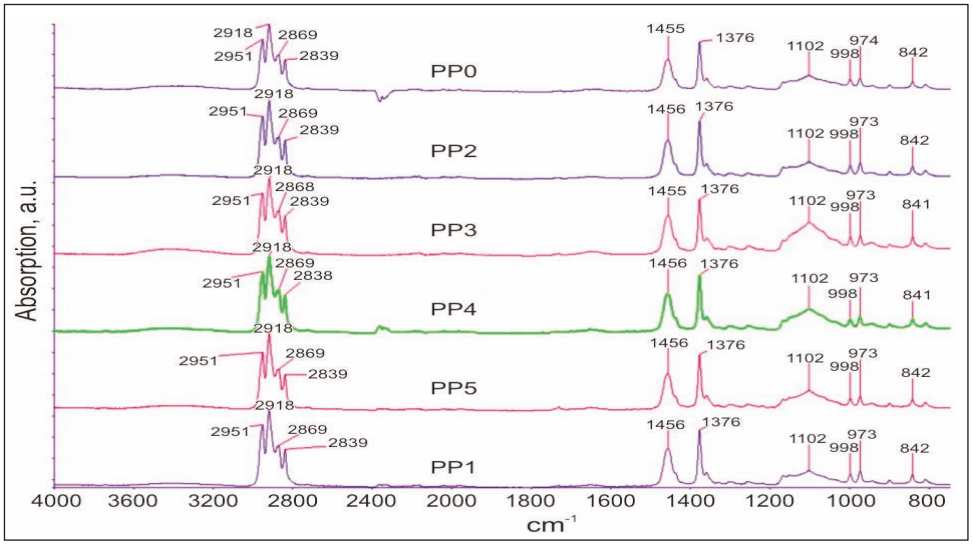

Fig. 3. FTIR spectra of the PP filaments

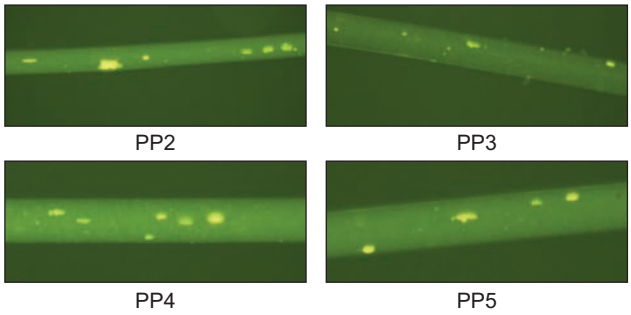

Fig. 4. Longitudinal views of PP composite filaments

Longitudinal views of PP composite filaments

To track the distribution and homogeneity of jute particles throughout the PP composite filaments, an optical control method was performed by using fluorescence microscopy technique. As shown in figure 4, the jute particles are seen in different colors due to their auto-fluorescence characteristics. Optical observations also indicate the agglomeration of the jute particles in the composite filaments.

Tenacity of the PP filaments

In order to investigate the effect of jute particles and the compatibilizer on the tenacity of the PP filaments, single fiber tensile tests were performed. Tenacity of the PP filaments is presented in figure 5. Tenacity results of PP0, PP1, PP2, PP3, PP4 and PP5 are 2.72 g/denier, 2.26 g/ denier, 2.19 g/denier, 2.71 g/denier, 2.27 g/denier and 2.83 g/denier, respectively. It is clearly understood from figure 5 that standard deviation values are observed to be high for all tested PP filaments. This may be due to non-homogeneous distribution of jute particles in the filament. As also noticed in figure 5, there are slight differences between the tenacity values of the PP filaments. This can be due to the addition of low jute particle content.

Moisture absorption of the PP filaments

Representative results of moisture absorption values of the PP filaments are shown in figure 6. It is observed that moisture absorption of PP filaments increased in the presence of MAPP compatibilizing agent. And moisture absorption values are extremely higher in PP composite filaments as compared to PP0 filament. It is found that PP2 have noticeably higher moisture absorption results in comparison with PP3, PP4 and PP5 filaments incorporating ASJ, AFJ and APJ particles, respectively. In case of silane treatment, reactive silanol groups of silane coupling agent can from chemical bonds with hydroxyl groups of jute particles and can form polysiloxane layer on the surface of the jute particles. With fluorocarbon treatment, a great amount of fluorine containing groups may cause hydrophobicity on the surface of the jute particles [11]. Additionally, a reduction in hydrophilic groups of jute particles may exist after argon plasma modification [21].

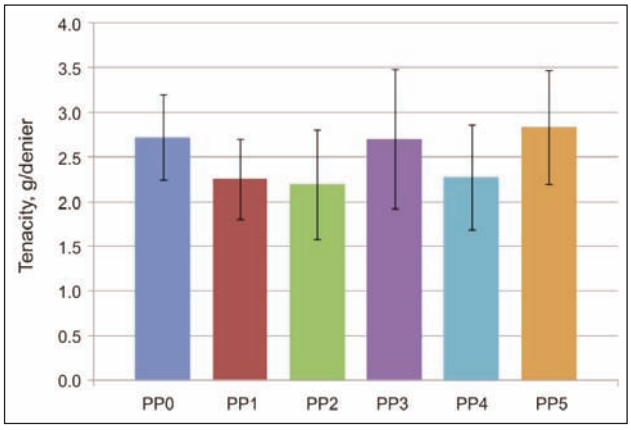

Fig. 5. Tenacity of the PP filaments

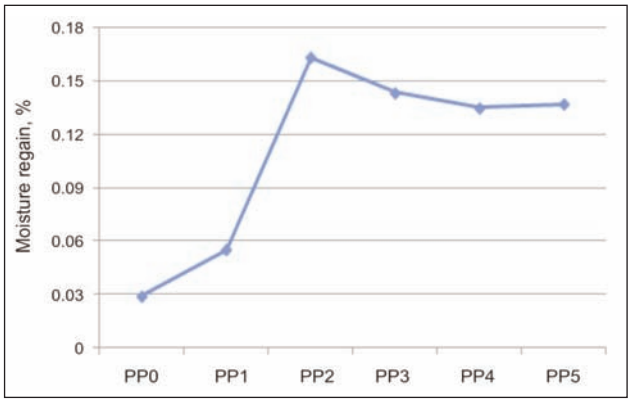

Fig. 6. Moisture absorption of the PP filaments

Colorimetric values of the PP filaments

The colorimetric values of the PP filaments are tabulated in table 4. As clearly seen in table 4, color difference (ΔE) value between PP0 and PP1 is calculated to be 2.212. But the addition of AJ, ASJ, AFJ and APJ particles increase this color difference by 190%, 180%, 150% and 176%, respectively. Yellowness (*b**) value of PP0 increase by 130.8% with the addition of 5% MAPP. The highest *b** value obtained for PP2 which have 0.5% AJ particle and 5% MAPP compatibilizer. But silane, fluorocarbon and plasma treatments of jute particles which were applied after alkali pretreatment may decrease the yellowness value of AJ particles. K/S and R% which

Table 4

THE COLOR DATA OF THE PP FILAMENTS

	ΔE (Color difference)	b*	R% (min) (400 nm)	K/S (max) (400 nm)
PP0	Control sample	1.58	55.806	0.175
PP1	2.212	3.646	68.45	0.0727
PP2	6.413	7.525	56.606	0.1663
PP3	6.193	6.146	56.79	0.1644
PP4	5.534	6.366	59.123	0.1413
PP5	6.113	6.724	52.27	0.1594

indicate the color strength and reflectance values of PP filaments are also given in table 4. It is determined that the addition of MAPP compatibilizer (PP1) increase reflectance value but decrease the color strength of PP0 filament. But the incorporation of treated jute particles decrease reflectance values whereas increase colour strength of PP1.

CONCLUSIONS

In this paper, polypropylene multi-filaments doped with modified jute particles and MAPP were manufactured and the effects of these fillers on structural characterization, tenacity and moisture absorption of the polypropylene filaments were investigated. The influence of surface treatments on compatibility of constituents and on distribution of jute particles along the filaments were also studied. It was found that the moisture absorption of filaments increase with the incorporation of MAPP agent. The addition of alkali treated jute particles extremely increase moisture absorption of the PP due to high polarity of the jute particles. However, surface coating of silane and fluorocarbon agents and surface modification with of argon plasma of the jute particles decrease the moisture absorption capacity of the PP by changing the surface character of the jute from hydrophilic into more hydrophobic. TGA analysis also pointed out that the polypropylene filaments achieved the maximum moisture absorption with the addition of alkali treated jute particles. DSC and XRD analyses showed the addition of both modified jute particles and MAPP had no significant effects on crystallographic structure of the PP due to the low content of fillers. Similar tenacity values were obtained for all of the filaments. On the other hand, the presence of fillers changes the color of PP filaments.

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Research on obtaining Calotropis gigantea floss from the pods with microwave drying

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REZUMAT – ABSTRACT

Cercetări privind obținerea de fibră de Calotropis gigantea din păstăi, cu uscare cu microunde

În cadrul procesului de obținere a fibrei din semințe de Calotropis gigantea (C. gigantea) din păstai, trebuie realizate initial uscarea și desfacerea păstăilor. În cadrul acestei lucrări, au fost studiate caracteristicile de uscare a păstăilor de Calotropis gigantea prin radiere cu microunde, folosind diferite metode de uscare, intensități ale microundelor, perioade de timp de expunere, precum și impactul acestora asupra calității fibrelor. Rezultatele experimentale au arătat că este posibilă obținerea de fibre din semințele de C. gigantea prin încălzire cu microunde. Comparativ cu uscarea la temperatura camerei sau cu aer cald, uscarea cu microunde poate îmbunătăți semnificativ eficiența, reducând timpul de uscare cu până la 5 ori. Intensitatea microundelor are o influență considerabilă asupra ratei de uscare. Totuși, tenacitatea fibrei și alungirea la rupere sunt reduse prin creșterea intensității microundelor, datorită modificărilor structurale cauzate de temperatura mare din interiorul păstăii. Comparativ cu iradierea intermitentă, rata de scădere a umidității este mai mare în cazul iradierii continue, dar temperatura internă este mai mare, ceea ce dăunează calității fibrelor. Modificarea puterii radiației, folosind o putere mai mică la început, apoi mai mare, poate scurta timpul de uscare, fără a modifica structura păstăii.

Cuvinte-cheie: Calotropis gigantea (C.gigantea), păstăi, uscare cu microunde, proces de tratare, calitatea fibrei

Research on obtaining Calotropis gigantea floss from the pods with microwave drying

In the process of obtaining Calotropis gigantea seed fibers from pods, drying and broadening pod suture are the prerequisites of harvesting fibers. In this paper, the drying characteristics of Calotropis gigantea pods on microwave radiation by using different drying methods, different microwave powers, and different time periods, are studied, and the corresponding impacts on fiber quality are analyzed. The experimental results show that obtaining Calotropis gigantea seed fibers by microwave heating the pods is feasible. Compared to natural air drying and hot air drying, the microwave drying (MW) can significantly improve the drying efficiency and reduce the drying time by more than 5 times. The microwave power has considerable influences on the drying rate. However, the fiber strength and break elongation are reduced with the increase of microwave power due to fiber structural change caused by the high temperature inside the pod. Compared with intermittent radiation, the moisture loss rate of the pods is greater in continuous radiation mode, but the pod internal temperature is higher, which is harmful to the quality of the fibers. Varying power radiation, which uses lower power first, then higher, could shorten drying time without impeding the pods' abdominal suture broadening.

Keywords: Calotropis gigantea (C.gigantea), pods, microwave drying (MW), treatment process, fiber quality

INTRODUCTION

Calotropis gigantea (C.gigantea) R.Br. Asclepiadaceae, commonly known as milkweed, is a common wasteland weed. Its fruit contains many seeds embedded in the bundle of fibers. The seed fibers are in the form of hollow tubes (fig. 1). The fiber has smooth, straight fiber contour with silky luster, and exhibits better moisture absorption property compared with cotton [1, 2]. This natural cellulose fiber is antibiotic, mothproof, mould-proof and nonallergenic [2–4]. The fiber is similar to cotton to a certain extent, in terms of fiber length (20.0–40.5 mm, average length is 34.2 mm, standard deviation is 4.249 [1]), fiber width (20–28 μm, average width is 24.9 μm, standard deviation is 3.88 μm) and fiber strength (dry breaking tenacity is 3.42 cN/dtex [1]), respectively. Its linear density is 0.93–0.97 dtex [1], which is much lower than that of cotton (The linear density of upland cotton is 1.43–2.22 dtex). To our knowledge, it is the first milkweed that was proposed for developing into textile fiber. Many literatures [2, 5–8] have reported developing milkweed blend yarn with cotton spinning system.

C.gigantea pod belongs to a kind of dehiscent fruits. Each pod contains about 0.9 g fibers. The middle-upper part of the fiber bundle is imbedded in the placenta between the leaves. The root of the fiber has an impermeable junction with the seed. As the pod becomes mature, it cracks naturally along the ventral suture, which allows the dehydration of the fibers, placenta and seeds. Then the fibers and seeds are blown away by air, which results in the reproduction of the seeds. The structure of C. gigantea pod is shown in figure 2.

Methods of manually acquiring C.gigantea seed fiber are different from that of cotton fiber and ceiba. C.gigantea pods need to be harvested when the pods are still green and prior to cracking. The postharvest

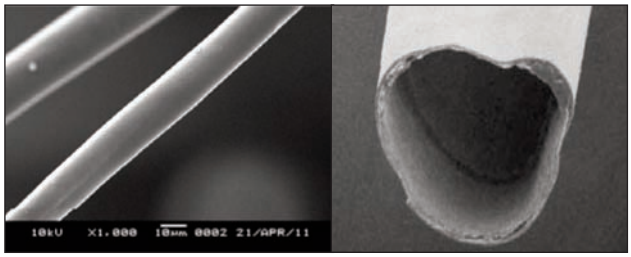

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Fig. 1. Morphology structure of C.gigantea floss: (a) longitudinal section of the floss; (b) cross section of the floss

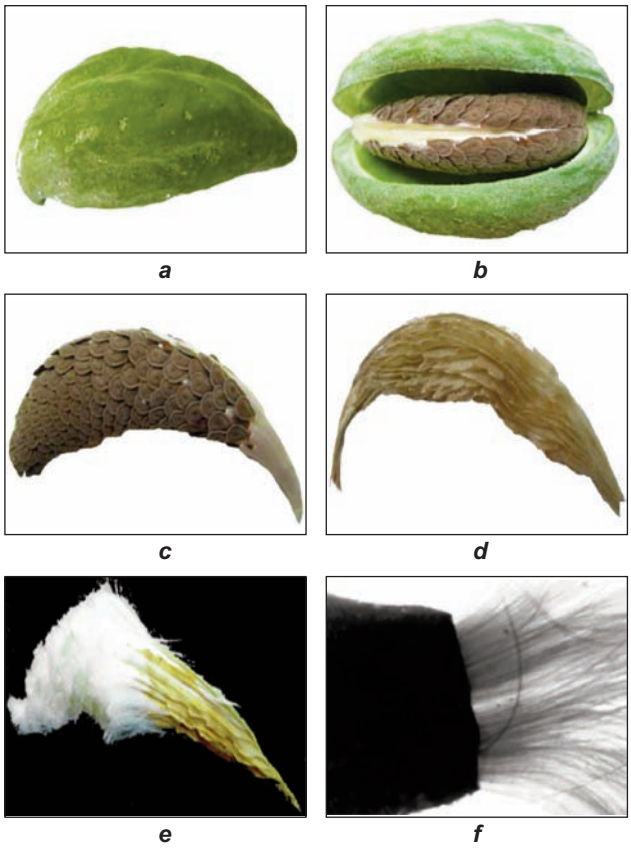

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Fig. 2. Structure of Calotropis gigantea pod: (a) Calotropis gigantea seed pod; (b) opened Calotropis gigantea follicle; (c) the things inside the pod; (d) placenta; (e) the middle-up part of fiber is imbedded in the placenta between the leaves; (f) fibers attached to seed (dry state)

pods will go through after-ripening, cracking and dehydrating processes. Then, with the help of machinery or airflow, floss-fiber and the placenta attached are separated from the pod, and so are the fibers from their seeds. The sufficient cracking and the proper dryness of the pod are of vital importance. If the openings are not big enough, the placenta cannot get out of the shell of the pod, and the fibers inside the pod cannot be entirely extracted. If the placenta, seeds and fibers are not dehydrated sufficiently, the placenta will clamp the fibers too tight, and the connection between the fibers and the seeds will be much too strong. Then it will be difficult to separate the fiber and the placenta as well as the fiber and the seed. Forced separating would lead to the breakage of the placenta and thus impact the fiber purity. In addition, pods with high moisture (mature milkweed pods freshly harvested at 300% moisture content (MC) (d.b.) or greater) are subject to enzymatic and respiratory activity [9, 10]. These activities can generate heat detrimental to floss quality [10]. Also, high MC of pods provides an excellent environment for mold development under high temperature [9] which is not desirable.

Conventional drying methods include air drying and hot air drying. Air drying is of lower cost, but is time and space consuming, inefficient and uneven. Also, a low temperature and low humidity environment in winter are unfavorable to sufficiently cracking the pods, which results in dry-shrinkage of pods. For instance, the drying process of milkweed pods in the wild usually takes a month or longer [9]. In drying bins with air flow of 0.25 $m^3/s \cdot m^2$ setting, it takes at least 48–60 hrs that the average MC decreases from approximately 66.7% to 11.1% (d.b.) [9]. Hot air drying is external heat. The wet material is dried from outside to inside which results in unevenness and discontinuity in the drying process. Thus, this is not ideal for separating floss-fiber from the pods. Besides, hot air drying has a deleterious effect upon the seed germination rate by reducing it up to 20% [10].

Unlike conventional heating, microwaves penetrate the material to be heated, and utilize volumetric heating rather than surface heating, which is faster, more uniform and more energy-efficient. Generally, power-saving reaches 30–50 % [11]. Microwave drying (MW) technology has been widely applied to the drying of timber [12] and agriculture products [13, 14] as well as the textile field such as bast fiber degumming, silk product scouring, yarn and textile dyeing, fabric finishing, textile humidity testing, slashing and fabric drying, and yarn setting [15–17].

Microwave radiant energy reduces exponentially from the outside to the inside of material. With radiation frequency of 2450 MHz (wavelength of 12.2 cm), the penetration depth can reach from a few centimeters to several decadal centimeters. C.gigantea pod is about 7–9 cm long, and 3–4 cm wide. Thus, microwave radiation can uniformly dry the hull and placenta of it. Water helps absorbing and converting microwaves to heat. With high moisture content, C.gigantea pods can be dried efficiently. However, microwave irradiation may affect the chemical and morphological structure of fibers, including some physical properties. It should be pointed out that high-power microwave (for example, 4000 W) has a negative impact on cottonseed germination rate [18]. After the seed cotton being microwaved by the high power microwave (4000 W), the strength of the cotton fiber was decreased, and the strength loss increased with increased irradiation time (0 s, 20 s, 30 s, 40 s and 50 s) [18]. The strength of the sample fiber processed by the microwave of 345 watts was higher than that of the unprocessed [19]. This suggests

that the fiber strength tends to be improved when using the appropriate microwave power.

In this paper, an attempt was made to apply microwave drying to C.gigantea dehiscent pods. The changes of pod suture width and connection strength between the fibers and the seed during the microwave processing were observed. The relationships between different parameters, which include microwave power, radiation time, processing mode, dry rate, and the pod's MC, were studied, and the corresponding impacts on the fiber quality were analyzed. In order to efficiently extract floss from pods and produce the floss on a commercial scale, the stopping point and the processing parameter which has the least impact on the fiber strength were explored. Subject to limited experimental conditions, the changes in material temperature under different processing conditions are not tested, which is part of the future work.

MATERIALS AND METHODS

Test apparatus and material

A domestic microwave oven (Galanz GF70F20N2L-DG (S0), China) with five power levels, 126, 252, 406, 567 and 700 W (2450 MHz) was used for microwave drying. The size of the microwave cavity is 20 litres. For the mass determination, an electronic scale of 0.01 g accuracy (HZ-HS-302R, China) was used. An XQ-2 electronic single fiber strength tester of 0.01 cN load measurement resolution (China) was used to test fiber tensile properties.

Sample pods were collected from wild C.gigantea plants which grew in four sample plots with different site conditions in Yunnan province, China. About 10 kg of pods were collected. Pods were naturally maturation split open or by manually cracking before being tested. The naturally cracked pods were stored at 4±0.5 ºC before they were used in experiments.

Experimental methods

All experiments of drying pods were done at room temperature and pressure, and the tensile property of seed fibers which had been conditioned at the standard temperature and relative humidity (20.0±2 ºC and 65±1 %), were measured at 20ºC, 65% RH.

Drying characteristics of different components of the pod. Ten mature pods were opened by hand. The placenta, fiber and seeds attached to the pod, were separated then weighed. Microwave radiation power was set as 252 W and power-to-weight ratio 2 w/g. The samples were quickly taken out of the oven then weighed at 2-minute intervals during drying.

Pods' drying characteristics under different microwave powers. The sample pods were separated into 5 groups with very close total weight (around 69 g), MC and maturity. The samples were placed in a single layer in the microwaveable vessel. Five microwave output powers (126 W, 252 W, 406 W, 567 W and 700 W) were investigated. The sample pods were weighed at 2 minute intervals during drying. Drying was stopped when these five sample groups reached the approximate dryness.

Influence of microwave radiation time on the tensile properties of the seed fiber. The sample pods were separated into four groups with similar dehiscent sizes, each of which weighed approximately 62 g. Four groups were continuously microwaved with 126 W for 10 min, 20 min, 30 min, and 40 min separately. Then fibers were collected from pods and the tensile properties of them were tested.

Pods' drying characteristics under different microwave power control modes (continuous and pulsed drying, constant power and variable power drying). Sample pods were separated into four groups with similar MC and maturity, each of which was approximately 75 g. Two groups were microwaved with 126 W. One group was quickly taken out and weighed at 5-minute intervals throughout radiation for continuous heating and the other group was treated by alternating 5-minute radiation and 5-minute stopping for intermittent heating. The other 2 groups were microwaved using constant and variable powers respectively and intermittent heating (alternating 5-minute radiation and 2-minute stopping). For variable power control mode, the radiation was 126 W for the first 10 minutes, then 252 W for the rest time. For constant power control mode, the power was kept at 126 W. Drying was terminated when the samples of the two groups reached approximately the same dryness.

Tensile properties of the seed fiber. For each processing type, 50 tests were performed. Fibers were randomly selected from the sample fibers. Fiber tensile strength tester was set with clamp distance 10 mm, drawing rate 10 mm/min, pre-tension 100 mg. The average breaking strength, and break elongation, etc. and the corresponding standard deviation of 50 fibers was calculated.

Identify the drying stopping point. The drying stopping point was determined by the dryness of the pod shell, the pods split width and whether the seeds and floss can be removed from the placenta by air current. The drying was conducted until the crack of the pod was wide enough to let the placenta detach, and some floss clusters on the placenta were scattered as umbrella.

Test indexes

MC of the pods in the drying experiments was calculated on a dry basis, $W(\%) = \frac{G\varphi - Gc}{Gc} \times 100$, where W (%) is the MC of the pods; $G\varphi$ (g) is the pods weight during drying process; Gc (g) is the pods weight at drying termination. The drying rate U was calculated as the quantity of the moisture removed from the pod per unit time

$$U\,(\text{g/min}) = -\frac{G_i - G_{i-1}}{T_i - T_{i-1}} \times 100 = -\frac{\Delta G_i}{\Delta T_i} \times 100,$$

where U is drying rate (g/min); ΔG_i is the i-th weight of the water evaporated(g); ΔT_i is the i-th time consumed (min).

Statistical analysis

The statistical analysis of variance (ANOVA) of the samples was performed to assess which treatments had significant effects (P≤0.05) on measured parameters using Statistical Product and Service Solutions (SPSS Statistics 17.0). Means were compared using Duncan's Multiple Range test.

RESULTS AND DISCUSSIONS

Desiccation properties of the different components of the pods

Different parts of fresh pods have different moisture contents. The shell and the placenta have high MC, which are 585.40 (±1.06) % d.b. and 855.11 (±0.95) % d.b., respectively. The floss fiber has 219.37 (±5.23) % d.b. The seed has the least MC, which is only 188.43 (±4.58) % d.b. for pods harvested in August. Thus, different components of the pods have different drying characteristics. Figures 3 and 4 show the MC and dryness rate versus time curves for microwave drying of C. gigantea pods. From the pictures, we can conclude that:

– During the first 20 minutes of microwave heating, the rate of water loss of the shell was much higher than that of the placenta with fiber and seeds attached. The initial water content (dry basis) of the shell is 2.4 times of the placenta with attachments. According to the microwave drying mechanism, the energy is absorbed by the material, the heat dissipates and the material then dries. The internal heat develops an internal pressure which helps water to come out to the surface by diffusion. Clearly the higher the MC of the material, the bigger the impact that the pressure gradient has on transferring water from material, and the faster the water flows towards the material surface. Thus, the shell reached equilibrium moisture content faster than the placenta with attachments (fig. 3), though the placenta has higher MC.
– The drying rate curves (fig. 4) show that the drying process of C. gigantea pods can be divided into 3 periods, namely, rising rate, constant rate and falling rate period. The 4–16 min period is the constant rate drying period of the shell and the 6–22 min period is that of the placenta with attachments. Approximately 67% of the water in shell and 70% in placenta are removed in this period. During the constant speed drying period, the radiant heat absorbed by pods, the water boiled off and the temperature of the pods reach relative equilibrium. When the MC of the pods becomes less than 50–60 %, the water of the inside diffuses slower than that of the surface, and the pods are in a falling rate period. At the constant speed drying period, the drying rate of the shell, which has a higher MC, is much faster than that of the placenta. While in the falling rate period, the water in the shell, which is fewer (this causes a decrease in the absorption of microwave power) and mainly exists in the form of bound water, is difficult to be transported, so the drying rate of placenta with attachments is faster than that of the shell.

Regardless of using hot air drying or microwave drying, the temperature of the material in the constant rate period cannot be very high. But in the falling rate period, the heat of vaporization becomes less, so the temperature of the material surface becomes higher. If the drying process cannot be terminated at the right point in time, the high temperature would do harm to the material. To ensure the seed fiber quality, under conditions of microwave power 252 W, power-to-weight ratio 2 w/g, and constant power drying, the pod's drying time should not exceed 22 minutes. Besides, the dehydration rate of the shell is much faster than that of the placenta. When the placenta reaches a status that fibers can be extracted, the shell has become brittle. Continuing such process could break the shell and increase the impurity of the fibers. Thus, it is very important to set a proper stopping point of drying.

The pods' drying characters under different microwave output powers

Water absorbs most of the microwave energy and more than 95% of water can be turned to latent heat of vaporization to evaporate [20]. It is clear that the higher the microwave power, the higher the drying

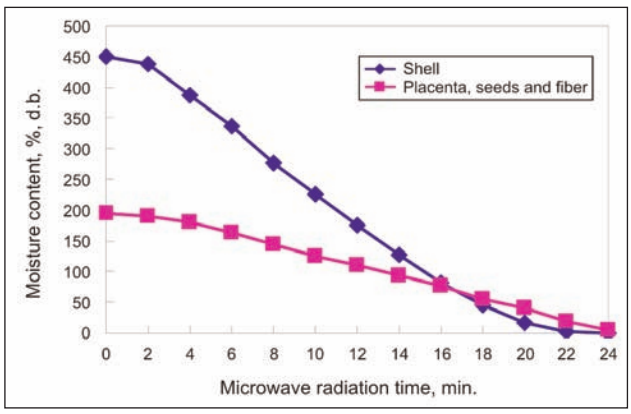

Fig. 3. Drying curves for the different components of the pods under 252 W microwave power

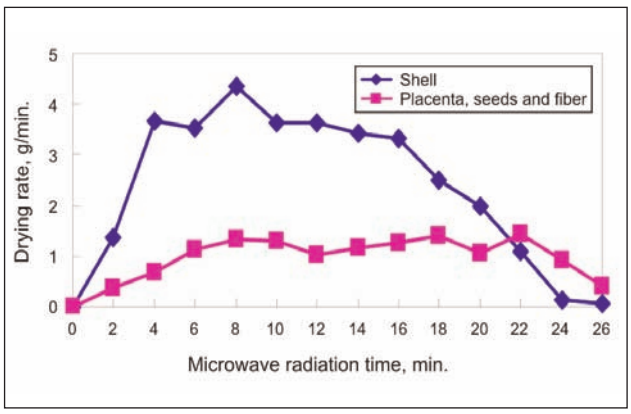

Fig. 4. Drying rate curves for the different components of the pods under 252 W microwave power

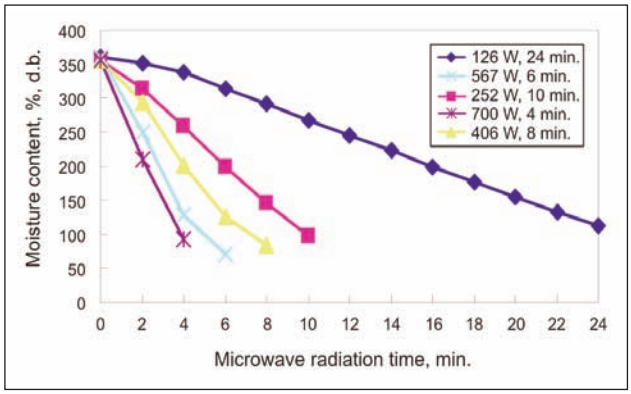

Fig. 5. Drying curves for the pods under various microwave output powers

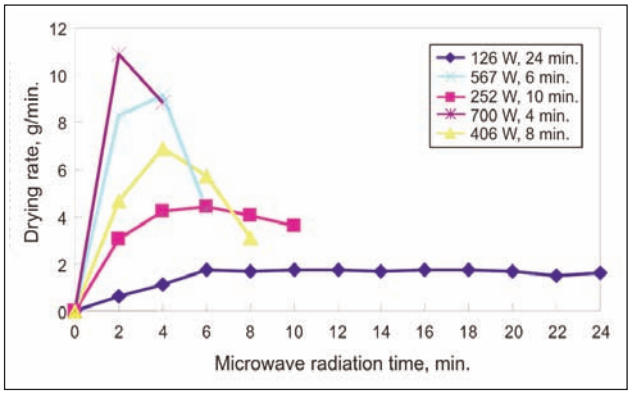

Fig. 6. Drying rate curves for the pods under various microwave output powers

rate, and the fewer the time needed for materials to reach the acceptable dryness. This scenario is also observed during C. gigantea pods' MW procedure, see figures 5–6. The times when the pods reach acceptable dryness are at the 24th, 10th, 8th, 6th and 4th minute for power 126 W, 252 W, 406 W, 567 W and 700 W, respectively. The moisture contents of the pods were reduced from 355.07–360.73 % to 70.33–112.40 % (d.b.) during the processing. By working at 700 W instead of 126 W, the drying time could be shortened by about 83%. Air drying process takes about 20 to 30 hours in summer and 72 hrs to 96 hours in wintertime in Yunnan, China, and takes even longer in rainy season and low temperature days. It takes 2–8 hours using illuminating incubator at 55 °C temperature to fully open the pod sutures and dry pods till air flow could blow the floss away. Compared to air drying and hot air drying, MW has increased efficiency significantly.

The strength of C. gigantea seed fiber after microwave radiation is less than that of the unprocessed fiber, and there is a trend towards decreased strength with respect to increased microwave power (fig. 7). The phenomenon is consistent with what it was observed during microwave processing of cotton fiber [18, 19]. With microwave power increasing from 126 W to 700 W, the average strength of fibers are decreased by 18% – a significant difference ($P < 0.0001$). Microwave radiation affects the tensile property of the fibers, which implies that microwave radiation may change the internal structure of the fibers or cause thermal degradation of the fibers. It may also decrease the polymerization degree of the cellulose. The X-ray diffraction analysis of the crystallinity of the fiber samples untreated and microwave treated 4 min with power 700 W is illustrated in table 1. It can be seen that compared with untreated fibers, the crystallinity and crystallite size as well as the degree of orientation of the treated fibers decrease. The decrease can be caused by the breakage of some molecular chains and the reorganization of macromolecular under the effect of the microwave electromagnetic field.

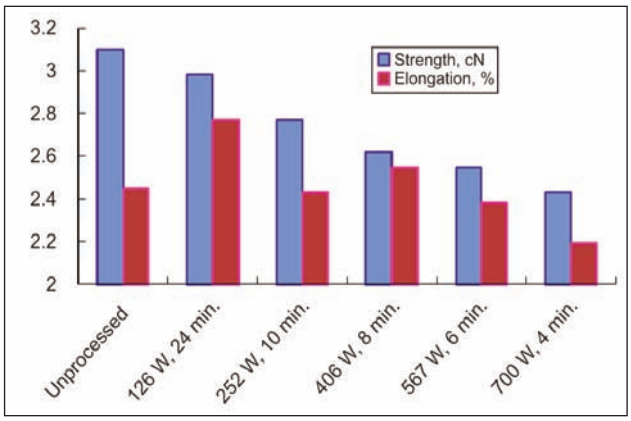

Fig. 7. Tensile property of the seed fibers under different microwave powers and process times

Table 1

CRYSTALLINITY, DEGREE OF ORIENTATION AND CRYSTAL SIZE OF FLOSS FIBER UNTREATED AND TREATED 4 MIN WITH 700 W

Samples	Crystallinity, %	Degree of orientation,%	Crystal size, nm
Untreated pod fiber	29.74	80.9	0.8 (101) 2.7 (101) 3.2 (002)
Treated pod fiber	20.28	78.3	1.0 (101) 1.9 (101) 2.8 (002)

Impacts of microwave radiation time on floss fiber

The temperature change of material by MW is closely related to the processing time. Figure 8 [11] shows the temperature variation curves with processing time. It takes only 10 minutes' processing time to increase the material temperature to about 50°C, and 35 minutes to 125 °C, under the setting microwave frequency 2450 MHZ (normal setting used in industry), electric field strength 1200 V/m, and initial MC of material 7.74 kg/kg (d.b.). The maximum temperature which ordinary textile fiber can endure is about

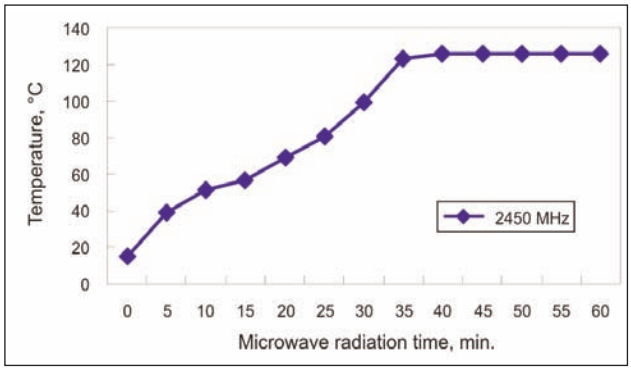

Fig. 8. Temperature changes of material with microwave radiation time at 1200 V/m electric field strength

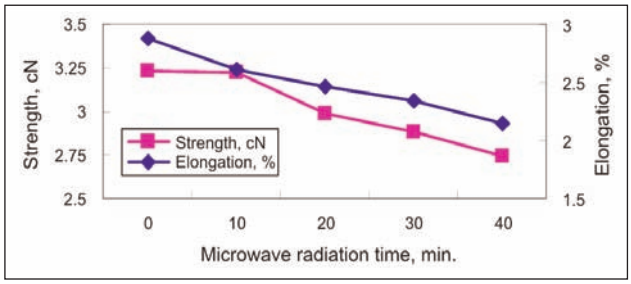

Fig. 9. Effect of radiation time on the tensile property of the floss under 126 W microwave and 2 W/g power-to-weight ratio

50–60°C [11], and temperature goes beyond this will cause damage to fibers. C.gigantea fiber has a low degree of crystallinity [1, 2, 20, 21] and a high content of hemicelluloses (about 20% [20, 21]), which makes its heat resistance smaller than that of the cotton fiber. Thus, the physical-mechanical properties of C.gigantea fiber are more vulnerable to microwave energy. Figure 9 illustrates the tensile properties of the pod fibers after being radiated with low power (126 W) for four different time durations. It shows that by increasing the microwave processing time, the fiber strength and elongation tend to fall down. Therefore, radiation time should be controlled. When deciding the processing time, both the efficiency of extracting fiber and the quality of the fibers should be taken into consideration.

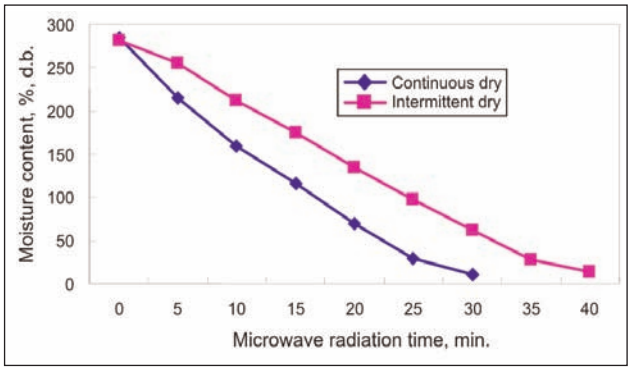

Fig. 10. Changes of MC of the pods with microwave radiation time with two process modes under 126 W microwave

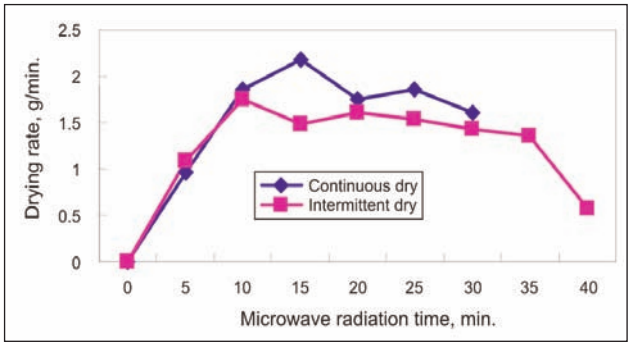

Fig. 11. Changes of dry rate of the pods with microwave radiation time with two process modes under 126 W microwave

Impact of different microwave radiation modes on the pods' drying character

Continuous dry and intermittent dry with low power

Figures 10 and 11 present the changes of C.gigantea pods' MCs and drying rates with time using two different microwave processes. It shows that compared with intermittent processing, pods reach proper MC in shorter time with continuous processing. During the first 10 minutes, the dehydration rates of the two methods are similar. While in the constant speed stage, the dehydration rate of continuous processing is larger than that of the intermittent processing, which shortens the drying time. Table 2 exhibits the comparisons of the fiber's tensile properties. Fiber breaking strength, elongation, initial module and specific breaking work under continuous processing are

Table 2

EFFECT OF MICROWAVE RADIATION ON TENSILE PROPERTIES OF THE SEED FIBER

Microwave radiation methods	Strength, cN		Elongation at break, %		Initial module, cN/dtex		Specific work of rupture, cN/dtex	
	Mean	Standard deviation	Mean	Standard deviation	Mean	Standard deviation	Mean	Standard deviation
Intermittent processing	2.83	1.20	2.29	0.64	134.78	39.33	0.04	0.025
Continuous processing	2.32	0.71	2.08	0.55	121.19	32.27	0.03	0.016
Changing rate,%	–18.02		–9.17		–10.08		–25.00	

decreased by 18%, 9%, 10%, and 25% respectively, compared with intermittent processing. It implies that by continuous drying, the pod could end up with a high temperature, which has great damage to fiber quality. To reduce the impact of processing on fiber quality, intermittent processing mode is recommended.

Constant radiation power and variable radiation power drying

Experimental results show that when using 126 W microwave heating for 10 minutes then turning to 252 W until pods' are properly dried, the time can be shortened by 42.86% compared with 126 W constant heating (fig. 12). Besides, using lower power in the first stage can avoid the shell shrinkage as well as unsatisfied opening size of the pod suture because of the quick loss of water.

Compared with hot air drying methods, microwave drying cracked pods is an efficient and power-saving way for acquiring the seed fiber, and has an antimicrobial effect on the fibers. Alone using the MW, the internal temperature of the pods can easily exceed the temperature threshold of fibers. Microwave-assisted drying at controlled temperatures, which involves using microwave energy combined with convective air, and the vacuum microwave drying which is a low temperature drying technology, has been proved to be capable of retaining the fiber quality [11, 22]. For instance, by microwave-convective drying, a significant increase in tensile strength and elastic modulus with an increase in the process temperature from 40°C to 80°C was found for flax fibers; MW with a hot air supply, which can offer uniform heating, can retain the quality of the flax fiber [22]. The viability of using above methods for drying C.gigantea pods should be explored in future work.

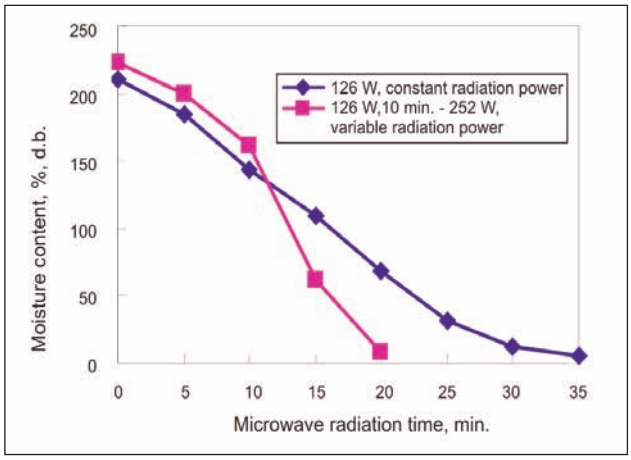

Fig. 12. Changes of moisture content of the pods with microwave radiation time with two process modes

CONCLUSIONS

C.gigantea seed fiber is a natural cellulose fiber which has a very prospective future in textile field. It is an innovative attempt to obtain such fibers from the pods by microwave drying. This study focuses on the drying characteristics of C.gigantea pods on various microwave radiation conditions and the corresponding impacts on the fiber quality. The major conclusions are as follows:

– Dehydration rate of the pods' hull is far larger than that of the placenta. The hull has already started shrinkage and becomes brittle when the placenta is dried to the acceptable degree. Brittle hulls are susceptible to crumble and thus violate the impurity of the fibers. Therefore defining the proper drying time is critical. The drying stopping point should be controlled in the constant rate period to avoid damaging fiber quality by high pod temperature.
– The drying time decreases considerably with the increase of microwave power. However, high microwave power would result in the decrease of the strength and the elongation of fibers. Thus lower power is preferred.
– Fiber strength and elongation tend to fall down with the increase of microwave processing time. So the time to radiate pods should be controlled within proper time duration. The setting of the drying parameters (drying time, microwave power and power-to-weight ratio) should be able to balance between the fiber quality and getting the fiber from pods efficiently.
– The dehydration rate of continuous microwave processing is larger than that of intermittent processing, but the former has greater damage to the fiber quality because of the high temperature of pods. Therefore continuous MW exposure should be avoided. First using lower microwave power then turning to higher can shorten the drying time without impeding the broadening of the pod sutures.
– Using MW alone, the internal temperature of pods can easily elevate to a high level which will cause the loss of fiber strength. Microwave-convective drying and vacuum microwave drying have proved to better retain the fiber quality. The viability to dry dehiscent pods of C.gigantea should be studied.

ACKNOWLEDGEMENT

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Effect of structural parameters on the sound absorption properties of warp-knitted spacer fabrics

SI CHEN HAI-RU LONG

REZUMAT – ABSTRACT

Efectul parametrilor structurali asupra proprietății de absorbție acustică a tricoturilor din urzeală distanțate

În cadrul acestui studiu au fost realizate mai multe variante de tricoturi din urzeală distanțate, având diferiți parametri structurali, cum ar fi gradul de înclinare, diametrul firului de spațiere și structura stratului de suprafață, în vederea investigării relației dintre proprietățile de absorbție acustică și parametrii structurali. Tricoturile din urzeală spațiale au fost investigate cu ajutorul unui tub de impedanță, referitor la proprietatea acestora de absorbție acustică. Rezultatele obținute indică faptul că parametrii structurali au o influență semnificativă asupra performanței de absorbție acustică a tricoturilor din urzeală distanțate. Tricoturile realizate cu un grad de înclinare al firului de spațiere mai mic, au o capacitate mai mare de absorbție acustică, comparativ cu materialul corespondent. În contrast, tricoturile cu o grosime mai mare și fire de distanțare mai groase, au demonstrat o capacitate de absorbție acustică optimă. Tricoturile realizate cu stratul de suprafață mai apropiat au demonstrat o și mai bună capacitate de absorbție acustică. Rezultatele obținute pot oferi indicații prețioase în ceea ce privește alegerea parametrilor structurali și studiul proprietăților tricoturi din urzeală spațiale.

Cuvinte-cheie: absorbție acustică, tub de impedanță, tricoturi din urzeală distanțate, parametri structurali

Effect of structural parameters on the sound absorption properties of warp-knitted spacer fabrics

In this study, a series of warp-knitted spacer fabrics conducted of different structural parameters including the inclination degree of spacer yarn, thickness, surface layer structure and diameter of spacer yarn, have been successfully manufactured in order to investigate the relationship between sound absorption properties and structural parameters of warp-knitted spacer fabrics. The spacer fabrics were characterized for sound absorption behaviors by using an impedance tube. The findings obtained indicate that the structural parameters have significant influence on the sound absorption performance of spacer fabrics. The fabric made with smaller inclination degree of spacer yarn has superior sound absorption abilities as compared to the corresponding fabric. In contrast, the fabrics produced by higher thickness and coarser spacer yarn exhibit preferable sound absorption performance. Furthermore, the fabric conducted of closer surface layer possesses better sound absorbability. All the results could offer valuable guidance on the selection of structural parameters and properties study of warp-knitted spacer fabrics.

Keywords: sound absorption properties, impedance tube, warp-knitted spacer fabrics, structural parameters

INTRODUCTION

Recently, the reduction of noise damage has drawn a lot of attention from scientists and engineers, since the noise pollution has great adverse effect on the environment, human health and social economy [1]. In general, the methods of controlling noise can be divided into two ways: active control and passive control [2]. The former means that the noise can be reduced at the locations of noise sources, but only the noise of a narrow frequency range can be controlled by this way. The passive control is accomplished by introducing sound-absorbing materials, which can be used to absorb noise over a large frequency range.

Porous materials are typical passive noise-controllers widely used for sound absorption. The sound absorption abilities of these materials depend on the sound waves frequency and increase with the increase of sound waves frequency. The mechanisms of sound absorption in porous materials are basically the viscous effects due to the internal friction between the material and airflow, thus the heat loss due to the friction [3].

As a kind of porous materials, textile structures such as nonwoven, woven and knitted fabrics have attracted great attention for sound absorption applications due to their low-cost and low environment impact [4–6]. Nowadays, a number of investigations on nonwovens in terms of their sound-absorbing behaviors and theoretical analyses have been conducted [4, 7–8]. However, despite their excellent sound absorption properties and low-cost, the sound absorption materials made with nonwoven textiles cannot meet the end-use requirements of complex-shape performance [9]. Thus, the nonwoven textiles are usually draped with woven or knitted fabrics. Liu et al. [10] introduced the sound absorption behaviors of weft and warp-knitted spacer fabrics and their combinations. It is found that the sound absorbency can be efficiently increased at low sound frequency by combining the weft and warp-knitted spacer fabrics with different method. Dias et al. [11] focused on the sound absorption properties and theoretical modeling

Table 1

CHAIN NOTATIONS FOR OUTER-LAYER STRUCTURES	
Structure	**Chain notation**
Locknit	GB1:1-0 0-0/ 3-2 3-3// fully threaded GB2:2-1 1-1/ 1-0 0-0// fully threaded GB5:0-0 2-1/ 1-1 1-0// fully threaded GB6:3-3 1-0/ 0-0 3-2// fully threaded
Chain+Inlay	GB1: 0-0 0-0/5-5 5-5// fully threaded GB2: 1-0 0-0/1-0 0-0// fully threaded GB5: 0-0 1-0/0-0 1-0// fully threaded GB6: 0-0 5-5/5-5 0-0// fully threaded
Rhombic Mesh	GB1:1-0 0-0/1-2 2-2/2-3 3-3/2-1 1-1// 1 fully 1 empty threaded GB2:2-3 3-3/2-1 1-1/1-0 0-0/1-2 2-2// 1 fully 1 empty threaded GB5:1-0 1-0/0-0 1-2/2-2 2-3/3-3 2-1// 1 fully 1 empty threaded GB6:2-3 2-3/3-3 2-1/1-1 1-0/0-0 1-2// 1 fully 1 empty threaded
Hexagonal Mesh	GB1: (1-0 3-3/3-2 1-1)×2/(1-0 3-3/3-2 4-4)/(5-4 3-3/3-2 4-4)×2/5-4 3-3/3-2 2-2// 2 empty 2 thread GB2: (5-4 3-3/3-2 4-4)×2/(5-4 3-3/3-2 1-1)/(1-0 3-3/3-2 1-1)×2/1-0 3-3/3-2 4-4// 2 empty 2 thread GB5: (1-1 1-0/3-3 3-2)×3/(4-4 5-4/3-3 3-2)×3// 2 empty 2 thread GB6: (4-4 5-4/3-3 3-2)×3/(1-1 1-0/3-3 3-2)×3// 2 empty 2 thread

of weft-knitted spacer fabrics, consisting of two plain knitted outer layers and a spacer area made of multifilament yarns. The results showed that these fabrics exhibited reasonable absorbability at mid-high frequency, but in a narrow sound frequency range. Referring to the above researches, it is known that the spacer fabrics obtain great potential to be used as sound absorbers due to their characteristics of designable structures and appearances.

This paper reports the sound absorption properties of warp-knitted spacer fabrics. With an attempt to discuss the effect of structural parameters on the sound absorption properties of fabrics, a series of warp-knitted spacer fabrics with different structure parameters including the inclination degree of spacer yarn, thickness, spacer yarn's diameter and surface layer structure, have been produced. The aim is to find out a regular pattern for tailoring warp-knitted spacer fabrics with promising sound absorption properties.

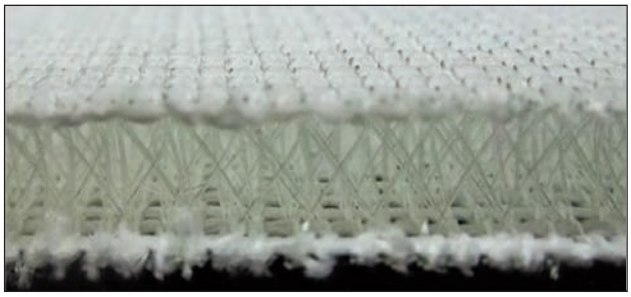
EXPERIMENTAL

Preparation of warp-knitted spacer fabrics

Nine types of warp-knitted spacer fabrics were manufactured by using double-needle-bar Raschel warp knitting machine. The PET monofilament of 0.2 mm and 0.16 mm in diameter were used as spacer yarns, and 300D/96F PET multifilament was used to knit the surface layers of fabrics. Four different outer structures, i.e. Locknit, Chain+Inlay, Rhombic Mesh and Hexagonal Mesh were introduced for knitting outer layers. The chain notations for each outer layer structure are listed in table 1. Four types of spacer yarns were used to connect the two surface layers with different lapping movement of spacer yarn. The detailed spacer yarns are listed in table 2. The structural parameters of warp-knitted spacer fabrics are shown in table 3. Furthermore, the front and right view of WSF_2, which is chosen as the representative of nine samples, are provided in figure 1.

Table 2

DETAILS OF SPACER YARNS		
Code	**Diameter (mm)**	**Lapping movement**
I	0.2	GB3:1-0 2-1/2-1 1-0// 1 full 1 empty GB4:2-1 1-0/1-0 2-1// 1 empty 1 full
II	0.2	GB3:1-0 3-2/3-2 1-0// 1 full 1 empty GB4:3-2 1-0/1-0 3-2// 1 empty 1 full
III	0.2	GB3:1-0 4-3/4-3 1-0// 1 full 1 empty GB4:4-3 1-0/1-0 4-3// 1 empty 1 full
IV	0.16	GB3:1-0 4-3/4-3 1-0// 1 full 1 empty GB4:4-3 1-0/1-0 4-3// 1 empty 1 full



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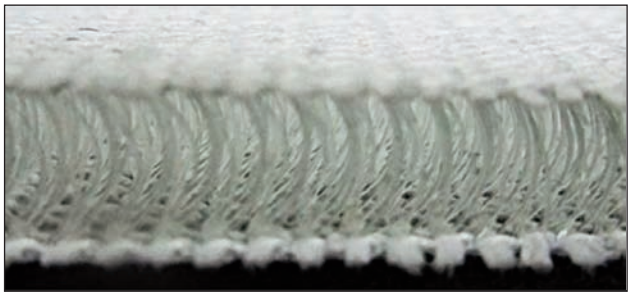


b

Fig. 1. The front and right view of WSF_2

Table 3

STRUCTURAL PARAMETERS OF WARP-KNITTED SPACER FABRICS

Sample	Thickness (mm)	Course-wise density (w/cm)	Wale-wise density (c/cm)	Area density (g/m^2)	Surface layer structure	Spacer yarn	Porosity (%)
WSF_1	7.65	6.99	5.65	891.7	C	I	91.56
WSF_2	7.68	7.07	5.57	911.8	C	II	91.29
WSF_3	7.72	6.83	5.65	919.2	C	III	90.99
WSF_4	7.71	7.15	5.70	760.4	C	IV	92.21
WSF_5	5.64	6.68	5.41	820.3	C	II	89.47
WSF_6	10.62	7.02	5.61	1027.1	C	II	92.29
WSF_7	7.59	6.93	5.51	765.4	H	II	92.81
WSF_8	7.62	7.06	5.84	776.8	R	II	91.78
WSF_9	7.64	6.97	5.82	936.8	L	II	92.58

Note: 'C', 'H', 'R' and 'L' represent Chain+Inlay, Rhombic Mesh, Hexagonal Mesh and Locknit, respectively.

Sound absorption tests

The transfer function method [12] was employed to measure the sound absorption coefficient of fabrics by using impedance tube (figure 2). The mechanism of impedance tube is the calculation of the transfer function between the sound pressures, Mic1 and Mic2. The tests were conducted in an environment of 23 °C and 65% relative humidity according to the ISO 10534-2:1998 standard, and the impedance tube is made by Beijing BSWA Technology Co., Ltd.

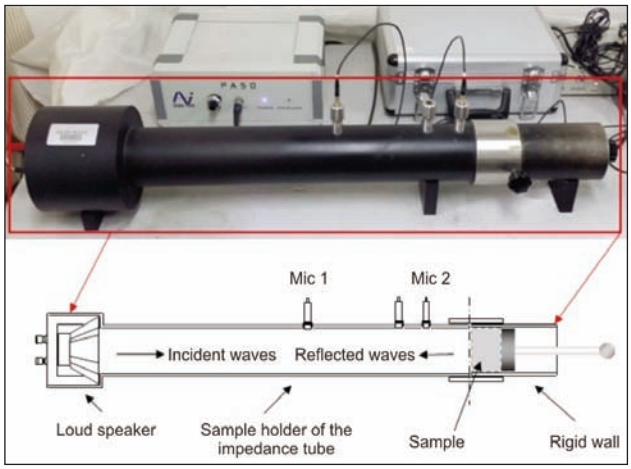
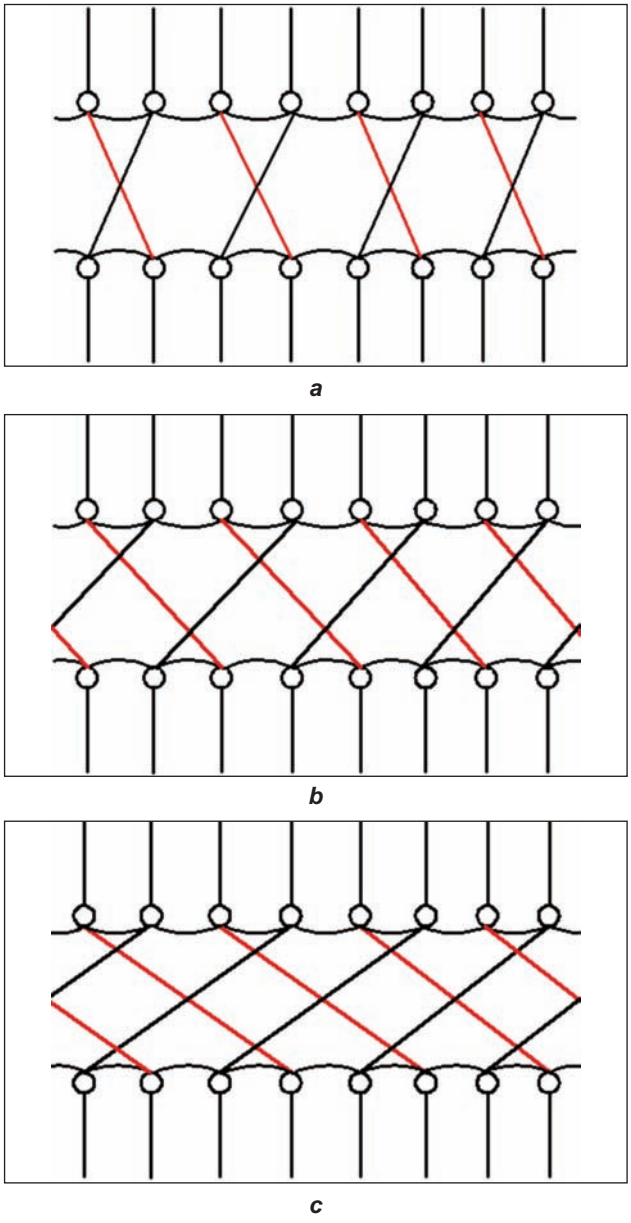


Fig. 2. Sound absorbency measurement instrument based on transfer function technique

RESULTS AND DISCUSSIONS

Effect of the inclination degree of spacer yarns on the sound absorption properties

Three spacer fabrics (WSF_1, WSF_2 and WSF_3) with different number of needles under-lapped by spacer yarns, i.e., 1-0/2-1, 1-0/3-2, and 1-0/4-3, are involved for comparison study. Figure 3 (a), (b) and (c) show the spacer yarn lapping movements of 1-0/2-1, 1-0/3-2, and 1-0/4-3, respectively. It is evident that the inclination degree of spacer yarn decreases as the number of needles under-lapped by spacer yarns



a

b

c

Fig. 3. The lapping movements of spacer yarn (the red line represents the lapping movement of GB3, while the black line displays the lapping movement of GB4)

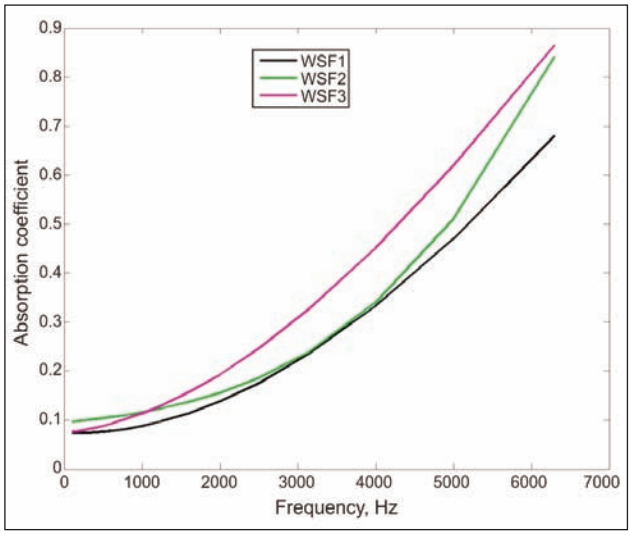


Fig. 4. Sound absorption coefficient for WSF_1, WSF_2 and WSF_3

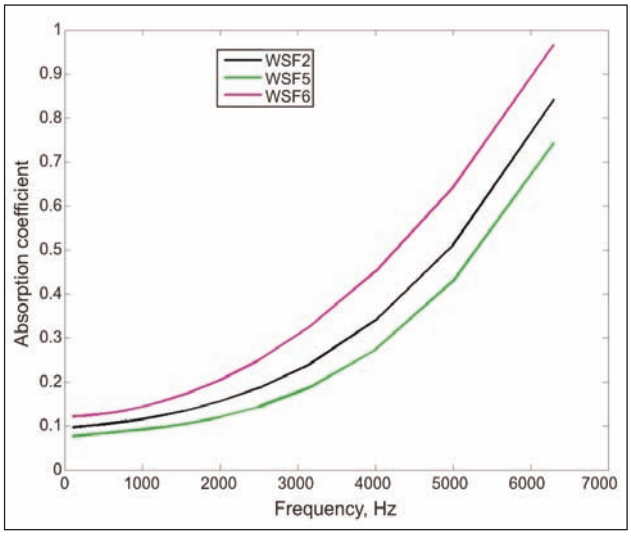


Fig. 5. Sound absorption coefficient for WSF_2, WSF_5 and WSF_6

increases. Meanwhile, the other structural parameters of these three fabrics are nearly the same.

The relationship between frequency and absorption coefficient at number of needles spacer yarn underlapped is shown in figure 4. It can be seen that the absorption coefficient is bound to change with the changes of spacer yarn inclination degree and the difference is significant. Furthermore, the absorption coefficients increase with the increase of frequency, which exhibits the typical sound absorption behaviors of porous materials. Referring to figure 4, it is clear that WSF_3 and WSF_1 have the highest and lowest absorption coefficient, respectively, while WSF_2 exhibit moderate performance on all the occasions. The results indicate that the sound absorption abilities of fabrics decrease with increasing the inclination degree of spacer yarn. The reasons can be explained as follows: first, when the sound waves reach the internal part of fabrics, the paths of scattering, reflection and refraction of the sound waves happened in them are different due to the different lapping movements of spacer yarn, resulting in varied dissipation degree of sound waves energy. Second, the smaller inclination degree of spacer yarn increases the contact area and friction between sound waves and fibers, leading to an increase of energy dispersion of sound waves. Third, the smaller inclination degree of spacer yarn makes the sound waves more difficult to reach the bottom face-layer of fabric, indicating that more sound waves energy can be consumed for the fabric conducted of smaller inclination degree of spacer yarn.

Effect of the thickness on the sound absorption properties

The sound absorption coefficients of WSF_2, WSF_5 and WSF_6 increase as their thicknesses increase, as shown in figure 5. For the same spacer yarn type and outer layer structure, the sound absorption coefficient of fabric conducted with a thickness of about 10 mm is larger than that of the fabrics of about 7 and 5 mm thick. The tendency shows great agreement with results of previous studies, the thicker the materials, the better sound absorption performance. The reason is attributed to the fact that the sound waves transmit mostly through the thicker materials and they can be absorbed along the air path and through the materials. In addition, the air layer increases with increasing the distance between the upper and bottom face-layer, resulting in the increase of vibration and energy consumption of sound waves. Therefore, the increase of fabric thickness could enhance the sound absorption performance of fabrics significantly.

Effect of the surface structure on the sound absorption properties

The distribution, binding condition and spacer yarn's inclination angle are influenced by the outer layer structure of spacer fabric since the monofilaments in the spacer area are bound by multifilament loops in the outer layer. In this section, four fabrics (WSF_2, WSF_7, WSF_8 and WSF_9) with different outer layer structures, i.e., Chain+Inlay, Rhombic Mesh, Hexagonal Mesh and Locknit are introduced to study the effect of fabric surface structure on the sound absorption properties of fabrics. These samples have the same number of underlapped needles for spacer yarns and nearly the same thickness. Figure 6 shows each outer layer structure. It has been known that the surface layer structures could slightly influence the outer layer density and inclination angle of spacer yarn, although these parameters were kept constant during the knitting process.

The sound coefficient diagram (figure 7) indicates that fabrics made of different surface layer structures exhibit different sound absorption properties and the sound absorption behaviors show great variation with the changes of face structures. It can be found that the fabric with closer outer layer structure possesses higher sound-absorbing coefficient as compared to

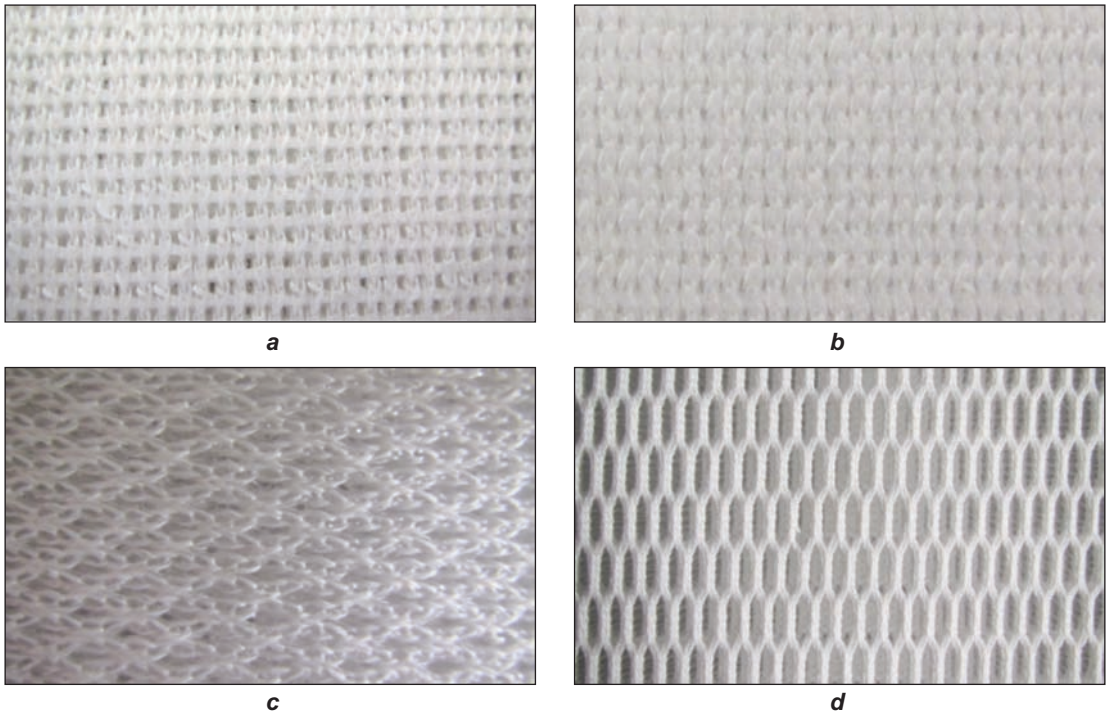


Fig. 6. The surface layer structures:
a – Chain+Inlay; *b* – Locknit; *c* – Rhombic Mesh; *d* – Hexagonal Mesh

fabric conducted of opener surface layer structure. In another word, the sound absorption abilities of fabrics increase as the outer layer structures become closer. This can be explained by the law of mass action [13], which indicates that the quality of fabric per unit area and the propagation path and energy dispersion of sound waves in fabric would increase with increasing the apparent density of fabric. Obviously, the closer outer layers have higher apparent densities as a result of the better sound absorption performance. Additionally, the closer surface layers exhibit higher fiber volume fraction, leading to more friction and energy consumption of sound waves as well.

Effect of the diameter of spacer yarn on the sound absorption properties

To investigate the influence of spacer yarn's diameter on the sound absorption behaviors, two samples (WSF_3 and WSF_4) with the same lapping movement of spacer yarn, but with different spacer yarns' diameters (0.2 mm and 0.16 mm), are employed in this regard. And the thickness and outer layer densities of these two fabrics are very close.

Figure 8 shows the acoustical performances of these two fabrics. It is realized that these two fabrics exhibit different sound absorption behaviors and the difference is great. Fabric made with coarser spacer yarn exhibits higher sound absorption coefficient, compared

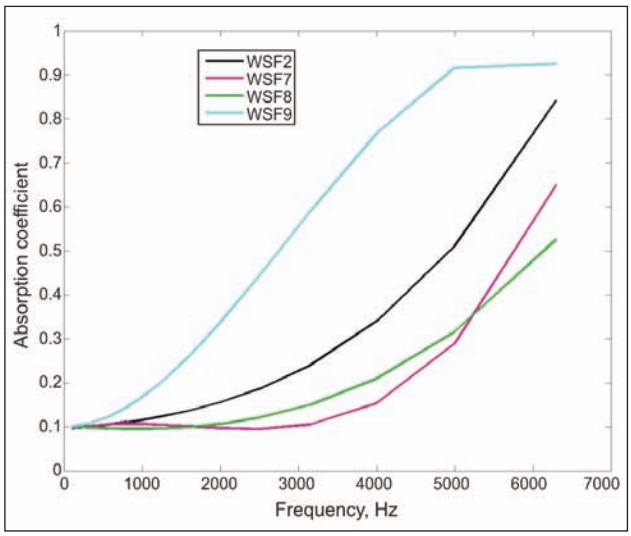


Fig. 7. Sound absorption coefficient for WSF_2, WSF_7, WSF_8 and WSF_9

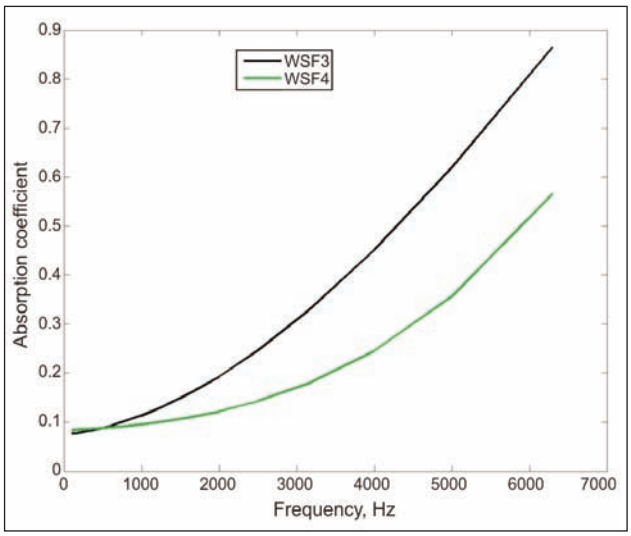


Fig. 8. Sound absorption coefficient for WSF_3 and WSF_4

to fabric made with finer spacer yarn on all the frequency. The reason can be explained by the fact that the apparent density of WSF_3 is higher than that of WSF_4. According to the law of mass action, WSF_3 obtains superior sound absorbability as compared to WSF_4. On the other hand, the coarser spacer yarn increases the contact area between the spacer yarn and sound waves as a result of the increase of friction between spacer yarn and sound waves. This results in further energy dissipation of sound waves.

CONCLUSIONS

The study shows the relationship between the sound absorption properties and the structural parameters of warp-knitted spacer fabrics. The results obtained indicate that the structural parameters have strong influence on the sound absorption performance of spacer fabrics. The spacer fabric made with smaller inclination degree of spacer yarn has superior sound absorption abilities as compared to the corresponding fabric. In contrast, the fabrics produced by higher thickness and coarser spacer yarn exhibit preferable sound absorption performance. Furthermore, the fabric conducted of closer surface layer possesses better sound absorbability when compared to the corresponding fabric. All the findings offer valuable reference function on the structure optimization and properties analysis of the warp-knitted spacer fabrics.

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Strength distribution of PMMA plastic optical fiber

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REZUMAT – ABSTRACT

Forța de distribuție a fibrelor optice din plastic PMMA

Uniformitatea rezistenței mecanice ale fibrelor optice din plastic (POF) este semnificativă pentru aplicațiile acestora în textile luminiscente, acestea fiind investigate cu ajutorul dispozitivului Instron. Rezultatele indică faptul că atât distribuția Weibull cu doi parametri, cât și distribuția Weibull cu trei parametri ar putea fi utilizate pentru evaluarea defectelor de rezistență mecanică și a comportamentului elastic-plastic al fibrelor optice POF. Dependența rezistenței la tracțiune de lungimea fibrei POF poate fi estimată prin modelul de distribuție Weibull cu trei parametri.

Cuvinte-cheie: fibră optică din plastic; forța de distribuție; distribuție Weibull; puterea medie a fibrei

Strength distribution of PMMA plastic optical fiber

Mechanical tensile failure of naked plastic optical fiber (POF) is significant for its applications in shining textiles and was investigated by Instron. The results indicate that both two parameter Weibull distribution and three parameter Weibull distribution could be utilized for the evaluation of tensile failure and the elastic-plastic behavior of POF. The dependence of tensile strength on fiber length of POF could be estimated by the model of three parameter Weibull distribution.

Keywords: plastic optical fiber; strength distribution; Weibull distribution; mean fiber strength

Naked plastic optical fibers (POFs) display a broad strength distribution due to severe flaws on surface, non-uniform quality of filaments, components of core and cladding, and interface properties between them. It is well known that it is hard to manufacture POFs with diameter smaller than 0.1 mm; during the complex manufacturing processes, a lot of imperfections can be found in the POFs, leading to great optical attenuation for end products. For special lighting design in textiles such as the soft shining fabrics [1–3] and active safety textiles [4], it is significant to understand the mechanical properties (such as tensile properties, flexibility, fatigue behavior and mechanical deformation) for POFs utilized as the raw materials (like floats, warps and wefts). Previous researches have exhibited that there are critical gauge-length and strain-rate dependences on fiber strength [5–7] following a statistical distribution. Various modifications have made great efforts to the behavior of classical Weibull distribution for fiber strength [8–9].

In this contribution, tensile tests with different gauge length were conducted to measure the tensile properties of POFs, and then Weibull strength distribution was applied to analyze the effects of fiber length of POFs on fiber tensile strength and estimate the statistical distribution of fiber strength.

EXPERIMENTAL

Materials

The polymethyl methacrylate (PMMA) plastic optical fibers (POFs) with 0.75 mm diameter were provided by Grace POF Co., Ltd. from Taiwan. The core material of POFs is PMMA and the cladding material is the blend of PMMA and Teflon. The refraction indices of fiber core and cladding are 1.49 and 1.42, respectively. The limit of bending radius of POFs is eight times of fiber diameter, which is 6 mm.

Methods

The fiber tensile strength distribution can be generally derived from these assumptions [10–11]: fiber facture happens to some specific place with catastrophic flaw and fracture probabilities at individual places are mutually independent; fracture mechanism can be characterized by the distribution function $P(\sigma)$.

It is widely recognized and accepted that fiber strength can be described by three-parameter Weibull distribution:

$$P(\sigma) = 1 - \exp\left[-\frac{l}{l_0}\left(\frac{\sigma - A}{B}\right)^C\right] \quad (1)$$

where P is the probability of fiber failure, σ is the applied strength, l is the fiber length, l_0 is the reference length. A, B and C are Weibull parameters, A is the limited strength representing Weibull shift, B is the Weibull scale, C is the Weibull shape.

If $A = 0$, equation (1) turns into two-parameter Weibull distribution:

$$P(\sigma) = 1 - \exp\left[-\frac{l}{l_0}\left(\frac{\sigma}{B}\right)^C\right] \quad (2)$$

The most direct and simple experimental method to obtain Weibull parameters is the single fiber test with large number. The failure probability is obtain as follows,

$$P = \frac{i - 0.3}{N + 0.4} \quad (3)$$

where N is number of measurements. The values of fiber tensile strength are arranged in a rising order.

Parameters A, B, C can be estimated from the following equations:

$$C = \frac{\ln(2)}{\ln(m_1 - m_2) - \ln(m_2 - m_4)} \quad (4)$$

$$A = \frac{m_1 \cdot m_4 - m_2^2}{m_1 + m_4 - 2m_2} \quad (5)$$

$$B = \frac{m_1 - A}{\Gamma(1 + 1/C)} \quad (6)$$

where $\Gamma(x)$ is the Gamma function. m_r is the so-called Weibull sample moment which can be defined as:

$$m_r = \sum_{i=0}^{N-1} (1 - i/N)^r (x_{i+1} - x_i) \quad (7)$$

where $x_{(0)} = 0$ when $i = 0$.

For two-parameter Weibull distribution:

$$C = \frac{\ln(2)}{\ln(m_1) - \ln(m_2)} \quad (8)$$

Weibull parameters can be calculated from the linear fitting function of equation (1), that is $y = ax+b$. Here: $y = \ln[-(l_0/l)\ln(-P_i)]$, $x = \ln[(\sigma_i) - A]$, $a = C$ and $b = -C\ln(B)$.

The mean fiber strength $E(\sigma)$ can be predicted:

$$E(\sigma) = A + B(l/l_0)^{-1/C}\Gamma(1 + 1/C) \quad (9)$$

The stress-strain measurements of POFs were carried out to evaluate the tensile properties by an Instron-4411 tester at 27 °C temperature and 65% relative humidity. The gauge length was designed as 30, 50, 75, 100, 150 and 200 mm, the testing speed was set as 100 mm/min.

RESULT AND DISCUSSIONS

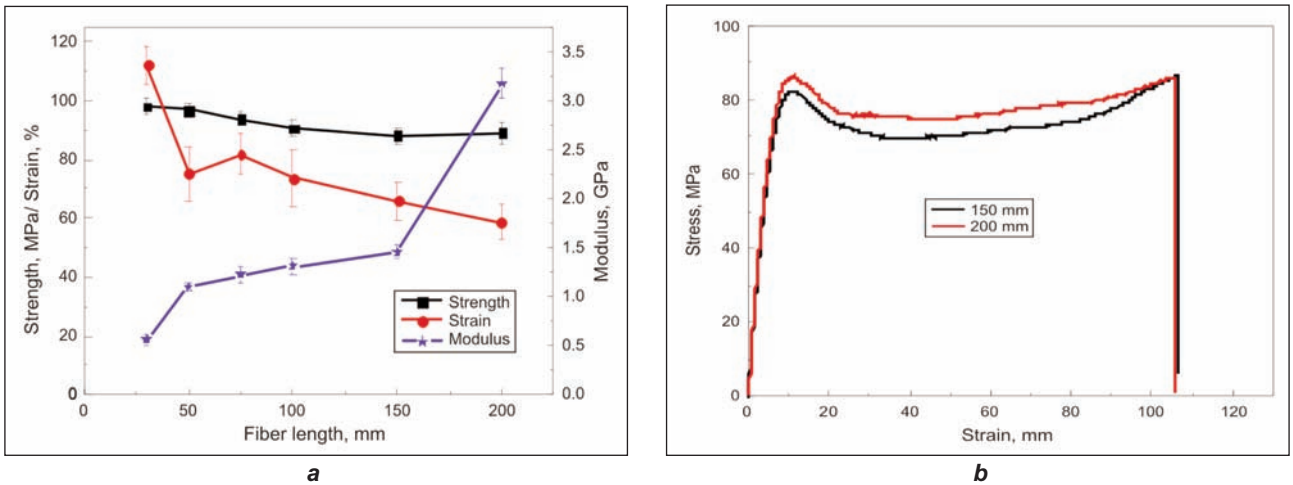
Experimental data of tensile testing are presented in figure 1 *a*, the results show that with the increase of fiber length, the strength drops slightly and strain decreases markedly while the modulus increases.

The trend in tensile strength might be explained by the weakest-link theory. The surface flaws occur with a statistical nature increase with large surface area, which leads to small strength. The crosshead speed for all fibers with six lengths is the same, that is to say, the extension rate for each fiber keeps constant, as a result, longer fiber initiates lower strain rate as expected. The results of fiber modulus are surprising because it is contrary to the widely accepted assumption that the material modulus is intrinsic property and should be a constant. In present work, it points out that the longer the POF, the higher the fiber modulus, the same tendency of the mechanical properties was found in [6]. Longer fiber appears to exhibit greater stiffness. It might reveal that the increases in both strength and strain with smaller fiber length are attributed to the accumulation of each point in POF, or distributed over the whole mass of POF. Modulus is changed as a result of dissimilar increases in strength and strain. The reason for such behavior might be not only related to the non-linear ductile properties (bi-linear curve with an obvious "knee" shown in figure 1 *b* of POF, but also relevant to the interface properties between core and cladding of POF (shown in figure 2) and the visco-elastic behavior of the whole fiber.

It is difficult to figure out specific contributions of each part, more measurements should be considered to make it clear. As we see in figure 2, the core is the main part of POF and plays a significant role in mechanical properties. However, the cladding should not be neglected due to different material (core is pure PMMA and cladding is made of PMMA and Teflon).

The experimental and theoretical results from figure 3 show that it is almost coherent for the strength distribution of POFs with different fiber lengths when fiber length is the same as reference length. The related Weibull parameters are given in table 1.

Weibull strength distribution can have a good fit for most of our experimental data in the case that the fiber length equals to the reference length, but still some fitting results are not perfect enough, e.g. fitting



a *b*

Fig. 1. *a* – Tensile properties of POFs; *b* – Typical curve of stress versus strain of POFs

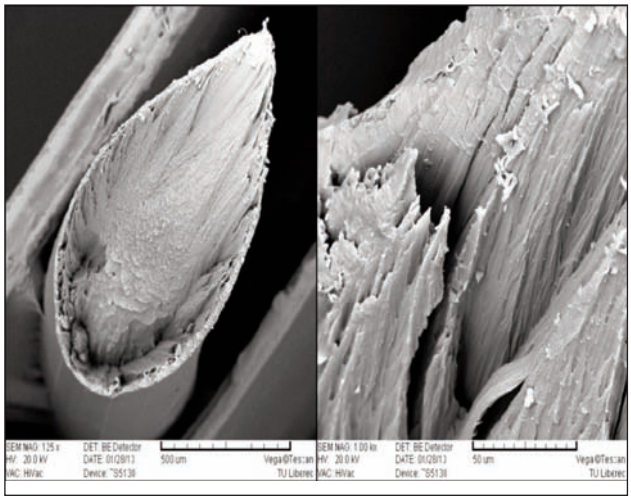


Fig. 2. SEM images of tensile failure of POFs

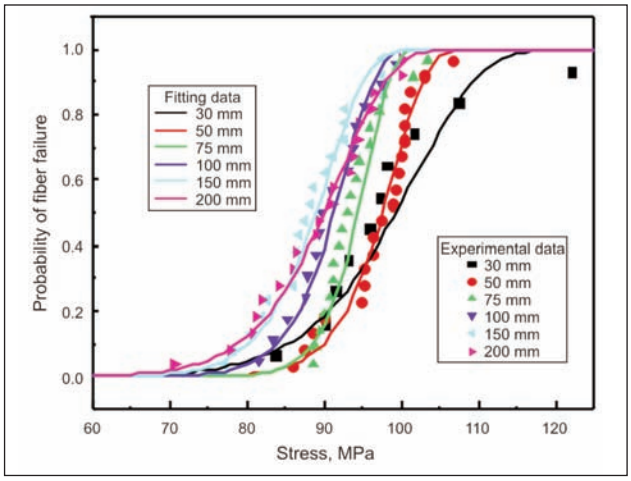
Table 1

PARAMETERS FOR WEILL DISTRIBUTION OF POFS AT 100 %/MM STRAIN RATE ($l = l_0$)

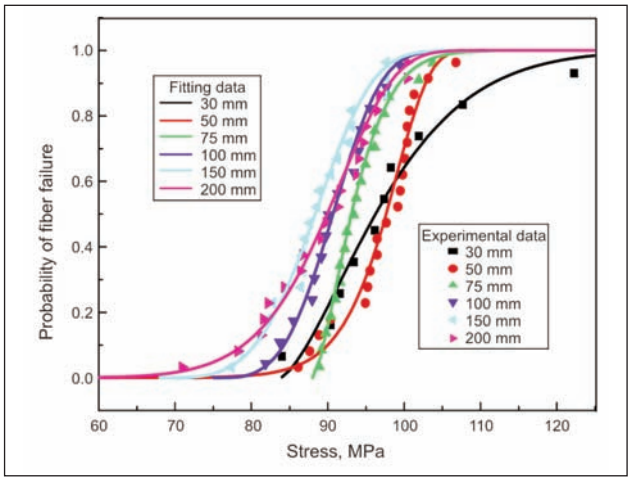
Length (mm)	Shift *A* (MPa)	Scale *B* (MPa)	Shape *C*
30		102.2859	12.4116
	83.7037	15.998	1.5042
50		99.1633	23.1491
	–23.8617	123.0419	28.9377
75		95.6498	29.3814
	87.975	6.5356	1.4958
100		92.8518	22.4366
	74.9636	17.3996	3.5826
150		90.834	18.0758
	67.1485	23.2067	4.0403
200		92.3976	14.2382
	26.0789	66.2277	9.9661

curves for 30 mm and 50 mm fiber lengths with two parameter Weibull distribution in figure 4,*a*.

Three-parameter Weibull distribution can be not only used to estimate the strength scatter, but also used to predict the dependence of strength on fiber length. The shift parameter *A* is the lower limit of strength

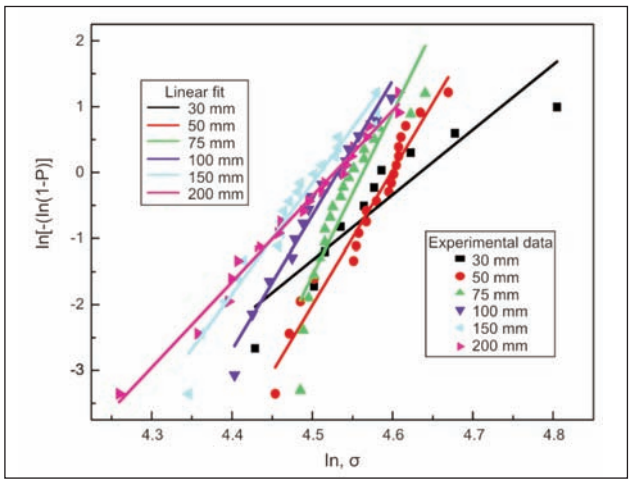


a

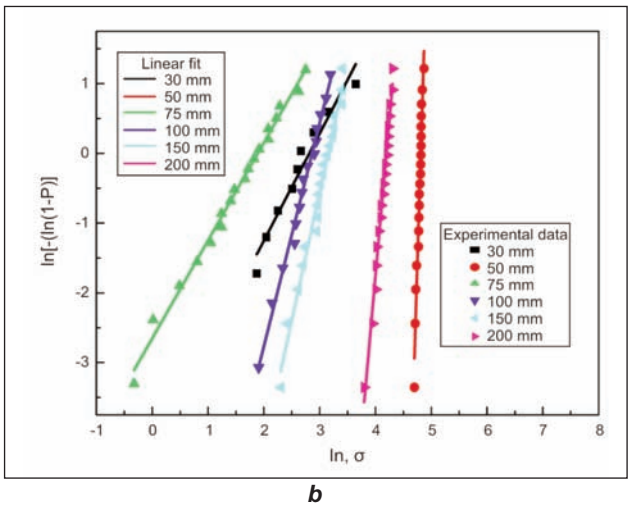


b

Fig. 3. *a* – Two parameter Weibull strength distribution of POFs ($l=l_0$); *b* – Three parameter Weibull strength



a



b

Fig. 4. *a* – Plots of ln (–ln (1–*P*)) versus ln (σ) for POFs with different lengths. The lines correspond to linear fitting equation when $A=0$, $l=l_0$; *b* – Plots of ln (–ln (1–*P*)) versus ln (σ) for POFs with different lengths. The lines correspond to linear fitting equation when $A=0$, $l=l_0$

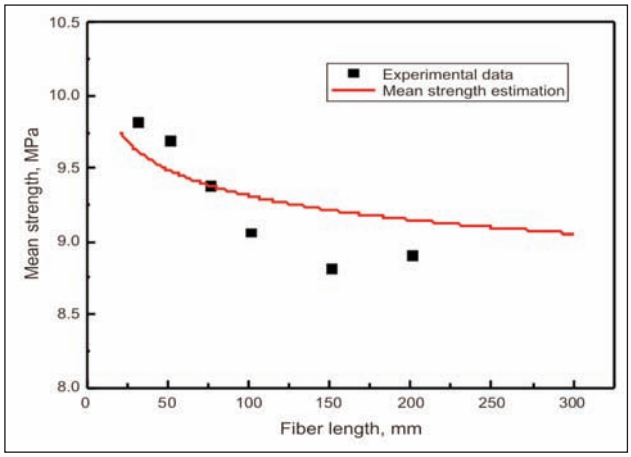


Fig. 5. Relation between mean fiber strength and fiber length distribution of POFs ($l = l_0$)

and equals to 70.77 MPa from figure 3, the scale and shape parameters can be obtained from the fitting equation of equation (1) based on 1 mm reference fiber length, as shown in equation (10).

$$y = -33.04 + 9x \quad (10)$$

Here, $C = 9$ and $B = 39.38$.

The variance of mean fiber strength can be predicted by equation (11), and the relationship between mean fiber strength and fiber length is shown in figure 5:

$$y = 70.77 + 37.29\,x^{-0.11} \quad (11)$$

CONCLUSIONS

It is significant to investigate the effects of fiber length on strength, strain and modulus. Strength and strain decline with the increase of fiber length. Modulus from tensile experiments may be dependent on the changes of strength and strain, rather than keep constant during fiber testing. The effects of fiber length on tensile characterizations indicate the probably visco-plastic properties and interface properties between core and cladding, which are needed to be investigated deeply and in detail in the future work. Both two parameter and three parameter Weibull distributions can be good models for investigation of statistical distribution of fiber strength. The relation between mean fiber strength and fiber length can be predicted by three parameter Weibull distribution.

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Determination of fabric drape using image analysis and fuzzy-logic methods

MUSA KILIC

REZUMAT – ABSTRACT

Determinarea drapajului țesăturii folosind analiza de imagine și metode fuzzy-logic

În cadrul acestui studiu a fost determinat atât prin analizele de imagine, cât și prin metode fuzzy-logic, coeficientul de drapaj, unul dintre cei mai importanți factori estetici ai unei țesături. În contextul acestui studiu, au fost realizate țesături pentru costume bărbătești cu fire 100 % lână pieptănată. În etapa experimentală au fost capturate, cu ajutorul unei camere digitale de înaltă rezoluție, imagini ale țesăturii, plasate pe o masă suport cu diametrul de 18 cm. Coeficientul de drapaj a fost calculat după ce imaginea captată a fost procesată, cu ajutorul tehnicilor de analiză a imaginii. În cea de-a doua etapă, a fost stabilit un model fuzzy-logic pentru a determina coeficientul de drapaj. În acest scop au fost măsurate prin metodele convenționale proprietățile fizice și mecanice ale țesăturii, cum ar fi: drapajul, masa, grosimea, rezistența, alungirea la rupere, rigiditatea la flexiune și forfecarea. De asemenea, proprietățile fizice și mecanice au fost utilizate ca variabile independente în modelele de regresie liniară multiplă, pentru a estima coeficientul de drapaj. În final, parametrii cu un nivel de seminificație ridicat au fost selectați ca variabile de intrare în modelul fuzzy-logic. Rezultatele au demonstrat că analizele de imagine și modelele fuzzy-logic pot determina coeficientul de drapaj, cu un grad ridicat de acuratețe. Trebuie menționat că rezultatele metodei fuzzy-logic s-au dovedit a fi mai apropiate de metoda Cusick convențională.

Cuvinte-cheie: drapaj, analiză de imagine, fuzzy-logic, regresie liniară multiplă

Determination of fabric drape using image analysis and fuzzy-logic methods

In this study, drape coefficient, one of the most important aesthetic properties of a fabric, was determined by both image analysis and fuzzy-logic methods. Within the context of the study, suiting fabrics produced from 100% wool worsted yarns were used as material. In the experimental part, image of the fabric placed on a circular support table with 18 cm diameter was captured by a high resolution digital SLR camera. Drape coefficient was calculated, after the captured image was processed by using image analysis techniques. At the second part, a fuzzy-logic model was established to predict fabric drape coefficient. For this purpose, physical and mechanical properties of the fabrics such as fabric drape, unit weight, thickness, tensile strength, tensile elongation, flexural rigidity and shear properties were measured by conventional methods. Furthermore, physical and mechanical properties were used as independent variables in the multiple-linear regression models to predict the drape coefficient. At last, parameters with high significance levels were selected as input variables in the fuzzy-logic model. Results showed that image analysis and fuzzy-logic models can determine drape coefficient with high accuracies. Nevertheless, results of fuzzy logic method were found closer to the conventional Cusick method.

Keywords: fabric drape, image analysis, fuzzy-logic, multiple linear regression

INTRODUCTION

Drape which is among the most important aesthetic properties of a fabric could be described as the degree of change in shape of a fabric with its own weight (ISO 9073-9). Drape properties of fabrics have become important especially in recent years with increasing comfort and aesthetic perceptions of the end users.

The first research on fabric drape was carried out by Pierce in 1930 [1]. Pierce measured fabric bending to determine the two dimensional drape value and developed the Cantilever device. Chu *et al.* [2, 3] who developed "Fabric Research Laboratories" to measure three-dimensional drape emphasized the effect of bending and shear properties on fabric drape. In 1960s, Cusick [4, 5] adopted a similar approach and developed "Cusick Drape Tester" to measure the drape of a fabric. In this method, fabric drape was determined by cut-and-weigh principle.

Researches on fabric drape have continued from 1960s to today. However, in parallel to the developments in science and technology, researches on fabric drape have moved to a different dimension. New techniques focused on technology have suggested new ways to save money, time and labour by eliminating the conventional cut-and-weigh process. Image analysis methods, particle-based models, lattice models, finite element techniques, co-rotational grid-based models, 3D simulations, artificial neural network (ANN) and fuzzy-logic methods may be some examples of these new techniques [6–13].

In recent years, image analysis methods have been widely used to find conjectural data about textile products. Many researchers have focused on determination of drape coefficient by using image analysis methods. New devices, which use the same principle with Cusick drape tester, have also been developed. Novel devices have generally been equipped with a CCD camera mounted on top of the fabric and image

analysis techniques have been used for the simulation of draped fabric behavior. Tsai et al. [14] designed a drape instrument to acquire the contours of draped images to calculate the drape coefficient. Stylios and Wan [15] developed a virtual 3D fabric drape measurement system by using image analysis method. In the system, a physical based model was used to predict the draping performance and static and dynamic drape of a given fabric sample. Mitzutani et al. [16] developed an image analysis based new apparatus called "Drape Elevator" to analyze the drape generation mechanism. In the study, a new parameter evaluating the shape of the drape was defined in terms of drape projection. Jeong [17] calculated the drape with using image analysis method and suggested a new parameter, called "Drape Distance Ratio (DDR)", alternative to the drape coefficient. Behera and Mishra [18] used image analysis method to simulate the aesthetic properties of fabrics and developed an index called "Fabric Appearance Index (FAI)" to express the most important aesthetic features of an apparel fabric such as pilling, drape, texture and wrinkle. Behera and Pattanayak [19] also investigated the measurement of drape of apparel fabric by using digital image processing. For this purpose "Drapemeter", based on image analysis technique, was developed. Platturk and Kilic [20] developed a new device based on image analysis technique to measure the fabric drape and flexural rigidity at the same time. Matsudaira et al. [21] investigated the changes in dynamic drapability of polyester fabrics in point of weave density, yarn twist and yarn count by the help of image analysis techniques.

Fuzzy logic, becoming important especially after the middle of 1980s has been applied extensively in many areas spreading rapidly. The concept of "Fuzzy Logic" was introduced by Lotfi Asker Zadeh in the 1960s [22]. Fuzzy logic converts measurements to sensory judgments and numerical calculations to evaluation of verbal statements. Fuzzy logic means to express the values which are in between wholly right or wholly wrong with 0 to 1 degree of membership. Fuzzy logic is to be able to see grey tones between black and white. It took a long time for fuzzy logic to be accepted and applied in the science and industrial areas. However, after the success of fuzzy logic based "Automatic Subway Control System" used in the construction of the Japanese subway in 1987, fuzzy logic concept spread worldwide. Universities and industry started researches to improve and apply fuzzy logic into their areas. One of these areas of application was textiles. Work done in the last years showed that fuzzy logic can be applied almost in all areas of textile processing instead of conventional ones. Huang & Yu [23] studied on controlling the dye concentration, pH, and temperature in dyeing processes with fuzzy logic. Kuo *et al.* [24] proposed a novel approach for color and pattern analysis of printed fabrics. An unsupervised analysis method was developed using a fuzzy C-means (FCM) clustering algorithm and a specific cluster-validity (SC) criterion. The experimental results showed that the approach was very suitable to analyze the colors and patterns of printed fabrics. Huang & Chen [25] worked on an image classification by a neural-fuzzy system for normal fabrics and fabrics with eight kinds of defects. The results demonstrated that the neural-fuzzy system is fairly successful for classifying the defects. Huang & Yu [26] also proposed image processing and fuzzy neural network approaches for classification of seven kinds of dyeing defects. Chen *et al.* [27] studied on objective evaluation of fabric softness. Fuzzy comprehensive evaluation method was used to solve the problem of fabric softness grading. Results were found promising for further investigation. Park *et al.* [28] studied on applying fuzzy logic and neural networks to total hand evaluation of knitted fabrics. Park and Kang [29] proposed a new quantitative method to evaluate seam pucker with five shape parameters by using three-dimensional image analysis and neuro-fuzzy systems. Lau *et al.* [30] suggested a fuzzy expert system with gradient descent optimization for selection of the fabric specimens in fashion product development. Compared with the traditional methods, this advisory system was more closely related to the sensory judgments made by individuals during fabric selection. Ertugrul & Ucar [31] predicted the bursting strength of cotton plain knitted fabrics before manufacturing using neural network and neuro-fuzzy approaches. Among many parameters that affect fabric bursting strength, fabric weight, yarn breaking strength, and yarn breaking elongation were taken as input elements for the predictions. Results were satisfying for an approximate knowledge of the bursting strength. Fan *et al.* [32] proposed to use a fuzzy-neural network system to predict and display the draped image of garments made of different fabrics and styles. The new approach was used to develop a prototype drape prediction system to predict the drape of a lady's dress style made from different fabrics.

Aim of this study was to analyse and compare the soft computing techniques which are alternative to the conventional Cusick method. In the study, fabric drape coefficient was determined by both image analysis method and newly established fuzzy-logic model and success of the methods were compared in consideration of the conventional Cusick method.

EXPERIMENTAL

In the study, 20 types of woven suiting fabrics produced from 100% wool worsted yarns were used as material.

The study mainly consisted of four main parts.

In the first part of the study, actual drape coefficients (%) of the fabrics were measured by conventional Cusick method based on cut-and-weigh principle.

In the second part, images of draped fabrics were captured by a high resolution digital SLR camera mounted on top of the Cusick drape tester with a distance of 80 cm between the camera lens and fabric support disc. Real colour images were transformed into gray level and binary images respectively by

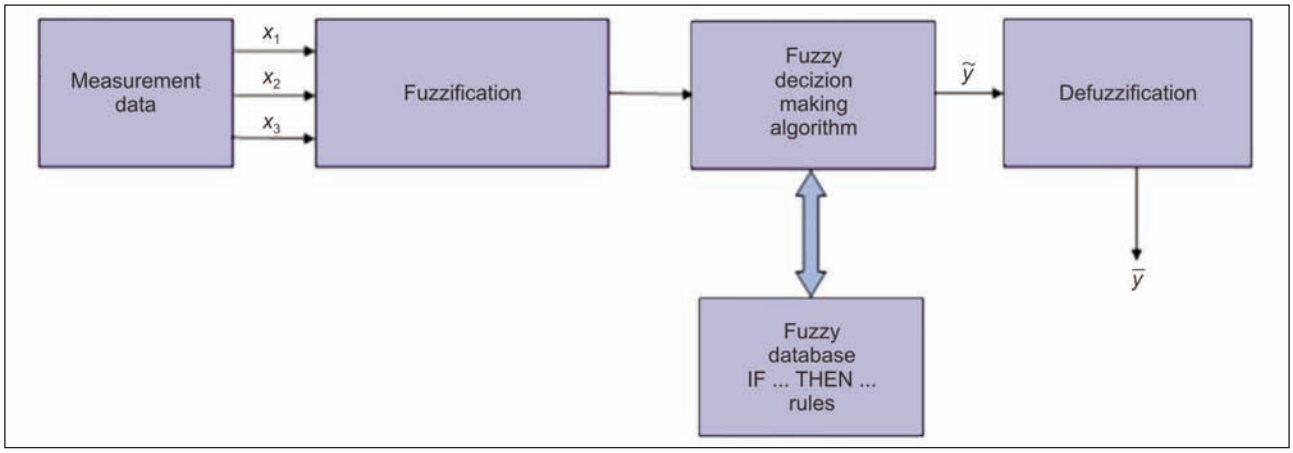

Fig. 1. The structure of fuzzy expert system

Matlab package programme. Drape coefficient (%) was calculated according to the eq. 1 by the help of codes written in Matlab.

$$DC_{IA}\,(\%) = \frac{A_{DF} - A_{SD}}{A_{FS} - A_{SD}} \cdot 100 \qquad (1)$$

where: DC_{IA} represents the drape coefficient (%) calculated by image analysis method, A_{DF} represents the area of draped fabric image (cm^2), A_{SD} represents the area of support disc (cm^2) and A_{FS} represents the total area of fabric sample (cm^2).

In the third part, drape coefficient (%) was estimated by the help of multiple linear regression analysis where unit weight (g/m^2), thickness (mm), flexural rigidity (mg.cm), tensile strength (kgf) and elongation (%) in both warp and weft directions, shear strength (kgf), shear elongation (%), shear modulus in initial state (kgf/mm) and shear modulus (kgf/mm) were taken as independent variables.

After this stage, a fuzzy logic model was developed using Mamdami fuzzy inference method. In the fuzzy logic model (figure 1), three of the fabric parameters that have the highest contribution to regression models were taken as input variables.

At last, drape coefficients (%) obtained by image analysis, fuzzy logic, regression analysis and conventional Cusick methods were compared.

RESULTS AND DISCUSSION

In the study, drape coefficients (%) of the fabrics were measured by conventional Cusick method. A statistical summary of drape coefficients (%) are given in table 1.

Drape coefficients (%) of 20 types of sample fabrics are also given in figure 2 as boxplot diagrams. As it is seen from table 1 and figure 2, drape coefficients of sample fabrics varied from 33% to 53%.

At the second step, drape coefficients (%) of the fabrics were measured by image analysis method. Drape coefficients (%) calculated by eq.1 are given in table 2.

At the third step of the experimental part, drape coefficient (%) was estimated from various fabric quality parameters which are thought to be in relation with drape. These parameters were fabric unit weight (g/m^2), fabric thickness (mm), flexural rigidity (mg.cm), tensile strength (kgf) and elongation (%) in both warp and weft directions, shear strength (kgf), shear elongation (%), shear modulus in initial state (kgf/mm) and shear modulus (kgf/mm). Mean values of these parameters are given in table 3.

Table 1

STATISTICAL SUMMARY OF DRAPE COEFFICIENTS (%)

Fabric	Mean (%)	Std. Dev. (%)	%95 Confidence Intervals (%)	
			Lower Bound	Upper Bound
1	39.69	0.516	38.8697	40.5172
2	36.14	0.448	35.4217	36.8500
3	52.23	0.625	51.2370	53.2196
4	45.08	0.832	43.7636	46.4009
5	40.02	0.449	39.3005	40.7258
6	49.68	1.388	47.4756	51.8935
7	34.66	1.084	32.9388	36.3791
8	41.18	0.806	39.8949	42.4684
9	38.80	1.138	36.9923	40.6122
10	38.21	0.686	37.1207	39.3032
11	41.03	0.968	39.4883	42.5643
12	43.94	0.295	42.5643	44.4063
13	39.90	0.754	38.7051	41.0997
14	44.80	1.653	42.1699	47.4274
15	39.24	0.914	37.7842	40.6955
16	44.62	1.112	42.8584	46.3847
17	39.67	1.276	37.6337	41.6986
18	39.56	1.385	37.3570	41.7633
19	41.91	0.696	40.7991	43.0102
20	39.49	0.567	38.5792	40.3951

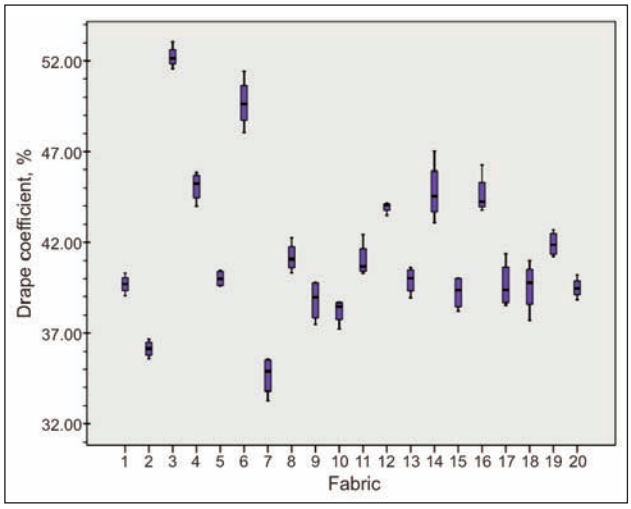

Fig. 2. Boxplots of drape coefficients (%) of sample fabrics

Multiple linear regression analysis was carried out according to stepwise method which is an automated process of building a model by adding or removing variables based on the t-statistics of their estimated coefficients. After the regression analysis was held, five regression models were obtained (eqs. 2 to 6).

$$DC_{RA}\ (\%) = 24.9 + 0.047 \times FR \quad (2)$$

$$DC_{RA}\ (\%) = 43.0 + 0.049 \times FR - 0.120 \times W \quad (3)$$

Table 2

DRAPE COEFFICIENTS (%) CALCULATED BY IMAGE ANALYSIS METHOD	
Fabric	**Drape Coefficient (%) by Image Analysis (DC_{IA})**
1	42.36
2	37.78
3	53.78
4	47.95
5	40.91
6	50.90
7	37.04
8	42.27
9	40.40
10	39.43
11	42.84
12	44.87
13	40.73
14	46.20
15	38.34
16	44.81
17	40.61
18	39.86
19	40.91
20	40.37

Table 3

VALUES OF FABRIC PARAMETERS USED FOR ESTIMATION OF DRAPE COEFFICIENT (%)

Fabric	Unit Weight (g/m^2)	Thickness (mm)	Flexural Rigidity (mg.cm)	Tensile Strength (kgf)		Tensile Elongation		Shear Strength (kgf)	Shear Elong. (%)	Initial Shear Modulus (kgf/mm)	Shear Modulus (kgf/mm)
				Warp	*Weft*	*Warp*	*Weft*				
1	164.2	0.318	330.1	33.9	28.1	33.6	39.0	27.8	47.3	0.239	0.190
2	150.7	0.279	332.5	30.2	20.0	17.8	32.5	22.9	50.1	0.177	0.141
3	163.8	0.308	437.4	38.3	22.0	56.4	32.2	25.1	57.8	0.163	0.130
4	139.5	0.266	430.6	29.8	18.8	23.5	18.4	21.2	45.2	0.181	0.143
5	181.5	0.369	328.4	32.3	24.4	26.8	30.5	26.4	43.5	0.218	0.173
6	134.2	0.278	383.8	25.9	19.8	29.9	28.4	20.7	45.2	0.160	0.127
7	174.6	0.306	348.1	30.7	24.8	32.8	30.2	26.4	51.8	0.174	0.138
8	148.2	0.320	372.1	27.1	20.4	23.4	26.5	21.5	45.3	0.184	0.146
9	184.5	0.376	400.2	33.1	24.4	26.3	35.5	26.4	45.6	0.210	0.166
10	176.1	0.357	354.1	34.2	24.1	22.3	26.7	27.2	41.0	0.215	0.173
11	151.3	0.291	344.4	31.4	19.7	38.7	29.7	21.6	44.9	0.168	0.133
12	140.1	0.283	410.8	30.9	18.6	22.9	24.2	21.1	40.2	0.184	0.146
13	148.2	0.276	299.7	32.1	22.5	35.4	28.6	27.3	43.6	0.192	0.152
14	165.2	0.325	410.1	35.5	25.7	21.7	21.7	22.7	30.8	0.256	0.203
15	151.2	0.327	259.4	27.7	21.0	24.8	27.4	22.4	39.8	0.178	0.141
16	133.9	0.284	335.6	28.4	20.3	16.1	14.3	23.0	33.0	0.233	0.185
17	185.4	0.395	396.4	42.3	24.8	21.0	27.9	28.3	32.4	0.255	0.203
18	152.6	0.298	293.2	29.3	19.6	38.4	26.2	26.6	51.9	0.163	0.129
19	149.7	0.310	312.5	36.7	20.2	19.4	27.7	22.1	31.9	0.206	0.164
20	176.4	0.357	322.1	33.0	25.8	31.1	28.9	29.7	49.6	0.196	0.155

Table 4

COEFFICIENTS OF MULTIPLE LINEAR REGRESSION MODELS BY STEPWISE METHOD						
Model	**Fabric Parameter as Independent Variable**	**Unstandardized Coefficients**		**Standardized Coefficients**	**t**	**Sig.**
		B	**Std. Error**	**Beta**		
1	(Constant)	24.924	2.980		8.365	0.000
	Flexural Rigidity	0.047	0.008	0.536	5.611	0.000
2	(Constant)	43.015	3.997		10.761	0.000
	Flexural Rigidity	0.049	0.007	0.568	7.059	0.000
	Unit Weight	–0.120	0.021	–0.467	–5.802	0.000
3	(Constant)	41.036	3.599		11.401	0.000
	Flexural Rigidity	0.047	0.006	0.544	7.543	0.000
	Unit Weight	–0.129	0.019	–0.502	–6.946	0.000
	Tensile Elongation (warp)	0.149	0.033	0.327	4.516	0.000
4	(Constant)	38.422	3.050		12.599	0.000
	Flexural Rigidity	0.049	0.005	0.565	9.335	0.000
	Unit Weight	–0.319	0.036	–1.237	–8.770	0.000
	Tensile Elongation (warp)	0.230	0.031	0.506	7.429	0.000
	Thickness	93.976	16.294	0.813	5.768	0.000
5	(Constant)	35.613	2.980		11.951	0.000
	Flexural Rigidity	0.047	0.005	0.538	9.383	0.000
	Unit Weight	–0.332	0.034	–1.290	–9.683	0.000
	Tensile Elongation (warp)	0.285	0.033	0.626	8.554	0.000
	Thickness	84.775	15.524	0.733	5.461	0.000
	Shear Modulus	45.811	13.640	0.261	3.359	0.001

Dependent Variable: Drape Coefficient (%)

$$DC_{RA}\ (\%) = 41.0 + 0.047 \times FR - 0.129 \times W + 0.149 \times TE_{warp} \quad (4)$$

$$DC_{RA}\ (\%) = 38.4 + 0.049 \times FR - 0.319 \times W + 0.230 \times TE_{warp} + 94.0 \times T \quad (5)$$

$$DC_{RA}\ (\%) = 35.6 + 0.047 \times FR - 0.332 \times W + 0.285 \times TE_{warp} + 84.8 \times T + 45.8 \times SM \quad (6)$$

where DC_{RA} (%) represents drape coefficient (%) obtained by regression analysis, FR represents flexural rigidity (mg.cm), W represents fabric unit weight (g/m^2), TE_{warp} represents tensile elongation in warp direction (%), T represents fabric thickness (mm), and SM represents shear modulus (kgf/mm). Coefficients of regression equations are listed in table 4.

Regression determination coefficients of the models are given in table 5. As it is seen from the table, models 4 (eq. 5) and 5 (eq. 6) explained the dependent variable around 75%. This value can be admitted satisfactory with a statistical perspective. On the other hand, researches on estimating drape coefficient generally obtained higher adjusted R square values such as 80% to 99%. In regression analysis, large experimental designs are essential to obtain higher success in the models. Moreover, independent variables have to be measured separately for the estimation. Because of these reasons, use of regression analysis technique is not a practical way to estimate drape coefficient.

Table 5

COEFFICIENTS OF MULTIPLE LINEAR REGRESSION MODELS BY STEPWISE METHOD				
Model	**R**	**R Square**	**Adjusted R Square**	**Std. Error of the Estimate**
1	0.536	0.288	0.278	3.59343
2	0.710	0.504	0.491	3.01682
3	0.780	0.609	0.594	2.69634
4	0.854	0.729	0.715	2.25912
5	0.875	0.765	0.749	2.11858

At the fourth step, a fuzzy-logic model was constituted to estimate the drape coefficient (%) by the help of "Fuzzy-Logic" toolbox in Matlab package programme. Input variables of the fuzzy-logic model were selected by the light of the literature and multiple linear regression models. From this point of view, flexural rigidity (mg.cm), fabric unit weight (g/m^2) and tensile elongation in warp direction (%) that have relatively high contributions to the regression models were taken as input variables. In the fuzzification step, these parameters were expressed in verbal way by the help of the literature and experts. Figures 3–5

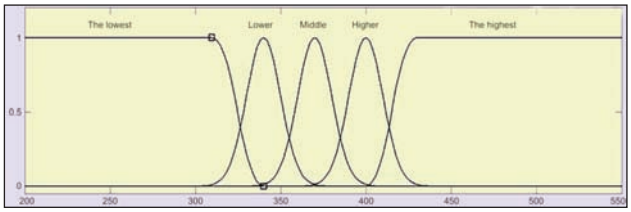

Fig. 3. Fuzzy set of flexural rigidity (mg.cm) as input variable

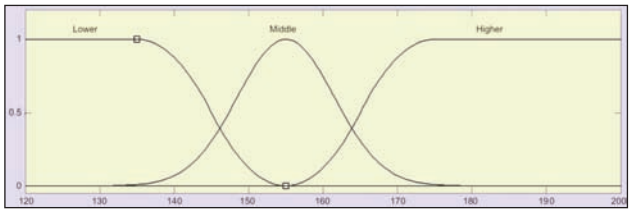

Fig. 4. Fuzzy set of unit weight (g/m^2) as input variable

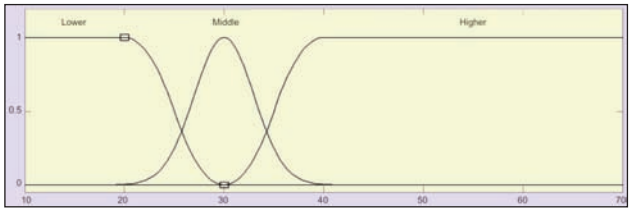

Fig. 5. Fuzzy set of tensile elongation in warp direction (%) as input variable

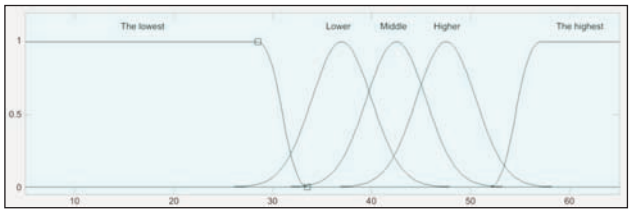

Fig. 6. Fuzzy set of drape coefficient (%) as output variable

show the fuzzy sets of flexural rigidity (mg.cm), unit weight (g/m^2) and tensile elongation in warp direction (%).

Moreover, figure 6 shows the fuzzificated output variable. Fuzzy set of drape coefficient (%) was expressed with five membership functions that were thought to contain every possible drape coefficient value for worsted suiting.

The next step of the fuzzy logic method was the determination of "If … Then … Else …" rules depend on the experts' opinions. In the study, 45 rules were determined to estimate the fabric drape (figure 7).

At the last step, defuzzification of the fuzzy drape coefficient result was carried out by the most common method: "Center of Area – *CoA*". Defuzzification is the process of converting the degrees of membership of output linguistic variables into numerical values. In other words, defuzzification is the process of converting fuzzy grade to crisp output. Center of Area (Center of Gravity or Centroid) method is one of the most prevalent and physically appealing of all the

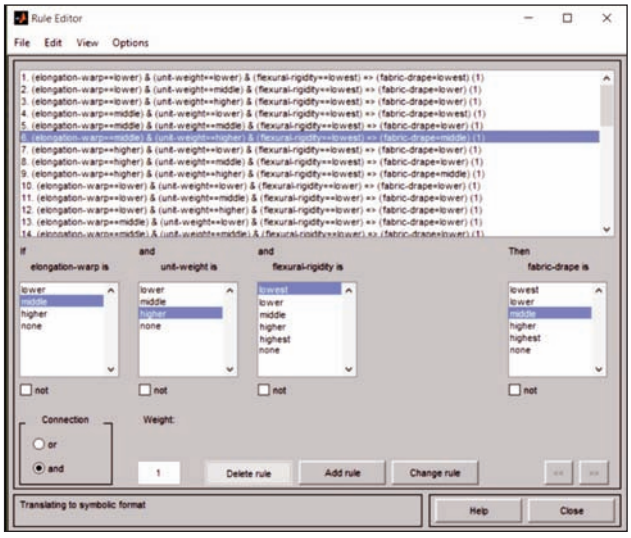

Fig. 7. Some rules at Matlab Rule Editor

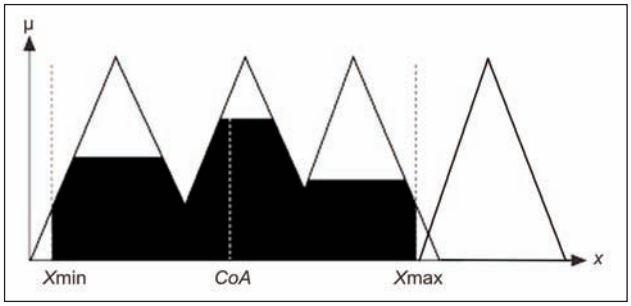

Fig. 8. Center of Area (*CoA*)

defuzzification methods. In the Center of Area (*CoA*) defuzzification method, the fuzzy logic controller calculates the area under the scaled membership functions and within the range of the output variable using the following formula [33]:

$$CoA = \frac{\int_{x_{min}}^{x_{max}} f(x)\, x\, dx}{\int_{x_{min}}^{x_{max}} f(x)\, dx} \quad (7)$$

where *CoA* is the Center of Area, x – the value of the linguistic variable, and x_{min} and x_{max} – the range of the linguistic variable.

Figure 8 shows the *CoA* defuzzification method.

In table 6, a comparison between the drape coefficients obtained from the Cusick method, regression model 4, regression model 5, image analysis method and fuzzy-logic model are given. For this evaluation, a new set of randomly selected worsted suiting fabrics were used and values of flexural rigidity (mg.cm), unit weight (g/m^2), tensile elongation in warp direction (%), thickness (mm) and shear modulus (kgf/mm) were measured to be used in regression and fuzzy-logic models.

The effect of test method on drape coefficient (%) was analyzed by the help of ANOVA. Results showed

Table 6

A COMPARISON BETWEEN THE DRAPE COEFFICIENTS BY DIFFERENT METHODS										
Test No	DC (%) by Cusick Method	Inputs for Regression and Fuzzy Models					DC (%) by Model 4	DC (%) by Model 5	DC (%) by Image Analysis	DC (%) by Fuzzy Logic
		Flexural Rigidity (mg.cm)	*Unit Weight (g/m^2)*	*Tensile Elong. Warp*	*Thickness (mm)*	*Shear Modulus (kgf/mm)*				
1	53.0	*446.0*	*164.1*	*58.0*	*0.306*	*0.129*	50.0	50.5	54.9	53.2
2	40.4	*337.9*	*181.5*	*29.2*	*0.363*	*0.174*	37.9	38.3	41.3	42.2
3	40.3	*320.0*	*182.1*	*26.5*	*0.370*	*0.168*	36.9	36.8	40.6	41.0
4	39.6	*340.3*	*180.3*	*26.1*	*0.370*	*0.171*	38.3	38.4	40.3	40.3
5	39.6	*315.3*	*182.0*	*25.1*	*0.373*	*0.180*	36.6	37.0	41.5	39.0
6	40.3	*357.7*	*148.3*	*24.2*	*0.320*	*0.154*	44.3	44.3	41.4	40.4
7	40.9	*386.7*	*150.1*	*26.0*	*0.320*	*0.150*	45.5	45.4	42.9	42.3
8	37.2	*365.1*	*175.9*	*21.9*	*0.359*	*0.177*	39.0	39.2	38.9	37.2
9	40.8	*326.9*	*151.6*	*40.2*	*0.293*	*0.130*	42.8	42.9	42.9	39.7
10	44.0	*403.4*	*139.2*	*24.1*	*0.283*	*0.143*	45.9	45.8	43.8	43.4

Table 7

DIFFERENCES BETWEEN THE CUSICK METHOD AND THE OTHERS				
Test No	**Difference between Cusick and Reg. 4**	**Difference between Cusick and Reg. 5**	**Difference between Cusick And Image**	**Difference between Cusick and Fuzzy**
1	2.99	2.53	1.9	0.20
2	2.50	2.10	0.9	1.80
3	3.43	3.49	0.3	0.70
4	1.26	1.22	0.7	0.70
5	2.97	2.58	1.9	0.60
6	3.97	3.96	1.1	0.10
7	4.63	4.46	2.0	1.40
8	1.76	1.95	1.7	0.00
9	2.05	2.09	2.1	1.10
10	1.91	1.76	0.2	0.60
Mean Difference	**2.70**	**2.51**	**1.28**	**0.81**

that there were no statistically significant difference between any methods (p=0.973). Figure 9 shows the pairwise comparisons of the methods by the help of interval plots.

Table 7, derived from table 6 shows the absolute values of differences between the Cusick method and the other methods.

As it is seen from the table 7, fuzzy logic method calculated the closest values to the real values measured by Cusick method. Regression model 4 needed four and regression model 5 needed five variables while fuzzy-logic method needed only 3 variables. Moreover, deviation from the real value is less than 1% in fuzzy-logic method.

Deviations from the conventional Cusick method were also analyzed by ANOVA. Results showed that differences between the deviations are statistically significant (p=0.000) (figure 10).

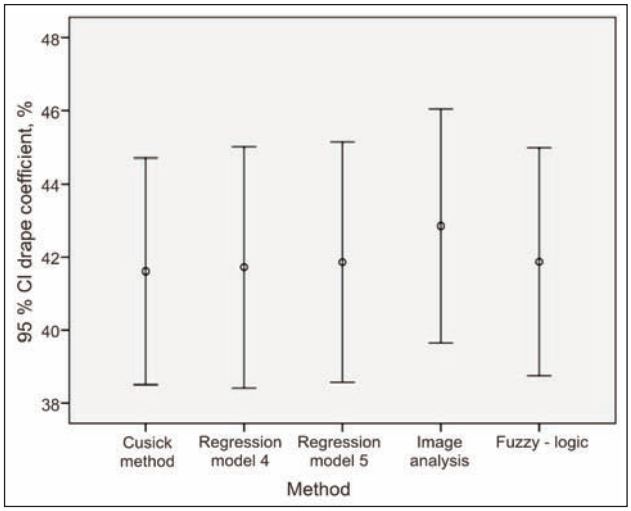

Fig. 9. 95% confidence intervals of drape coefficients (%) for each method

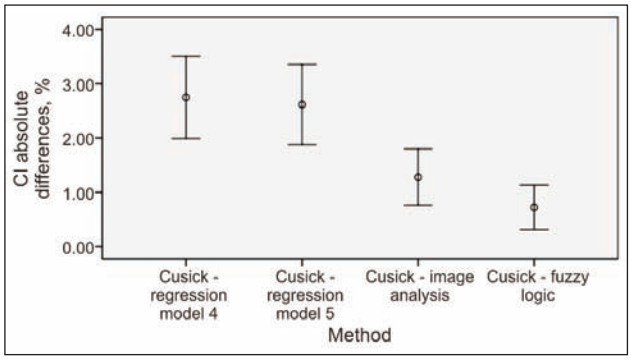

Fig. 10. 95% confidence intervals of deviations from the Cusick method

CONCLUSIONS

In this study, drape coefficient was determined by both image analysis and fuzzy-logic methods. The successes of the methods were evaluated by comparing the results both with each other and with the conventional Cusick method and multiple linear regression models.

In the study, drape coefficients were calculated by Cusick method first. Multiple linear regression analysis was also carried out to estimate the drape coefficient alternatively to the image analysis and fuzzy-logic methods. Stepwise technique suggested five regression equations to estimate the fabric drape. In image analysis method, fabrics were located on the support disc of Cusick drape tester and draped images were captured by a digital camera. In the fuzzy-logic method, flexural rigidity (mg.cm), fabric weight (g/m^2) and tensile elongation in warp direction (%) parameters were taken as input variables. All the fuzzification processes for input and output variables were carried out by the light of the literature and the opinions of the experts.

When the drape coefficients obtained by image analysis and fuzzy-logic methods were compared with the real results, it was seen that fuzzy logic method gives more accurate results.

This result probably occurred because of the problems caused by area density and area depth while using a camera. On the other hand, fuzzy logic method which is mostly depends on the experts' experiences and human perception determined the nearest drape coefficient values to the Cusick results.

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A study on the application of recycled fabrics as automotive seat upholstery

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REZUMAT – ABSTRACT

Studiu referitor la folosirea țesăturilor reciclate pentru tapițeriile scaunelor de automobile

Folosirea produselor reciclate prezintă un interes din ce în ce mai ridicat în cadrul industriei auto. În aplicațiile de înaltă tehnicitate este mai dificil de folosit fibre reciclate, în locul celor originale. Materialele textile destinate scaunelor automobilelor trebuie să posede performanțe sporite, cum ar fi rezistența la abraziune, rezistența la lumină, comportamentul la îmbătrânire etc. Din acest motiv, înlocuirea fibrelor de poliester originale cu fibre reciclate trebuie investigată și efectuate toate testările tehnice și de conformitate. Posibilitatea folosirii fibrelor reciclate în cadrul țesăturilor pentru scaune de automobile reprezintă obiectul acestui studiu. Au fost comparate diferite tipuri de fire și țesături, realizate din fire filamentare din poliester reciclate și originale. Ca rezultat al studiului s-a concluzionat că, deși există diferențe între rezultatele testărilor firelor și ale țesăturilor, valorile acestora se încadrează într-un interval acceptabil pentru specificațiile clienților. În concluzie, putem afirma că, folosind procese de prelucrare adaptate, este posibilă producerea unui produs finit din materiale reciclate, care să dețină aceleași proprietăți cu unul obținut din fibre originale.

Cuvinte-cheie: textile pentru automobile, tapițerie scaune, fibre reciclate, proprietăți fizice ale firelor, proprietățile țesăturii

A study on the application of recycled fabrics as automotive seat upholstery

Application of recycled products is high of interest in the automotive industry. In the high tech applications it is difficult to use recycled fibers instead of virgin fibers. Automotive seat fabrics must have high performance such as high abrasion resistance, light fastness, aging behavior etc. Therefore replacement of virgin polyester fibers by recycled polyester fibers in seat fabrics must be investigated and all technical tests and conformations must be done. Therefore application possibility of recycled yarns on automotive seat fabrics was investigated in this study. Different types of yarns and woven fabrics were produced with recycled and virgin polyester filament fibers and their properties were compared. As a result of the study, it was concluded that although there are some differences between the yarn and fabric test results, the values were in the acceptable range for customer specification. As conclusion, it can be said that by using proper process settings, it is possible to produce final recycled product which has same property that of virgin one.

Keywords: automotive textiles, seat upholstery, recycled fibers, physical yarn properties, fabric properties

INTRODUCTION

In recent years, the automotive consumption has been significantly increasing and this causes a high increase in the number of cars which complete their lifetimes. Therefore the management of end-of-life vehicle (ELVs) is recently in the subject of worldwide concern. Another important concern is evaluation of the recycling residues in automotive materials [1–3]. Global automotive companies put different regulations to their suppliers regarding recycling and recyclability of the materials in different regions of world [4–6, 8].

Automotive and their supplier companies follow different Research & Development (R&D) and Product & Development (P&D) projects in the sense of recycling. Some of the projects have been already complemented and their products are in mass production. One of the products which are already in the market belongs to Ford Motor Company, the „Interface Fabric“. This fabric is the first fabric which is developed and produced in USA for automotive seat fabrics. The “Interface fabric” is produced from 100% recycled polyester fibers. It has been announced that the “Interface fabric” has the same touch, view and technical performance that of virgin polyester fabrics [7].

The motivation behind the usage of recycled materials in automotive fabrics lies on environmental concerns rather than financial issues. Mostly the price of the recycled materials higher than that of virgin materials.

The aim of this study is to investigate the behavior of recycled fibers and virgin polyester fibers on different yarn counts and different woven constructions and compare their performances. Air-jet texturized yarns having different linear densities were produced and tensile strength, tenacity and elongation of yarns were tested and compared. In the second step woven fabrics with different patterns and weft/warp densities were produced with recycled and virgin polyester yarns. Abrasion resistance, tensile strength, tearing strength, mace snagging resistance, seam strength and pilling tendency of fabrics were tested and compared with that of virgin polyester fabrics.

EXPERIMENTAL STUDY

Materials

In the study virgin and recycled partially oriented yarns (POY's) have been used. Properties of POY's were tested and results of the materials are shown in table 1.
The recycled POYs used in the study were post-consumer materials which were produced from used PET bottles.

Methods

In the study different types of yarns and woven fabrics were produced with recycled (post-consumer) and virgin polyester filament fibers and their properties were compared. Yarns and fabrics produced in the study are listed in table 2.
Recycled and virgin air-jet texturized yarns were produced with the same parameters on Aiki airjet texturizing machine. Properties of yarns such as linear density, breaking strength, elongation and tenacity were tested and compared. For the weaving side, in order to see the effect of the pattern, two different patterns were produced. Processes and the materials used in the fabrics are shown in table 3. Both constructions were produced with 100% virgin and 100% recycled yarns.
Woven fabrics have been fixed on Stenter machines. After stentering process fabrics have been laminated with foam and scrim on the flame lamination process. The same parameters and same foam and scrim were used for recycled and virgin versions.

Table 1

TEST RESULTS OF VIRGIN AND RECYCLED POLYESTER POYs

	Titer (dtex)	B-Force (cN)	Elongation (%)	Tenacity (cN/tex)
%100 Virgin polyester POY	285	549	152.91	19.26
%100 Recycled polyester POY	277	524.8	160.7	18.94

Fabric Tests

After production of the fabrics, tests were realized. Physical tests realized in the study are; thickness, weight, breaking strength, tear strength, seam strength, resistance to abrasion taber, resistance to abrasion Stoll, resistance to mace snagging, resistance to pilling, Velcro, resistance to heat aging, soiling and clean ability.
Thickness measurement was realized according to ASTM 1777. Unit weights of the fabrics were also tested.
Breaking strength of the fabrics was tested according to the ASTM D 5034. Tear strength of the fabrics were measured according to the ASTM D 1117 Integration method.
Seam strength is the resistance required to break sewn fabrics. Three different test specimens are prepared for this test. The first, longitudinal specimen is

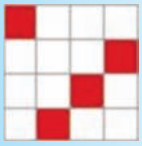
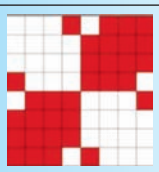
Table 2

YARNS PRODUCED IN THE STUDY

Yarn Type	Construction used	Purpose	Linear Density	Material
Yarn A-Virgin	Construction LAT	Warp-Weft	1330 dtex	Virgin POY
Yarn A-Recycled	Construction LAT	Warp-Weft	1330 dtex	Recycled POY
Yarn B-Virgin	Construction SEM	Warp	660 dtex	Virgin POY
Yarn B-Recycled	Construction SEM	Warp	660 dtex	Recycled POY
Yarn C-Virgin	Construction SEM	Weft	590 dtex	Virgin POY
Yarn C-Recycled	Construction SEM	Weft	590 dtex	Recycled POY

Table 3

PROCESSES AND MATERIALS USED IN THE FABRICS

Fabric	Pattern	Materials	Densities	Process
Construction LAT		Warp - Weft 1330 dtex virgin and recycled polyester	Warp: 28 end/cm Weft: 30 pick/cm	Weaving, Washing, Fixation, Lamination
Construction SEM		Warp - 660 dtex virgin and recycled polyester Weft - 590 dtex virgin and recycled polyester	Warp: 14 end/cm Weft: 13,5 pick/cm	Weaving, Washing, Fixation, Calendering, Lamination

a

b

Fig. 1. (a) Mace snagging test (b) One reference picture to evaluate mace snagging

sewn with the other longitudinal specimen. The second, transverse specimen is sewn with the other transverse specimen. The third, transverse specimen is sewn with longitudinal specimen. Zwick Test Equipment is used for measurements.

Resistance to heat aging was tested according to AATCC Procedure-2 while soiling and clean ability tests were performed according to ISO 105-A02/ AATCC.

Abrasion resistance tests are the most important tests for automotive upholstery fabric. There are many abrasive materials such as cloth / sand paper / Velcro / Taber wheels which are in use for different abrasion test methods. After these tests, yarn breaks / color change / surface deformation are analyzed for the evaluation [9]. In this study, Taber test was performed with Taber wheels and 1000 cycles was tested. Procedure of SAE J 948 was followed.

Abrasion tests were also realized on Stoll Abrasion Testing Device. Abrasion test was realized with sand paper. 1600 cycles has been applied and tests were evaluated according to the wearing performance visually.

Mace snagging is simulating yarn pull out from the fabric. Tests were realized with rotating Mallets on the machine which is shown in figure 1, *a*. After 600 cycles, the deformation on the fabric surface was evaluated and compared with the reference pictures which one sample is shown in figure 1, *b*.

Velcro resistance was also tested. Martindale abrasion test device was used and Velcro band is used as abrasion fabric. After 50 cycles, fabric test results were evaluated by comparing reference pictures.

Flammability tests were realized according to ISO 3795/SAE J 369. After the tests, results of the fabrics produced with recycled polyester and virgin polyester were compared and discussed.

RESULTS AND DISCUSSION

Yarn Test Results

In table 4, results of recycled and virgin polyester yarns were compared. As it can be seen in the table 4, although there are some differences in the yarn linear densities, these differences are not that high. Comparison of breaking forces showed that in each type of yarns, recycled versions have lower breaking strength than that of the virgin polyester yarns. Tenacity results also confirm the breaking force values. Furthermore as the yarn linear density decreases, difference between the tenacity results of recycled and virgin versions increases. In the case of yarn C which has approximately the linear density of 590 dtex, difference between tenacity of the recycled and virgin is high (30.23 versus 19.01). In the case of yarn A, which has approximately linear density of 1330 dtex, difference between tenacity of recycled and virgin is lower (23.11 versus 17.94). These results showed that recycled yarns have lower strength properties than the virgin versions. The elongation results are opposite of tenacity results as expected. Elongation of recycled yarns is higher than that of virgin versions.

Table 4

AIR TEXTURIZED YARN RESULTS

	Titer (dtex)	B-Force (cN)	*Elongation (%)*	*Tenacity (cN/tex)*
Yarn A-Virgin	1204	2782	28.38	23.11
Yarn A-Recycled	1270	2279	32.65	17.94
Yarn B-Virgin	593	1257	23.22	21.21
Yarn B-Recycled	623	1083	27.95	17.38
Yarn C-Virgin	551	1665	30.50	30.23
Yarn C-Recycled	585	1112	31.08	19.01

The comparison of tenacity values of raw materials with tenacity values of yarns shows that there is a big difference in yarn results around (20%) than the raw materials around (2%). This result may due to the irregularity of recycled materials which become more obvious after exposing to the mechanical forces during air texturizing process.

Fabric Test Results

Fabrics were produced from recycled and virgin polyester versions of Yarn A, Yarn B and Yarn C.

Table 5

TEST RESULTS OF CONSTRUCTION LAT AND CONSTRUCTION SEM PRODUCED WITH RECYCLED AND VIRGIN FIBRES

Property	Unit	Acceptance Criteria	*CONSTRUCTION LAT-Recycled*		*CONSTRUCTION LAT-Virgin*		*CONSTRUCTION SEM-Recycled*		*CONSTRUCTION SEM-Virgin*	
			Length	Width	Length	Width	Length	Width	Length	Width
Laminated Weight	g	-	640		654		526		528	
Laminated Thickness	mm	-	6.6		6.3		4.10		4.50	
Face Fabric Weight	g	-	640		654		371		360	
Flammability	mm/min	max. 100	SE*	SE*	SE*	SE*	94	97	80	95
Breaking Strength	N	min. 400	1520	1434	4128	2966	1457	1220	1646	1328
Tear Strength	N	min. 180	327	319	1177	979	248	277	448	556
Seam Strength	N	min. 400	422	**373**	1065	779	552	597	927	955
Resistance to Heat Ageing	Minute	min. 4	5		5		5		5	
Soiling and Cleanability**	Scale 1-5	min. 3	¾		4		4		4	
Resistance to Abrasion Taber	OK/NOK	Visual Evaluation	**Not OK**		OK		OK		OK	
Resistance to Abrasion Stoll	OK/NOK		OK		OK		OK		OK	
Resistance to Mace Snagging	OK/NOK	Visual Evaluation	OK		OK		OK		OK	
Velcro	OK/NOK		OK	OK	OK	OK	OK	OK	OK	OK

* SE: Self Extinguish
** Coffee/Soil/Grease/Ketchup

Those fabrics were tested and results are shown in table 5.
In table 5, properties and the minimum requirements for specifications are listed. Test results of recycled and virgin fiber fabrics (each fabric, construction LAT and construction SEM) are compared. As it can be seen in the table 5, weight, thickness and the face fabric results of recycled and virgin versions are close to each other. Flammability results are similar to each other in both fabric constructions.
The breaking strength results of recycled and virgin fabrics in construction LAT is very high although they meet the specifications. There is also a little difference between recycled and virgin versions of construction SEM. But it is not as obvious as that of construction LAT. This result means that the pattern of the fabric affect the performance of recycled fibers.
Tear strength and seam strength of the recycled and virgin fabrics are high. In construction LAT, tear strength and seam strength of recycled versions are lower than that of virgin version. Furthermore seam strength of recycled version in the length does not meet the required value. In construction SEM, both tear and seam strength of recycled version is lower than that of virgin version. But all the values are higher than the minimum required values. These results again confirm the effect of pattern.
Resistance to heat aging and soiling and clean ability results of recycled and virgin versions in both constructions are close to each other. These results meet the requirements.
The resistance to stoll abrasion, mace snagging and Velcro results of both versions in each construction are OK when compared with the visual requirements. There is not any difference between the test results. The critical difference was observed in Taber abrasion test. Taber test result of recycled version in construction LAT does not meet the requirements where virgin version meets. However in construction SEM both recycled and virgin versions have OK results.
As the Taber test is the most severe test in abrasion test, this test showed the difference between the materials and effect of the pattern on the abrasion resistance performance.

CONCLUSION

In this study performance of recycled and virgin fabrics in automotive seat fabric application was investigated. The effect of yarn linear densities and the effect of pattern were examined. Two different fabrics with different yarn linear densities and constructions were produced with recycled and virgin fibers and their properties were tested according to the automotive requirements.
As a result of the study, some differences were observed in the yarn form. Breaking strength and tenacity of recycled yarns are lower than that of virgin yarns. As the yarn linear density decreases, difference between recycled and virgin yarns' values increase. However, to compare whether recycled yarns to be used instead of virgin fabrics, fabric tests were realized. Breaking strength, tear strength and seam strength of recycled fabrics are lower than that of virgin fabrics. However values are in the limit. The other test results are in the limits according to the

specifications except the Taber and seam resistance test results.

As conclusion, it can be said that by using the proper settings to produce yarns it is possible to produce recycled yarns having same physical properties with that of virgin fibers. In the fabric research it was obtained that the yarn linear density used in the fabric, fabric pattern and warp/weft densities have influences on the performance of recycled fibers. Therefore proper parameters should be defined for each fabric. In this study, recycled polyester fibers which meet the automotive specifications have been successfully produced thanks to the appropriate settings in the fabric. As a result of this study 100% recycled polyester woven fabrics were developed.

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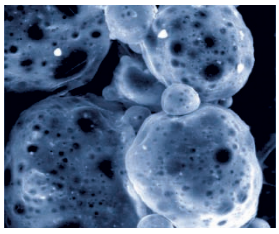

Improvement methodology of important clothing characteristics, by applying quality tools

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MIROSLAV DRAGIĆ
LJILJANA SORAK

REZUMAT – ABSTRACT

Metodologie de îmbunătățire a caracteristicilor importante ale produselor de îmbrăcăminte prin aplicarea instrumentelor de calitate

Această lucrare investighează posibilitatea de dezvoltare a unei metodologii care va permite îmbunătățirea cele mai importante caracteristici ale produselor și proceselor folosind instrumente calitative, în conformitate cu criteriile importanței acestora pentru satisfacția clientului. Acest lucru ar crește cota de piață, și, prin urmare, calitatea firmei. Prima fază a cercetării s-a axat pe stabilirea unei proceduri standard pentru identificarea dintre cele mai importante caracteristici de calitate ale produselor și proceselor, în conformitate cu criteriile de importanța lor pentru satisfactia clientului. În acest scop, a fost utilizată metoda QFD. A doua fază s-a concentrat pe stabilirea unei proceduri standard pentru identificarea caracteristicilor de calitate, cu cea mai bună clasificare, folosind metoda de benchmarking. A treia fază a fost realizată în două etape. Primul pas face referire la aplicarea procedurii standard pentru identificarea și compararea caracteristicilor importante de calitate ale produselor și proceselor. Al doilea pas s-a axat pe analiza rezultatelor de benchmarking, precum și la reproiectarea proceselor, în scopul de a creșterii caracteristicilor importante de calitate la nivelul celor mai bune din clasă. Aceste studii au indicat faptul că acele caracteristici de calitate ale produselor și serviciilor care nu sunt la cel mai înalt nivel ar putea fi îmbunătățite prin utilizarea instrumentelor de calitate corespunzătoare. Configurarea procedurile pentru aplicarea corespunzătoare a instrumentelor de calitate s-a dovedit a fi foarte importantă, de asemenea.

Cuvinte-cheie: produs de îmbrăcăminte, proces, benchmarking, metoda QFD, industria confecțiilor.

Improvement methodology of important characteristics by applying quality tools to clothing products

This paper investigated possibility of developing a methodology that will enable improvement of the most important characteristics of products and processes using quality tools, according to the criteria of their importance to customer satisfaction. This would increase market share, and consequently, the quality of the firm. The first phase of research focused on the establishment of a standard procedure for identification of the important quality characteristics of products and processes, according to the criteria of their importance to customer satisfaction. For this purpose, the QFD method was used. The second phase focused on setting up a standard procedure for comparison of the identified important quality characteristics with the best in the class, using the benchmarking method. The third phase was carried out in two steps. The first step referred to application of the standard procedure for identification and comparison of important quality characteristics of products and processes. The second step focused on the analysis of the benchmarking results, as well as on the process reengineering in order to raise important quality characteristics to the level of best in class. These studies indicated that the quality characteristics of products and services that are not at the highest level could be improved by using the appropriate quality tools. Setting up the procedures for the proper application of quality tools was found to be very important as well.

Keywords: clothing product, process, benchmarking, QFD method, clothing industry.

World market today is characterized by presence of very strong competition and many fast changes in the environment. The confluence of management and technology creates an environment with a huge potential of managerial innovation as a key vehicle towards the enterprise competitiveness [1]. It is a known fact that only companies which have learnt how to adapt quickly and efficiently and which are fast learners will survive. In order to live with the changes modern management has developed numerous techniques for achieving better business results.

Because of the aforementioned reasons companies must use different quality tools in order to be efficient and effective in the short and long term. They are used for tracking, measuring, analyzing and improving product and service performance. They are developed by successful companies with the goal to satisfy the customers and other parties involved.

If applied fairly, quality tools provide continues improvement with the minimum investment. It is well known fact that the product and service quality is the most important factor on which competitive position of the company in the market depends, because under today's circumstances most customers value quality over price.

The mentioned reasons imply that there is a need to develop a methodology which will improve identified important characteristics of product and process quality, according to importance criteria for customer

satisfaction, by comparison with the best in class. Because of that, the following specific goals were defined in this study:

- To develop a methodology for improvement of identified important characteristics of a product's quality;
- To identify through the research in the most precise way important characteristics for product quality, which are not on the same level as the best in class;
- To identify processes which result in the important characteristics of product quality, which are not on the same level as the best in class, and
- To suggest – through analysis of identified important characteristics of products and processes – ways to raise their quality to match the level of the best in class.

The purpose of increased attention to important characteristics of product quality is to use feedback to increase spotted applicable value of the product. Spotted applicable value is the value which is visible to a customer when buying or using a product. This is the reason why the companies aim to increase their competences necessary to permanently increase spotted applicable value of a product [2].

Development of textile industry in last decades on global level was marked by intensive structural adjustment in production in developing countries [3]. Bosnia and Herzegovina has a long tradition and good international reputation in textile and clothing sector. In BIH there are about 100 companies in this sector, with approximately 20 000 workers. Product structure is very diverse: heavy clothing male and female, light clothing, sports clothing, seat covers, lingerie, protective outfit, medical program, cooking program, home textile etc. Similar to other neighboring countries, textile, leather and footwear industry in BIH is significant segment of processing sector, it is work-intensive and low-accumulative industry branch. It employs mostly female work force and it contributes to lowering unemployment rate, although the average net salary in this sector are lower compared to other processing *industry branches* [4]. *Production is mostly based on Lohn-chores (sewing, cutting, processing, assembling)* for foreign partners, with very little participation in production of own products.

Since in recent past big structural changes occurred in textile-production business systems in the region, as a consequence of bankruptcy of big systems, privatization and adjustment of inflexible production business systems to the market [5], today we have in Bosnia and Herzegovina number of new small companies which – thanks to their own efforts – have managed to find a place in the market, create own collection and recognized and well accepted brand. But they should not stop here and be happy with what was achieved, they must accept the benchmarking concept, which essentially represents process of constant measuring and comparing own product and/or services with well-known and leading in that production.

DEVELOPMENT OF METHODOLOGY FOR IMPROVEMENT OF PRODUCTS AND PROCESS IMPORTANT CHARACTERISTICS BY APPLYING QUALITY TOOLS

Since all companies wish to constantly increase their effectiveness and efficiency, there is a need to develop a methodology for improvement of identified important characteristics of product, service and process quality. It should incorporate development of standard procedure for:

- Application of QFD method for identification of important characteristics of product, service and process quality, and
- Application of benchmarking method for comparison of identified important characteristics of product, service and process quality with the best in class.

Based on developed methodology, companies should:

- Identify important characteristics for product and process quality;
- Perform benchmarking of the product, service and process;
- Re-engineer process which leads to improvement of important characteristics of product, service and process quality, and
- Carry out self-evaluation of implemented methodology.

Identification of important characteristics in product, service and process quality

The main goal of QFD method is identification of important characteristics in product, service and process quality, which will enable customers and other parties involved requests and expectations satisfaction.

Namely, this method demands determination of product/service characteristics which are pre-required for fulfillment of previously defined customer demands. Based on that, critical points in product/service and in a production process or service performance are identified, points which are connected to defined characteristics. After that, procedures for handling those critical points are defined. In this way the final goal is reached, and that is elevation of quality level which corresponds to customer's demands.

QFD is abbreviation for *Quality Function Deployment,* which would literarily be translated as "deployment of quality function", but QFD usually mean "quality planning toward customer-user demand". It was developed in Kobe (Japan) during '70-ies in Mitsubishi shipyard, but soon it was applied in other industries. Toyota QFD has been using this method since 1973, and later it was accepted also by Honda (1979), Ford (1983), Volvo and Saab (1987). Wider use in developed countries of Western Europe and USA was reached in 1991. Today QFD method is an integral part of national standard methods which are used in Japan.

Experience in application of QFD method points to significant effects which derive from this method, such as:

- Simplifies and shortness time needed for product or service planning (through decreasing possibility for an error and returning during the planning);
- Improves quality of a product or service (customer is offered exactly the quality which was demanded);
- Decreases total expense of planning and production (risk of error is minimized) [6].

Standard procedure for QFD method implementation was established in order to apply this method in the most effective and efficient way. It is conducted in four steps.

The first step refers to identification of needs and expectations of product or service users and other parties involved, through market research and contracting. These are written in the left part of QFD map.

In the second step expert teams identify important characteristics of a product or service which satisfy expressed demands, and then determine their interdependence. In this way are identified 5 to 7 most important characteristics of a product.

The third step refers to relocation of chosen most important characteristics of a product or service to the left side of QFD map.

In the fourth step expert teams identify important processes which lead to chosen most important characteristics of a product, and then they determine their interdependence. In this way are identified 5 to 7 most important processes which lead to achieving products most important characteristics.

Benchmarking product, service and processes

During the eighties management techniques were complemented with Benchmarking, now widely acknowledged and affirmed tool of modern management. This technique implies that the goals and means of achieving them are defined according to the practice of the best companies in the world. Benchmarking as a part of strategic management gives guidelines to a company how to improve its business processes, technical solutions and functions.

Benchmarking is one of the most important TQM methods (Total Quality Management), because it contributes to better understanding of company's guidance. That is why today it represents acknowledged and often applied procedure in modern business. Term Benchmarking means continuing process of identification, understanding and adjusting a product, service, equipment and procedures of a company to the best in class, i.e. learning from the organizations which have established the best practice, with the goal to improve own business.

Benchmarking is becoming more popular especially in recent years. Benchmarking process is more than just a means of gathering data about how much is one company doing better business than others. Benchmarking can be used in different industries, companies engaged in production and services. It is a model for identification of new ideas and new ways to improve processes, so it has to be able to fulfill expectations of customers [7].

Etymology of the term "Benchmarking" originally marked distance or starting point from which the distance is measured. Set benchmarking shows the distance from that starting point. For every organization it is important to have set clear goals, and benchmarking is reliable indicator for organization where it is relative to mapped goals. It is a continuous process of identification, understanding and adjusting products, services, equipment and procedures of a company with the best in class. Best organizations in class means leading organizations which have the same or similar production or service program [8].

Learning from the organizations which have established the best practice (Best Practice Organization) improves own business. Identification of the best practices is also one of the critical phases in every benchmarking process [9]. Because of that Benchmarking also implies finding reasons and methods because of which some organizations perform the same tasks in more efficient and more effective way than others. The goal of benchmarking some organization is analysis of the best ideas and business modes of the best organizations in a certain class, which can be copied and possibly improved.

There is a significant number of different approaches to organizing and conducting styles in benchmarking research. Most of this research has in common that certain parameters (relevant for observed process) are in some way quantified and then compared with the best practice. Data is usually gathered by poll, then statistically processed and compared. Similar approach is present in many references, for example [10, 11, 12, 13].

Measuring performance and benchmarking are major techniques often used in leading researches for improvement of company's performances, like for example Gunasekaran with associates [14] suggests that company performance measuring should enable spotting priorities for improvement and spotting actions to conduct those improvements. However, in spite of great possibilities, quantitative approach still isn't sufficiently present in benchmarking. This especially refers to application of benchmarking in practical conditions in most countries in transition, where it is seldom used.

In this sense, this work has considered possibility of developing methodology for benchmarking method application, with the goal to improve characteristics of products and services in real conditions to satisfy customers demand regarding product and service characteristics. This is multidisciplinary problem in which experts from different areas should be involved. This specially refers to experts who are dealing with the problem of quality management, marketing and development of products and services. In this sense J. Todorović [15] emphasis that companies which want to gain and sustain competitive

advantage must perform benchmarking with the competition, to discover key success factors. In this way they can obtain key data which are the basis for creation of strategic and operational plans.
In order to apply benchmarking method in the most efficient and effective way in company working processes it is necessary to establish standard procedure for its application. This is done in five steps.
The first step is forming a team for benchmarking product. It should include five to ten members.
In the second step team leader should prepare samples of similar products from three to five companies for which it was established that are leaders in the class and deliver them together with the table "Product characteristics mark" in which are given five to seven most important product characteristics derived from QFD method for evaluation.
The third step refers to marking identified important characteristics of samples by team members with the mark from one to five. After that team members have to submit the filled table to team leader.
In the fourth step team leader should calculate average mark across all important characteristics of a product from own production program and products from production program of the best in class.
The fifth step refers to analysis of results and suggestions in which directions to improve identified important characteristics of product quality [16].

RESEARCH PART

Research part which refers to application of developed methodology for improvement of identified important characteristics of clothing product quality was conducted in textile-clothing company *Exclusive-commerce,* Banja Luka, Republic of Serbia, Bosnia and Herzegovina. Since the company wishes to be competitive on global market, for benchmarking were chosen four well-known European companies whose products are competitive to *Exclusive-commerce* by the characteristics, and they are:

- *LISCA moda* Ltd, Sarajevo, Bosnia and Herzegovina;
- *Textiles Well S.A,* Le Vigan, France;
- *Lormar,* Modena, Italy and
- *Simone Perele,* Paris, France.

The first company whose products were analyzed and at the same time the company where this research was conducted is company *Exclusive-commerce*, Banja Luka, which perform business in textile industry since 1988 and its specialized for production of fashionable lingerie. Entire production equipment is from well-known Japanese producer JUKI with modern appliances and devices, which provide achieving satisfactory level of production and reliability in performance. Company also has modern IT equipment with software for tailoring (CAD system) and software for support of business processes. Today is company *Exclusive-commerce* a modern company with highly qualified employees, modern technology, automated drive, work and computer equipment, working mostly with foreign materials. Company offers quality product on domestic and foreign market. It exports to fastidious French market 80% of its production. Company *Exclusive-commerce,* Banja Luka was certified in 2002 according to ISO 9001:2000 standard, and in 2009 with ISO 9001: 2008 standard by SGS Switzerland. In order to function with the highest quality and get included to international market, the company has established politics and goals of quality and procedures for application of quality tools for improvement of quality of own products and processes.
The second company whose product characteristics were analyzed is company *LISCA moda Ltd.* Sarajevo. Company was founded in 1955 as a small craft workshop for repairing stockings, which later specialized for lady lingerie. In all periods of its existence the company managed to keep own development and persist the crisis mostly thanks to its original production program, specialization and quality. Company *Lisca* pays great attention to product quality, but also to process quality, level of business results and factors which influence them. Today company *Lisca* is well-known producer of lingerie, swimsuits and blouses in European market, following the wishes of buyers with their modern products of high quality which adhere to female body and satisfy their need for beauty, attractiveness and self-confidence.
Next company whose products were analyzed in this research is *Textiles Well S.A.* which produces and sells whole specter of female and child underwear. It does business through the chain of retail stores in France. Company was registered in 1974 in Le Vigan, France, and as of November 2010, *Textiles Well S.A.* is a branch of *CSP International Fashion Group SpA*.
Among analyzed products are also products of company *Lormar* which was found in 1971 in Modena, Italian capital of lingerie. From a small workshop which produced corsets to 2000 when it grew into recognized company with stable retail network in over 20 countries. Today this company employs directly and indirectly more than 800 workers and sell more than 8 million products per year. Production program comprises lingerie, swimsuits, sleepwear, under the slogan „Love Nature by Lormar" and are created by using natural materials which were not treated with chemicals and by promoting recycling. They have three established brand names: "Infiore", "Chiarodiluna" and "Sollievo".
And the final company whose characteristics were analyzed is company *Simone Perele*, founded in Paris by corset maker Madam Perele in 1948. Brand is proud for its high standards, attention to details and quality of the finish. With over 60 years of experience, this house of exclusive underwear combines perfectly knowledge and style. The brand was intensively developing mostly thanks to innovative technologies combined with products adjusted to women's wishes and expectations.

Table 1

MARKS OF PRODUCT CHARACTERISTICS IN 2012					
CHARACTERISTIC'S NAME	PRODUCT BY COMPANY				
	EKSKLUZIV	LISCA	WELL	INFIORE	SIMONE PERELE
Product design	3,8	3,8	5,0	4,2	5,0
Quality of material	4,2	3,3	4,2	3,6	4,1
Quality of manufacturing	3,2	4,1	4,8	4,9	3,8
Packaging	3,4	4,1	4,1	4,8	4,1
Product's price	4,7	3,5	4,6	4,1	3,3
AVERAGE MARK	**3,9**	**3,8**	**4,3**	**4,3**	**4,1**

Table 2

MARKS OF PRODUCT CHARACTERISTICS IN 2013					
CHARACTERISTIC'S NAME	PRODUCT BY COMPANY				
	EKSKLUZIV	LISCA	WELL	INFIORE	SIMONE PERELE
Product design	3,6	4,3	5,0	4,4	5,0
Quality of material	4,2	3,4	4,1	3,8	4,2
Quality of manufacturing	3,8	4,2	4,8	4,9	4,5
Packaging	3,7	4,1	4,1	4,8	4,3
Product's price	4,6	3,8	3,6	4,2	3,2
AVERAGE MARK	**4,0**	**4,0**	**4,3**	**4,4**	**4,3**

Research results

The first phase of the research referred to identification of important characteristics in process and product quality, according to the criteria of their importance for satisfaction of consumers. For that purpose team of nine members was formed: three from company "Exclusive-commerce" Banja Luka where the experiment was held (company's CEO, development manager and marketing manager), two representatives from company "WELL" from France, for which the LOHN tasks are performed, two representatives, two representatives from Faculty of Technology, Banja Luka, from textile department and two representatives from Faculty of Technology and Metallurgy from Belgrade, Serbia, also from textile department. Marketing manager was chosen for a team leader.

Team has identified in the first phase by applying QFD method five most important characteristics of the products. These are: design of a product, quality of material, quality of manufacturing, packaging and price of the product.

The second phase referred to benchmarking of the products. First product benchmarking was performed at the end of 2012, and second at the end of 2013. Marketing manager gave a form "Marking products characteristics" to all team members (table 1) so that they could mark aforementioned five most important characteristics of the products.

Marketing manager gathered the results of marking, calculated average marks across important characteristics of products and wrote them in the same form.

Average mark across characteristics from the benchmarking team for 2012 are given in table 1.

Since "quality of manufacturing" characteristics showed to be the most critical, it was suggested to carry out re-engineering of operation "sewing rubber band" by buying 2 machines which perform this task. The result of this improvement is increased average mark for quality of manufacturing for 0.4 by product benchmarking team in 2013 (table 2). Also, to improve characteristics of packaging switch was made from hangers to hard packaging. The result of that improvement is increased average mark for packaging for 0.5 by product benchmarking team in 2013 (table 2).

Since in 2013 "product design" characteristic showed to be the most critical, it was suggested to carry out improvement by hiring designer.

Analysis of table 1 and 2 showed that competitors also invested significant effort to increase performance of their products. The result of such effort is increased average mark for product quality by product benchmarking team in 2013 (table 2).

Finally, self-evaluation of performed methodology was conducted. It showed that quality tool application is far more than strategy of coping and imitating. Their major usefulness is in encouraging innovative and creative abilities of employees in textile and clothing company *Exclusive-commerce* Banja Luka.

CONCLUSIONS

The research conducted in this study had the goal of showing that with correct application of quality tools through defined procedures for its application it is possible to improve important characteristics of a product. In this way spotted utilizing value of a product by a buyer is increased, and as a consequence market share and quality of company's business is increased.

From the aforementioned, it can be drawn the following conclusions to the development of methodology for improvement of identified essential characteristics of quality products, can properly direct the process of identifying and reviewing the essential characteristics of products and processes in order to improve them. By comparing the (benchmarking) identified important characteristics of quality it is possible to reach important characteristics of product quality which are not in line with the best in class. Re-engineering and improving processes it is possible to raise the level of important characteristics of product quality which are not in line with the best in class, and important characteristics of product quality should constantly be questioned, because that is what the best in class do

in order to raise spotted value of a product. Strategy and goals of the organization and its benchmarking partner should be similar, otherwise the critical success factors and the relative importance of individual business processes of these organizations can vary significantly.

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Application of amorphous silica, colemanite and pumice on cotton fabric by screen printing method

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REZUMAT – ABSTRACT

Aplicarea de SiO_2 amorf, colemanit și piatră ponce pe un material din bumbac, folosind metoda de imprimare cu șabloane

Scopul acestui articol este acela de a descrie modalitățile de eliminare a dezavantajelor comune legate de utilizarea produsului final și eficiența de operare observată în diverse procese de finisare. Pentru a obține materiale textile funcționale, au fost utilizate doar substanțe naturale, în loc de substanțe chimice de finisare, obținându-se astfel diferite efecte. Țesătura de bumbac a fost imprimată cu colemanit, dioxid de siliciu amorf și piatră ponce, ulterior fiind investigate proprietățile sale funcționale, cum ar fi caracterul ignifug și hidrofob, rezistența la tracțiune și permeabilitatea la aer. Aplicând această metodă de producție, este posibilă obținerea unor țesături ignifuge, hidrofobe, cu rezistență ridicată la tracțiune, permeabilitate scăzută la aer și cost redus. Se poate concluziona că procesul de finisare investigat dă rezultate promițătoare și este ușor aplicabil în procesele textile.

Cuvinte-cheie: imprimare, tratament de finisare, material de bumbac, rezistența țesăturii, ignifugare

Application of amorphous silica, colemanite and pumice on cotton fabric by screen printing method

The aim of this article is describing the ways of eliminating the common disadvantages related with the usage of end product and operating efficiency observed in various finishing processes. In order to obtain functional textile materials, substances with completely natural essences were used instead of finishing chemicals and thus various effects were achieved. The cotton fabric was printed with colemanite, amorphous silica and pumice and then its functional properties such as tensile strength, flame retardancy, water repellency, air permeability were investigated. Applying this production method, it will be possible to obtain fabrics with high tensile strength, flame retardancy, water repellency, less air permeability and low cost. It can be concluded that the investigated finishing process is giving promising results and is easily applicable in the textile processes.

Keywords: Printing process, finishing process, cotton fabric, tensile strength, flame retardancy

In the last two decades, the growing global competition in the textile field has led to an increase in academic and industrial research activities towards new applications and innovatory solutions. The textile developments gave the materials various properties such as non-flammability, water repellency and weather resistance which make them more suitable. Synthetic fiber gains these properties at the stage of polymerization and natural ones at the stage of production through chemical finishing processes.

Today, cotton, which is composed of cellulose molecules arranged in fibers, and which is inexpensive, brings great importance and utility to the textile fiber [1, 2]. Cellulose is a biological and functional material, made of glucose with beta 1,4-glucoside bonds, and has more than ten thousand of these D-glucopyranose sections. Chemical and physical properties of cellulose involve not only strength and elasticity of the fiber but also water affinity (due to its many hydroxyl groups) as well as permeability.

In terms of the finishing processes, cellulose has a great potential because of its hydroxyl groups. As a result of these processes, the products gain such functional properties as flame retardancy, crease resistance and water repellency, etc.

Development of flame retardant treatments for materials has been increasingly gaining importance due to the fact that cellulose fibers ignite and burn easily. Since most compounds used in the textile market are water-soluble inorganic salts that are easily removed by water, rain or perspiration, the protection they provide is only temporary. Boron (polybromide diphenyl-ether PBDE) and phosphorous [3, 4] are among the widely used compounds. In order to obtain durable flame retardancy, organophosphorous compounds are used. As durable flame retardants for cotton, cellulose and cellulose-blend clothing fabrics, tetrakis-hydroxymethyl-phosphonium-salts such as Proban (Rhodia; - tetrakis-hydroxymethyl phosphonium chloride (THPC-urea) and phosphonoalkylamide such as Pyrovatex CP-new (Huntsman- organic phosphorus compound, fiber-reactive) are used.

There are various chemicals used to make fabrics water repellent. These include aluminum and zirconium soaps, waxes, wax-like substances, metal complexes, pyridinium- and methylol- compounds. A good water repellent finishing agent with adequate

fastness is provided by using methylol stearamide with partially formed urea-formaldehyde. Application of fluoropolymers such as Scotchguard (3M) and Teflon (Du Pont) [5] will help to obtain oil and soil repellency in addition to water repellency.
It is important that fabrics should have high resistance for marine, aviation and automotive industries and also for defense purposes. Various compounds and composites can be used to increase the resistance of textile materials [6, 7].
For each finishing process that is applied in order to give functional properties to the product, different chemicals and auxiliaries are needed. These finishing processes have some drawbacks as they cause some problems with the productivity of mechanical properties of the fabric. There are two factors contributing to the reduction of mechanical properties: one is the degradation of fibers caused by the acid catalysts and the other is restriction of stress distribution within the fibers owing to their rigid cross-linking agents [8, 9]. There are also many harmful substances such as nitrogen, phosphorus, formaldehyde on the fabric as a result of the finishing processes. Furthermore, some finishing processes also have excessive cost values. Flame retardancy is an example of this issue. Because of the high cost of the finishing processes the laboratory-to-bulk procedure for potential orders can not be realized.
Pigment printing, which enables the coloring and finishing processes to be applied at the same time, is one of the most essential methods. With finishing chemicals added to the pigment printing paste, various finishing processes and special printing effects such as relief, metallic, and peach effects are obtainable.
In this study, in place of fulfilling the finishing processes separately, the cotton fabric was printed with the pigment paste included in the completely natural amorphous silica and pumice, which are peculiar to Isparta and also the only nonmetallic ground colemanite. After printing, the fabric was dried and cured. The physical properties of all treated and non-treated fabrics have been tested. The test results of all samples have been discussed.
Thus, not only basic but also safer and more cost-efficient technology model is to be used in textile mills in combination with printing.

MATERIALS AND METHODS

Materials

All the experiments were carried out with desized, scoured and bleached %100 cotton fabric (186 g/m^2) obtained from the Anteks Corp., Turkey. The capillarity degree of the cotton fabric is shown in table 1.
Amorphous Silica: Amorphous silica is a sedimentary rock formed as silica sediments on the surface and as underground arteries near the waters with chloride and neutral alcali pH and as the fluids which contain colloidal silica particles with a certain degree of heat and balanced resolubility coming out to the surface of the earth and reaching over satiety [10, 11]. Amorphous silica is used in construction building. For example, concrete containing amorphous silica meets the demand for hard, durable concrete for high performance in industrial usage; provides optimum concrete flow within the plastic cracking limits, decreases shrinkage and also enables plastic settlement.

Table 1

CAPILLARITY VALUES OF COTTON FABRIC (DIN 53924)

Time (sec)	Weft (mm)	Warp (mm)
10	15	10
30	24	20
60	30	26

Boron's chemical symbol is B and it is the only nonmetallic element of the IIIA group. This is the element with the greatest similarity to carbon and silicium elements and also with a very high affinity to oxygen [12, 13]. Some commercially important boron minerals are tincal, kernite, colemanite, ulexite, datolite and hydroboracite. In this study, colemanite has been used. Colemanite is not a synthetic compound but a natural mineral that contains some impurities.
Pumice is amorphous foam produced during volcanic eruptions. There is a plenty of pumice formed during explosive volcanic eruptions in Isparta. Turkey has a seven billion m^3 total pumice reserve [14]. It is used in the fields of construction, chemicals, agriculture, and dentistry.
In this study, amorphous silica was obtained from Keciborlu, Isparta, colemanite from ETI Mining Balıkesir of Bigadic Boron corporations and pumice from the Gelincik zone in Isparta. These samples were sieved through Number 200 sieve with a diameter of 0.076 mm.
The physical and chemical compositions of these materials are shown as in table 2 [15].

EXPERIMENTAL MODELING METHOD

The pigment printing recipe was prepared with:

Colomanite, pumice or amorphous silica	50/100 g
Water	x g
Ammonium hydroxide solution (26%, Riedel-de haen)	4 g
Tubifast AS GKB (emulsion polymer self-crosslinking, copolymer based on styrene and acrylic acid ester, CHT [16])	150 g
Tubivis VP 681 (synthetic thickener, ammonium salt of carboxylic acid derivative, CHT [17])	17 g
Tubiprint Fixerer R (fixierer, melamine resin derivative, CHT [18])	10 g
	1000 g

The viscosity of the printing paste was 2600 mPa.s. (Brookfield DV-I prime viscometer, 20 RPM, 25 mm spindle) at 25 ºC degrees. The printing process was carried out using the flat screen technique. The paste

Table 2

THE PHYSICAL AND CHEMICAL COMPOSITION OF AMORPHOUS SILICA, COLEMANITE AND PUMICE				
Ingredients	**Unit**	**Colemanite**	**Pumice**	**Amorphous Silica**
B_2O_3	%	40.00	-	-
CaO	%	27.00	4.68	0.31
SiO_2	%	4.00-6.50	60.50	92.48
SO_4	%	0.60	-	-
As	ppm	35	-	-
Fe_2O_3	%	0.08	3.38	0.09
Al_2O_3	%	0.40	17.15	2.60
MgO	%	3.00	2.09	0
SrO	%	1.50	-	-
Na_2O	%	0.35	4.30	1.08
SO_3	%	-	0.16	0.09
K_2O	%	-	4.54	0.04
TiO_2	%	-	0.41	1.34
Loss of Ignition(LOI)	%	24.60	2.79	1.85
Specific Gravity (Gs)	-	2.42	2.40	2.39

was applied on the fabric at an amount of 271–272 g/m^2 (the screen fabric was made of 100 % PES monofilament and 55 yarn/cm).The printed samples were dried for two minutes at 100 ºC and cured for five minutes at 150 ºC (Mathis CH-8156). The printing procedure was repeated three times at different cases.

All the physical measurements after the printing process were carried out after conditioning the fabrics for 24 hours under the standard atmosphere conditions (20±2 ºC temperature and 65±2 % relative humidity).

The tensile strength of the fabric was measured with Lloyd and assessed according to TS EN ISO 13934-2; air permeability of the fabrics was measured by the Textest FX 3300 model Air Permeability Tester according to ASTM D737 – 04, and water repellency was measured according to AATCC 42-2007 Water resistance: Impact penetration test. The flame retardancy tests which were carried out using the 45º flammability tester, Bellmore, model no: TC–45 were evaluated according to ASTM 1230 standard. The laundry was done using a front-loading Wascator machine (Electrolux FOM) with 2.0 kg loads consisting of coated fabric samples and 100% PES ballast fabrics. All the washing cycles were carried out according to BS EN ISO 26330 Standard (5A program) with 4.0 g/L dose of standard European Colour fastness Establishment (ECE) reference detergent (non-phosphate detergent without optical brightener and enzymes) at 40 ºC with normal agitation. This laundering process was repeated 20 times. The samples were dried with dry flat in laboratory condition for 24 hours. For determination of bending rigidity, ASTM D1388-08 (Heart Loop Method) was used.

The chemical and morphological surface properties of the cotton printed with colemanite, amorphous silica and pumice were investigated using Fourier transform infrared spectroscopy (FT-IR), and scanning electron microscopy (SEM) methods. Fourier transform infrared (FT-IR) spectra were recorded on FT-IR System Spectrum BX, Perkin Elmer, using potassium bromide disks. A total of 16 scans for each sample was taken with a resolution of 2 cm^{-1}, within a range of 4000–600 cm^{-1}. SEM images were taken on a scanning electron microscope model Tescan Vega II LSU.

RESULTS AND DISCUSSION

Tensile strength values of printed fabrics

After the printing process, the tensile strength and elongation values of the cotton fabrics printed with colemanite, amorphous silica and pumice were measured. In table 3, the total tensile strength values (warp+weft) and elongation values of all printed and non-treated fabrics are given. Furthermore, the tensile strength and elongation values of the samples were tested again after 20 consecutive washing cycles. This process is done in order to determine the durability of the process carried out.

As seen in the values from table 3, the tensile strength of the cotton fabric increased significantly after the printing process. The greatest increase was observed in fabrics printed with colemanite and amorphous silica. Whether an amount of 50 g/kg or 100 g/kg of colemanite and amorphous silica was used and seen that there is no difference in the results. While the amount of increase is 126.05 % in the fabric printed with amorphous silica, it is as much as 139.27 % in the fabric printed with colemanite. While the fabric coated with 50 g/kg pumice shows an increase of

Table 3

THE TENSILE STRENGTH AND ELONGATION VALUES OF THE COTTON FABRICS PRINTED WITH COLEMANITE, AMORPHOUS SILICA, PUMICE AND AFTER 20 WASHING CYCLES

				After 20 washing cycles	
	Amount(g/kg)	F(kgf)	E(%)	F(kgf)	E(%)
Colemanite	50	1483.64±4.30%	25.67±4.25%	1097.17±3.85%	30.65±4.00%
Colemanite	100	1489.99±4.25%	26.14±4.35%	1116.56±4.27%	24.99±3.45%
Amorphous Silica	50	1404.12±4.40%	25.19±4.58%	1053.46±4.10%	28.85±4.21%
Amorphous Silica	100	1407.70±4.53%	25.98±4.20%	1069.32±4.43%	26.70±4.42%
Pumice	50	820. 85±4.18%	12.82±4.45%	809.28±4.15%	12.25±3.95%
Pumice	100	866.51±4.43%	11.17±4.27%	840.10±4.24%	11.28±3.98%
Cotton Fabric	---	622.72±4.52%	9.96±4.10%	624.16±4.20%	9.80±4.00%

31.82 %, the one coated with 100 g/kg yields an increase of 39.15 %. From table 3, it can be observed that the screen printing process is rather resistant to washing. The results are shown in table 3 where the elongation (E) values of the printed fabrics have increased significantly. When parallel strength is applied onto the fiber axis of the untreated fabric all the macromolecules counter-act this tensile strength together. In this process, the reaction to the excessive pull forces (attractive forces) such as hydrogen bonding and Van der Waals forces between the natural cellulose macromolecules with high average degree of polymerization (DP2500 – 9000) can occur. The rupture is similar to the breaking of the oxygen bridges that bond the glucose structural components [19]. In the case of the treated fabric, an insoluble matrix forms in the medium of the printing paste, cotton fabric, colemanite, amorphous silica and pumice in addition to this strength and the elongation values increase.

Air permeability and water repellency values of printed fabrics

After the printing process, air permeability and water repellency values of the cotton fabrics printed with colemanite, amorphous silica and pumice were measured. In table 4, air permeability and water repellency values of all printed and non-treated fabrics are given. Furthermore, air permeability and water repellency values of the samples were tested again after 20 consecutive washing cycles in order to determine the durability of the process carried out.

According to table 4, air permeability of the cotton fabric was significantly decreased as a result of the screen printing processes. The best air permeability results were obtained from the fabrics printed with colemanite (120%) and amorphous silica, while no significant decrease was measured in their air permeability values after 20 consecutive washing cycles. As seen in table 4, the water repellency values of the fabric printed with colemanite and amorphous silica still has the same level of water repellent properties even after 20 washing cycles according to the standard numbered AATCC 42-2007. While the water repellency value of the fabric printed with pumice is good, it slightly goes beyond the standards after 20 consecutive washing cycles.

Flame retardancy values of printed fabrics

After the printing process, flame retardancy values of the cotton fabrics printed with colemanite, amorphous silica and pumice were measured. In table 5,

Table 4

AIR PERMEABILITY AND WATER REPELLENCY VALUES OF THE COTTON FABRICS PRINTED WITH COLEMANITE, AMORPHOUS SILICA, PUMICE AND AFTER 20 WASHING CYCLES

	Amount (g/kg)	Air Permeability (L/m^{2}s)		Water Repellency (g)	
		20 washing cycles		20 washing cycles	
Colemanite	50	10.19±3.31%	11.30±3.00%	2.08±3.15%	3.71±3.10%
Colemanite	100	9.60±3.35%	9.80±3.10%	1.77±3.05%	4.97±3.45%
Amorphous Silica	50	9.01±3.83%	10.14±3.75%	3.39±3.21%	4.52±3.13%
Amorphous Silica	100	8.97±4.10%	9.80±4.00%	1.76±3.00%	1.77±3.18%
Pumice	50	13.72±3.48%	15.85±3.38%	4.93±3.12%	5.72±3.54%
Pumice	100	10.80±3.75%	11.20±3.27%	4.25±3.42%	4.173±3.59%
Cotton Fabric	---	21.16±3.82%	22.48±4.15%	21.12±3.22%	22.50±3.78%

Table 5

FLAME RETARDENCE VALUES OF THE COTTON FABRIC PRINTED WITH COLEMANITE, AMORPHOUS +SILICA, PUMICE AND AFTER 20 WASHING CYCLES

	Amount (g/kg)	Ignition Time (s)	Yarn breakage time (s)	After 20 washing cycles
Colemanite	50	70	67.4±4.74%	43±4.45%
Colemanite	100	70	72.1±4.39%	49±4.28%
Amorphous Silica	50	70	68.4±4.29%	48±4.16%
Amorphous Silica	100	70	75±4.56%	55±4.00%
Pumice	50	70	57±4.90%	32±4.55%
Pumice	100	70	62±4.13%	46±4.76%
Cotton Fabric	---	70	2±3.80%	3±3.95%

flame retardancy values of all printed and non-treated fabrics are given. Furthermore, flame retardancy values of the samples were tested again after 20 consecutive washing cycles in order to determine the durability of the process carried out.

In table 5, it can be observed that the fabric printed with amorphous silica, colemanite and pumice has quite high flame retardancy values even after 20 washing cycles.

Surface morphology analysis

In figures 1, 2, 3, 4, are shown the SEM photos of the cotton fabric together with the fabric printed with amorphous silica, colemanite and pumice.

In figure 5, FT-IR analyses of the cotton fabric printed with colemanite, the colemanite itself, the cotton fabric printed with only printing paste and the cotton fabric are illustrated. While the peaks found at 1033–1037cm^{-1} in raw and printed fabrics show the existence of the, C-O,C-O-C groups of cellulose [20, 21], the broad band seen at 3300–3633 cm^{-1} exhibit the OH groups in the fabric. As can be seen in table 2, B_2O_3 constitutes an important part of the colemanite. B bands occur at 1200–1500 cm^{-1}. The peak at 1230 cm^{-1} indicates the existence of B-OH groups [22, 23].

In figure 6, FT-IR analyses of the cotton fabric printed with amorphous silica, the amorphous silica itself, and the cotton fabric printed with only printing paste and the cotton fabric can be found. As is stated in table 2 values, SiO_2 accounts for almost all of the silica. The peaks observed at 797-830 cm^{-1} in amorphous silica and the fabric coated with amorphous silica exhibit the Si-Si bonds, the ones found at 1040–1022 cm^{-1} exhibit the O-Si-O bonds, and those at 1171–1204 cm^{-1} show the Si-O-Si bonds [24, 25].

Figure 7, shows FT-IR analyses of the cotton fabric printed with pumice, the pumice itself, the cotton fabric printed with only printing paste and the cotton fabric. As is stated in table 2, SiO_2 constitutes an important part of the pumice. The O-Si-O peak, which takes place at 1032–1030 cm^{-1} in the pumice and the fabric coated with pumice, can also be observed in the same figure [26, 27].

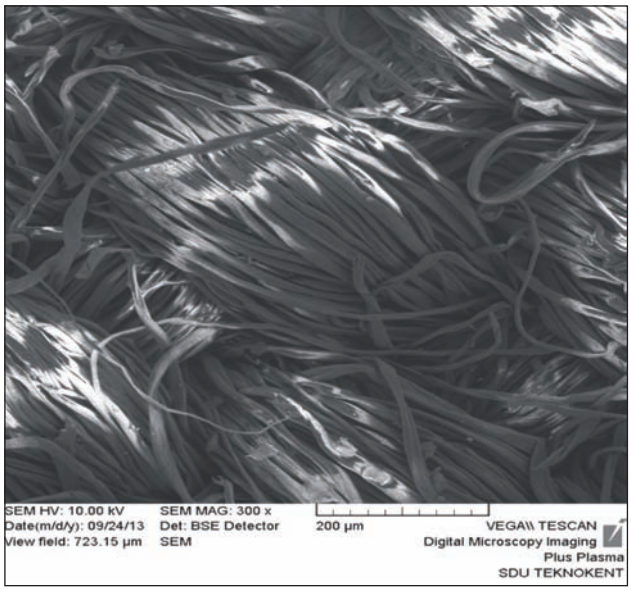

Fig. 1. SEM photo of cotton fabric

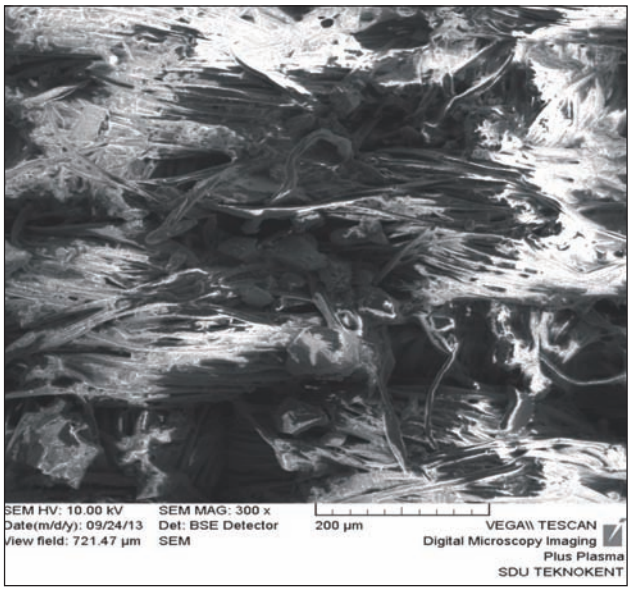

Fig. 2. SEM photo of cotton fabric coated with colemanite

Fig. 3. SEM photo of cotton fabric coated with amorphous silica

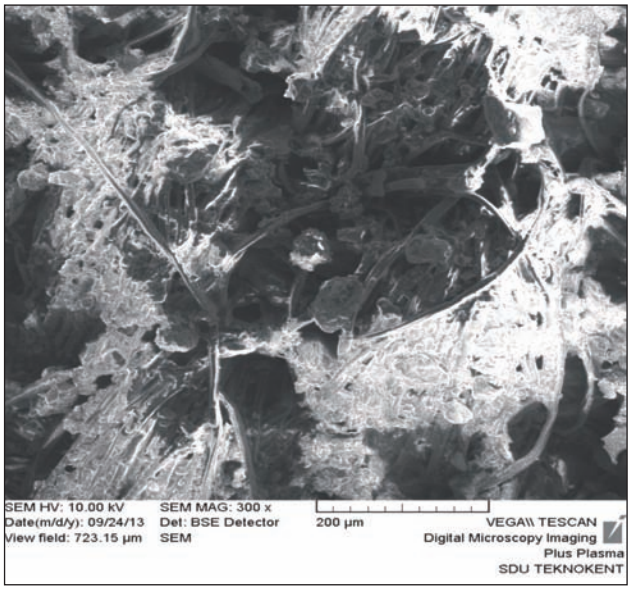

Fig. 4. SEM photo of cotton fabric coated with pumice

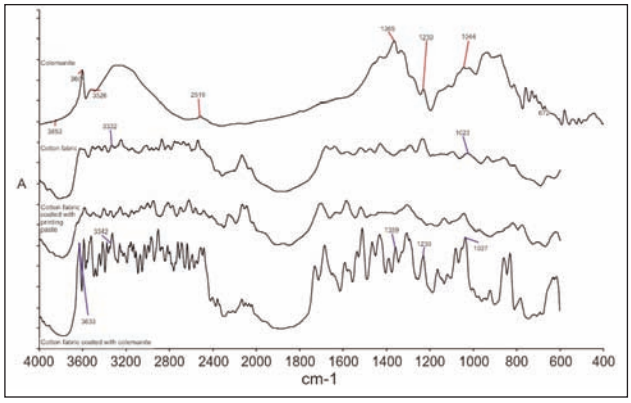

Fig. 5. FT-IR spectra of cotton fabric, cotton fabric coated with colemanite, cotton fabric coated with only printing paste and the colemanite

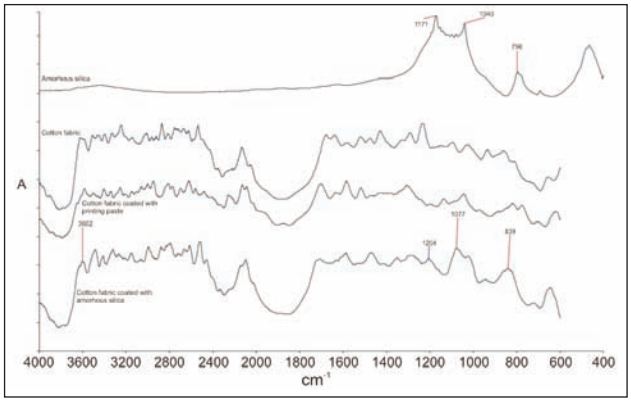

Fig. 6. FT-IR spectra of cotton fabric, cotton fabric printed with amorphous silica, cotton fabric printed with only printing paste and amorphous silica

According to these results, such values of the colemanite as high tensile strength and resistance to washing, water repellency and flame retardancy [28] are attributable to the bonding of the B_2O_3, which is present in the boron composition at a rate of 40 %. The presence of boron increases the high tensile strength and flame retardancy, water repellency and air permeability values of the cotton fabric. The fact that the amount of SiO_2 is bigger in amorphous silica than that in pumice increases the number of the bonding, which yields better physical properties of the fabric coated with amorphous silica compared with those coated with pumice.

Bending rigidity values of printed fabrics

The bending rigidity values of the cotton fabrics printed with colemanite, amorphous silica, and pumice and after 20 washing cycles are given in table 6.

From the table 6 values, it can be seen that the rigidity values increase as a result of the printing process and while this increase is quite high in the fabric coated with pumice, it is less significant in the fabric printed with amorphous silica and colemanite. The handle of these fabrics, which improves as a result of the washing cycles, is almost like the behavior of non-treated fabrics. As this study aims to obtain technical fabrics with high physical qualities, the drop in the handle values is considered negligible.

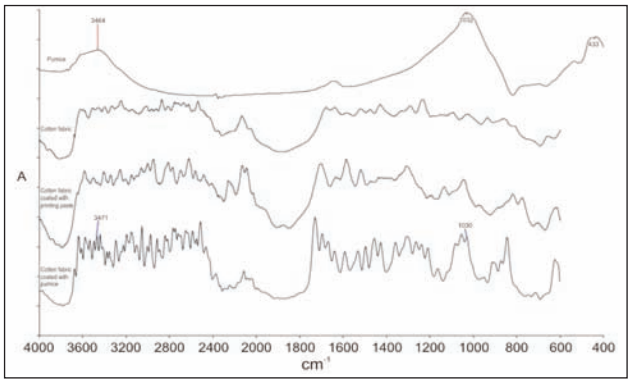

Fig. 7 .FT-IR spectra of cotton fabric, cotton fabric printed with pumice,cotton fabric printed with only printing paste and the pumice

Table 6

THE BENDING RIGIDITY VALUES OF THE COTTON FABRICS PRINTED WITH COLEMANITE, AMORPHOUS SILICA, AND PUMICE AND AFTER 20 WASHING CYCLES

					After 20 washing cycles		
		Bending Length (cm)*		Bending Rigidity (mg.cm)	Bending Length (cm)		Bending Rigidity (mg.cm)
		Warp	Weft		Warp	Weft	
Colemanite	(100g/kg)	2.48	2.22	291.59	2.10	1.92	191.07
Amorphous Silica	(100g/kg)	2.44	2.10	269.35	2.04	1.89	186.71
Pumice	(100g/kg)	2.54	2.33	343.86	2.33	2.03	250.62
Cotton Fabric	(100g/kg)	2.14	1.94	176.85	2.26	2.11	229.42

CONCLUSIONS

In this study, the physical properties of cotton fabric were investigated by adding amorphous silica, pumice and colemanite, the only nonmetallic element, to the printing paste instead of using finishing chemicals separately. An alternative production method was developed as a precaution against the problematic resistance drop in the finishing processes. After the pigment printing process, the tensile strength/elongation percentage, flame retardancy, air permeability values of the cotton fabric were investigated and it was found that these values were considerably increased. The values were resistant to 20 consecutive washing cycles. The SEM images of the printed and non-printed fabrics were taken and some important bonds of the samples were determined in the FT-IR analysis and the test findings were interpreted.

Since the purpose of this study is to obtain materials to be used in technical fields, the handling values that have been obtained is not important with those of the normal textile materials.

The paper, which focused on technological developments from laboratory up to pilot scale, has finally met the need for techniques to design and produce all the steps by natural products for technical textiles. Undoubtedly, the treatment of cotton fabrics with these innovative natural substances provides various advantages such as high quality of the end product, as well as economical and ecological textile production.

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The photocatalytic effects of textile materials treated with TiO_2 and Fe/TiO_2

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REZUMAT – ABSTRACT

Efectele fotocatalitice ale materialelor textile tratate cu TiO_2 și Fe/TiO_2

*Lucrarea de cercetare a avut drept scop determinarea activității fotocatalitice a materialelor textile tratate prin impregnare-uscare-condensare și cationizare-impregnare-uscare-condensare cu TiO_2 si TiO_2 dopat cu fier. Eficiența fotocatalitică a fost evaluată prin măsurarea diferențelor de culoare (dL*da*db, dE*, dC*, dH*) ale materialelor expuse și ne-expuse la lumina vizibilă și UV pe un spectrofotometru Hunter lab. Activitatea antifungică s-a evaluat conform standardului ISO 20743:2007. Rezultatele demonstrează că sub lumina vizibilă, independent de metoda de tratare, cea mai intensă decolorare se produce pe țesătura de poliester tratată cu TiO_2-Fe. Sub lumina UV, cea mai intensă decolorare este indusă de TiO_2-Fe pe materialele cationizate. Tratamentul de cationizare nu are efecte pozitive asupra fotodegradarii MB, diferențele de culoare și luminozitate fiind similare sau mai mici decât cele obținute în cazul tratării materialelor doar prin impregnare. Gradul de decolorare al țesăturilor din PET si PET/co pătate cu MB este mai mare sub lumina vizibilă decât sub lumina UV. Toate probele demonstrează o foarte bună reducere a Candida a., de 100% în cazul probelor tratate prin impregnare. Ratele de reducere ale Epidermophyton f. variază între 46,87% (D-V1-Fe) și 99,18 % pentru A-V2-Fe.*

Cuvinte-cheie: *TiO_2 dopat cu fier, fotocatalizatori, textile, anti-fungice, impregnare-uscare-condensare*

The photocatalytic effects of textile materials treated with TiO_2 and Fe/TiO_2

*The research has been focused on the photocatalytic activity of the textile materials treated with TiO_2 and TiO_2 iron doped by pad-dry-cure and cationization – pad-dry-cure. The fabrics have been exposed to UV and visible light. The photocatalytic efficiency was evaluated by measuring the color differences (dL*da*db, dE*, dC*, dH*) of the exposed and un-exposed samples on a Hunter lab spectrophotometer. The anti-fungal activity was assessed according modified ISO 20743:2007 standard. The results show that under visible light, independent of treatment used (padding or cationization – padding), the highest discoloration is produced by TiO_2-Fe on polyester. Under UV light, the highest discoloration is produced by TiO_2-Fe only for the cationized materials. The cationization pre-treatment has no positive effect on MB photodegradation, the colour and lightness difference being similar or smaller than those obtained in the case of padding treatment without cationization. The degree of discoloration of PET and PET/co fabrics stained with MB is higher under visible light then under UV light. In the case of cotton, a slightly higher discoloration under UV light is noticed. All samples yielded very good reduction rates of Candida a., with 100% reduction for samples treated by padding. The reduction rates of Epidermophyton f. varies between 46.87% (D-V1-Fe) and to 99.18 % for A-V2-Fe.*

Keywords: *Fe doped TiO_2, photocatalysts, textiles, anti-fungal, pad-dry-cure*

INTRODUCTION

Many attempts have been made to create photocatalytic textiles by the incorporation of titania with cellulose or cotton surfaces [1], graft nano TiO_2 on cotton fabrics using cross-link method [2–4], pad-dry-cure method [5, 6], using SiO_2 as binder [7], automated spray technique [8], sputtering [9].

Unfortunately, due to the low efficiency of TiO_2 under visible light and recombination between the photogenerated electrons and holes, the photocatalytic effect is almost non-observable. To increase the photocatalytic efficiency, TiO_2 was doped with metals [10, 11] and non-metals [12, 13]. Even a multitude of doped photocatalysts have been described by different authors, few data are found about the their deposition on textiles and the photocatalytic effects. In this research, TiO_2 and Fe^{3+} doped TiO_2 nanoparticles were deposited on three types of fabrics by padding and cationization–padding. The textiles coated as previously described were characterized by SEM, EDX, contact angles. The photocatalytic activity of the fabrics under UV and visible light was evaluated following the degradation of methylene blue.

MATERIALS AND METHODS

Materials

Fabrics: 100% polyester woven fabric (A), weight – 142 g/m^2, 100% cotton (D), weight – 260 g/m^2 and

50/50% polyester/cotton (G) weight – 201 g/m^2 fabricated by KIVANÇ Tekstil San. Ve Tic. A.S.
Chemicals
- **TiO_2**, rutile and anatase mixture (Aldrich), particles size below 100 nm.
- **TiO_2-Fe**, prepared by Kumoh National Institute of Technology (KIT), South Korea.
- **ITOBINDER AG:** polyacrylic binder (LJ Specialities, UK).
- **Itofix EZF**: polyethylene polyamine resin (LJ Specialities, UK).
- **BIOWET PB**: anionic surface agent.
- **PVP:** Polyvinylpyrrolidone (Aldrich).

Methods

Method to prepare photocatalytic solutions: 1 gram of Fe-TiO_2, respectively, TiO_2 was dispersed in 190 ml of distilled water on ultrasonic bath (US). 10 mL Biowet PB was added and stirred 30 minutes on US, then 1 g of PVP added and stirred for 30 minutes on US bath. 10 ml of polyacrilic binder (ITOBINDER AG) was dropped into above dispersion and intensively mixed by a mechanical stirrer.
Finally, the dispersion was diluted with 388 mL water and stirred for 1 hour on US to get a relatively stable solution with the following composition:
- solution 4: 1.67 g/L TiO_2-Fe, 16.67mL/L BIOWET PB, 1.67 g/LPVP, 16.67mL/L ITO; pH = 5
- solution 5: 1.67 g/L TiO_2, 16.67mL/L BIOWET PB, 1.67 g/LPVP, 16.67mL/L ITO; pH = 6

Methods to treat textiles

Two methods were used to coat the textiles with TiO_2 and TiO_2-Fe:

a. Pad-dry-cure: fabrics were padded 3 times with solutions 4 (**code 4-V2**) and 5 (**code 5 -**V1), and then dried at 120°C for 2 minutes and condensed to 150°C for 4 minutes.

b. Cationization – Pad-dry-cure: materials have been padded (foulard) in a solution containing 10 g/l Itofix EZF then were dried at 120°C for 2 minutes and condensed at 170°C for 1 minute. The dried materials were padded (foulard) with solution 4 (**code 4 - V4**), and respectively, solution 5 (**code 5 -V3**), dried at 120°C for 2 minutes and condensed at 150°C for 4 minutes.

Characterization of treated textiles

Analysis: shape, size and particle dispersion on fabrics' surface were analyzed on a scanning electron microscope (Quanta 200, FEI, Netherlands) equipped with an X-ray energy dispersive spectrometer (EDX) for identification of elements existing on materials. To evaluate the photocatalytic effect the fabrics were immersed in $2x10^{-5}$ mL/L methylene blue solution for 30 minutes, dried under infrared radiation source and exposed to UV (254nm) and in laboratory equipment Xenotest equipped with a xenon lamp that simulates the visible light. The photocatalytic efficiency was evaluated by measuring the color differences (dL*da*db, dE*, dC*, dH*) between the exposed and un-exposed samples on a Hunter lab spectrophotometer, at 10° observer angle and D65 light.

RESULTS AND DISCUSSIONS

SEM analyses

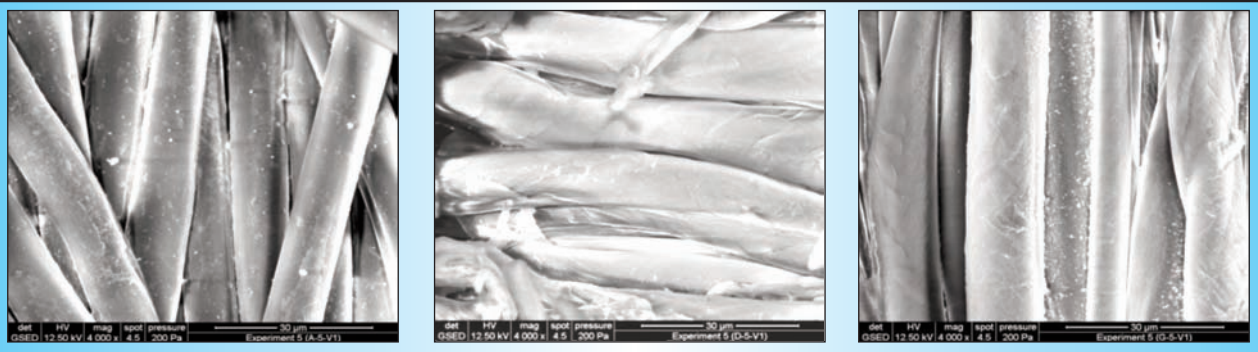
SEM images of treated materials are presented in table 1.
SEM images (table 1) demonstrates that by padding (V1, V2), regardless of used photocatalysts, polyester fibers are coated evenly with a higher number of particles, less agglomerated than those deposited on cotton. This phenomenon could be explained by the two slightly different types of interactions between the particles and materials surface. Thus, in the presence of water, the cellulose and polyester fibres gets a slight negative charge due to ionization of hydroxyl and, respectively ester groups. The polyester being hydrophobic and not having hydrophilic groups, the negative charge is less pronounced compared to the cellulose. The TiO_2/polyacrylate dispersion has a pH around 6–6.5. At this pH, sodium polyacrylate is dissociated into sodium ions and negative carboxyl groups. Sodium ions neutralize some of the negative charges (hydroxyl) of cellulose allowing the deposition of a larger quantity of polyacrylate than deposited on polyester material. In the same time, between the polyacrylate layers covering the textile materials and TiO_2 particles, which are also coated with a thin layer of polyacrylate, electrostatic repulsion forces occur, leading to a decrease of TiO_2 particles on cotton materials.
In the case of TiO_2 deposition on cationized materials (V3), SEM images show that TiO_2 particles are dispersed non-uniformly on all types of fibers, are present in the form of clusters with different shapes and sizes, the smallest being on the fibers polyester. Polyethylene polyamine cationic resin forms a more uniform and thinner layer on polyester fibers than that of cotton. In the case of TiO_2-Fe deposition on cationized materials (V4), Fe-TiO_2 particles are agglomerated, dispersed unevenly, in greater numbers on cotton than on polyester fabrics. The smaller particle clusters are found on polyester.

Quantification of the TiO2 and TiO2–Fe by EDX

The results of metal quantification by EDX are shown in tables 2 and 3.
The highest amount of TiO_2 is deposited by cationization – padding, on polyester fabrics (A5-V3) followed by polyester/cotton and cotton. The amount of TiO_2 deposited by padding (V3) on initially cationized material is ≈ 2.5 times higher on polyester fibers than on cotton fibers. By pad-dry-cure method (V1), the quantity of TiO_2 particles decreases in order polyester/cotton > polyester > cotton. On contrary, except the polyester fabrics, TiO_2-Fe particles are deposited in larger quantity by pad-dry-cure than by cationization-padding. The largest amounts of titanium dioxide doped with iron (V4) are deposited on the

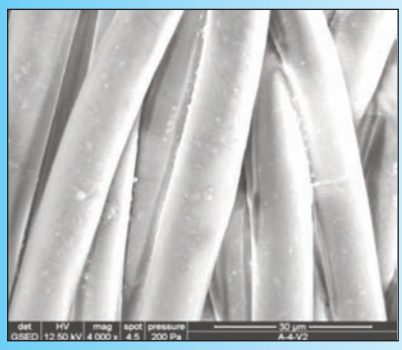
Table 1

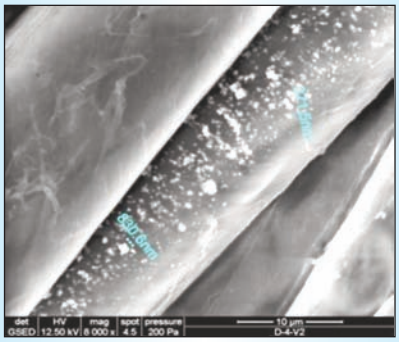
SEM IMAGES OF THE MATERIALS TREATED WITH SOLUTION 4 AND 5

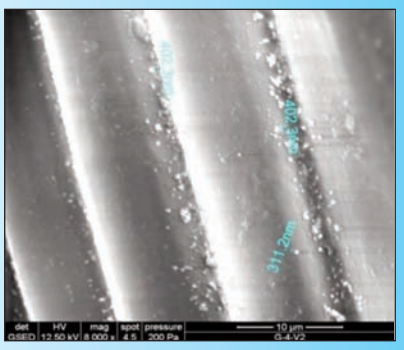

A5-V1

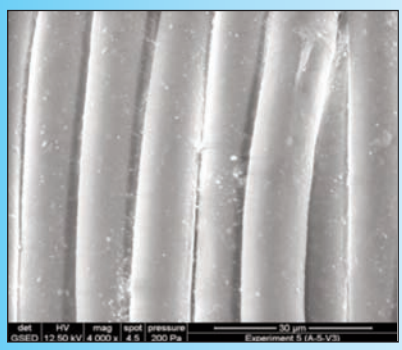
D5-V1

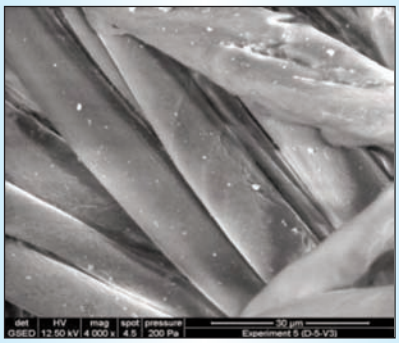
G5-V1

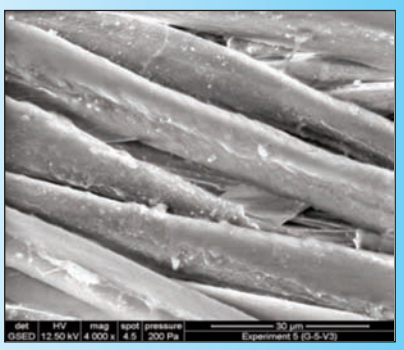

A4-V2

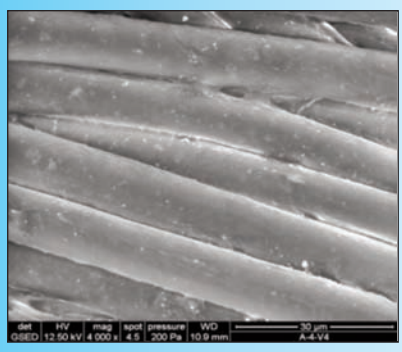

D4-V2

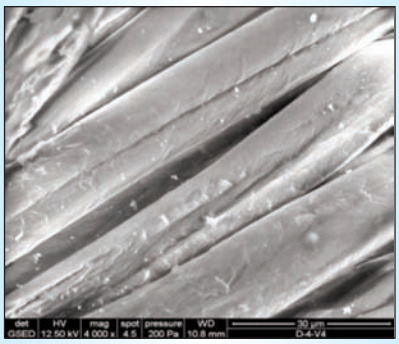

G4-V2


A5-V3


D5-V3


G5-V3


A4-V4


D4-V4


G4-V4

cotton/polyester fabric, followed by polyester and cotton. On 50/50% cotton/polyester materials treated by padding (V1, V2) a greater amount of photocatalysts, regardless of their composition, is deposited.

Due to the small particles size (less than 100nm), the amount of TiO_2 deposited on polyester and polyester/cotton is higher than that of TiO_2-Fe. Apparently, the material structure has a decisive influence on the amounts of deposited titanium dioxide. Thus, on cotton/polyester (G5-V1) fabric three times more TiO_2 is deposited than on polyester fabric (A5-V1) and five times more than on cotton fabric (D5-V1). This massive deposition is due both to the polyester fibers (less negatively charged) and to the nonplanar structure of the material, composed of yarns with different fineness

Table 2

EDX SPECTRUM OF THE MATERIALS COATED WITH TiO_2 AND TiO_2-Fe BY PADDING

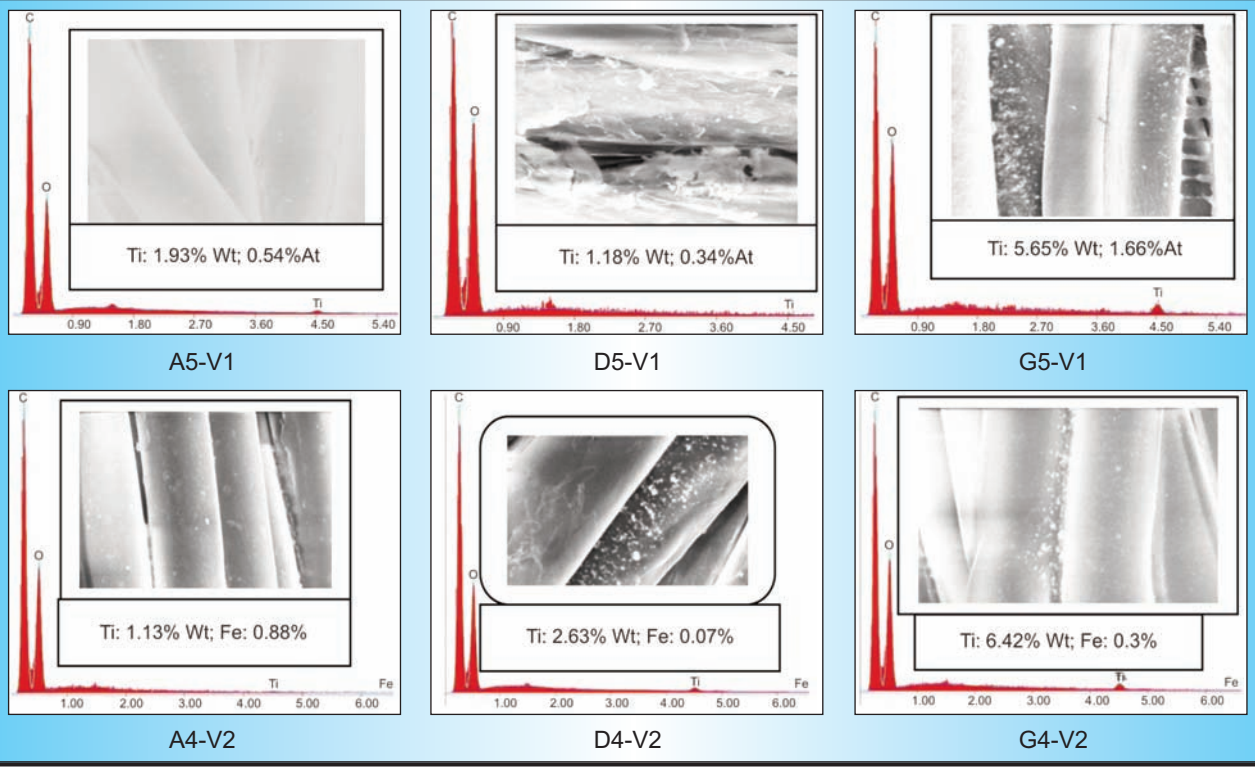


Table 3

EDX ANALYSIS OF THE MATERIALS TREATED WITH SOLUTIONS 4 OR 5 BY PADDING (V1, V2) AND CATIONIZATION-PADDING (V3, V4)

	TiO_2		TiO_2- Fe		TiO_2		TiO_2- Fe		TiO_2		TiO_2- Fe	
Element, %Wt	A5-V1	**A5-V3**	A4-V2	**A4-V4**	D5-V1	**D5-V3**	D4-V2	**D4-V4**	G5-V1	**G5-V3**	G4-V2	**G4-V4**
TiK	**1.93**	**8.10**	**1.13**	**2.55**	**1.18**	**3.30**	**2.63**	**1.40**	**5.65**	**7.54**	**4.07**	**2.81**
FeK	0	0	**0.88**	**0.81**	0	0	**0.07**	**0.88**	0	0	**0.30**	1.16

creating trenches favourable to physical deposition of particles.

The evaluation of the photocatalytic efficiency of the treated materials under visible light

The effect of the visible light on the materials treated with photocatalysts is shown in the tables 4 and 5.

As demonstrated by the dL* and dE* values, the highest discoloration at visible light is produced by the treatment with TiO_2-Fe without pre-cationization on polyester (A4 V2) and cotton/polyester (G4 V2). In the case of cotton treated with TiO_2-Fe (D4 V4), the cationization pre-treatment brings a small enhancement of discoloration degree, the dL* values being in this case bigger than the one obtained for the process without cationization (D4 V2). The samples treated with TiO_2 have a lower grade of discoloration, presenting smaller values of dL* and dE* compared to those treated with TiO_2-Fe. Nevertheless, the best results from discoloration point of view are obtained on polyester (A5 V1) followed by cotton/polyester (G5 V1) treated by padding without pre-cationization. Also, the color is more shifted to long wavelength for the materials coated with TiO_2-Fe than for TiO_2.

The evaluation of the photocatalytic efficiency of the treated materials under UV light

The effect of the UV light on the materials treated with photocatalysts is shown in the tables 6 and 7.

In the case of the materials treated with TiO_2 by padding without pre-cationization (V1) and exposed to UV light, the most intense discoloration (the highest dL* and dE* values and the smallest note on gray scale) is observed for 100% cotton fabric (D5-V1), followed by 100% polyester fabric (A5-V1) and that of 50/50% polyester/cotton (G5-V1). The cationization pre-treatment (V3) has no positive effect on MB photodegradation, the difference in lightness values being smaller (very close to one) in comparison with the variants treated without cationization. There cannot

Table 4

TREATED MATERIALS STAINED WITH 2x10^{-5}mol/L MB EXPOSED TO VISIBLE LIGHT 4 HOURS

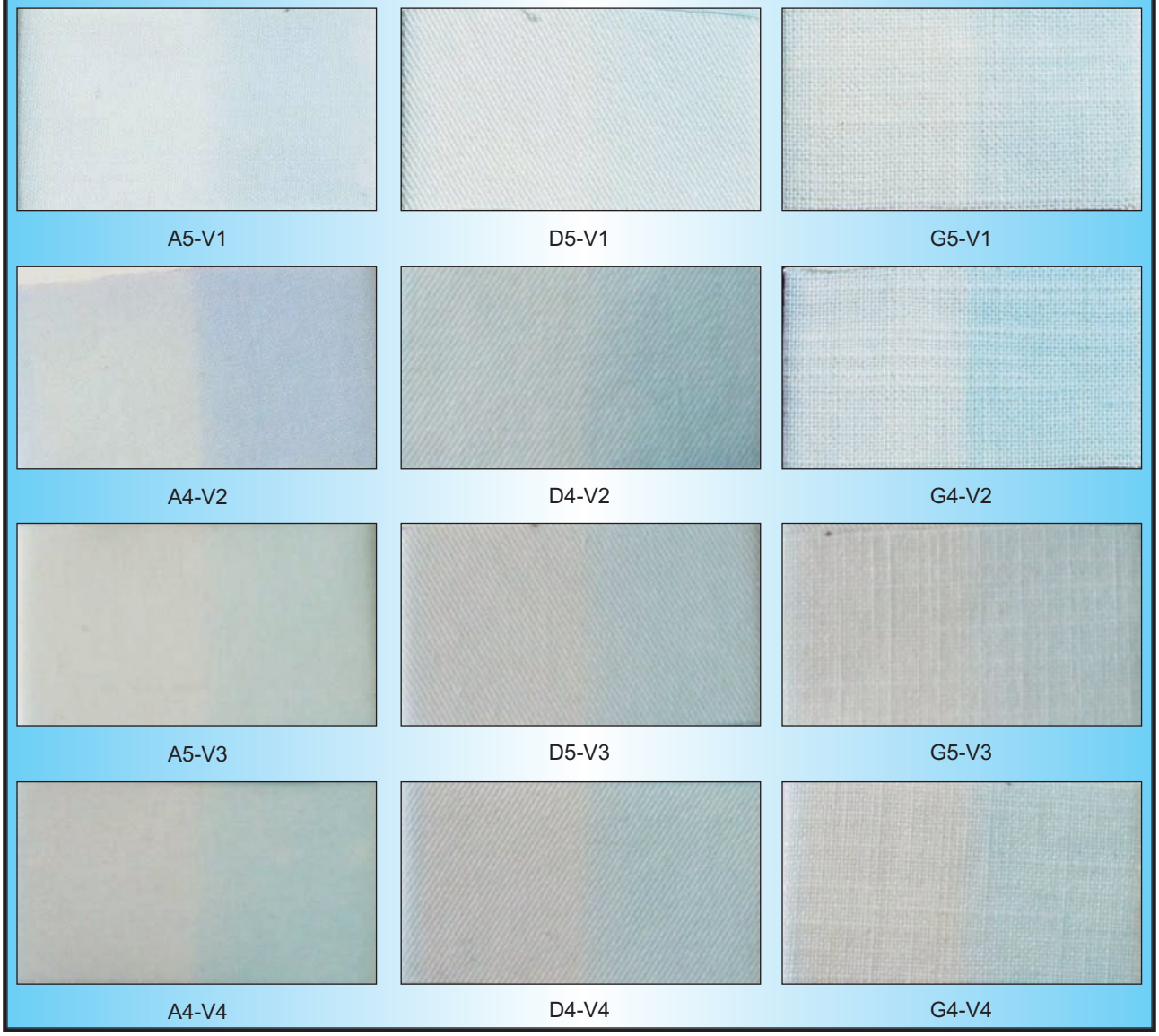


Table 5

CHROMATICITY COODINATES OF THE TREATED MATERIALS BY PADDING (V1, V2) AND CATIONIZATION-PADDING (V3, V4) STAINED WITH 2x10^{-5}mol/L MB EXPOSED TO VISIBLE LIGHT 4 HOURS

	L*	a*	b*	dL*	da*	db*	dE*	dC*	dH*	Note
A5 V1	92.29	–3.84	–1.14	5.37	4.99	7.47	10.47	–8.33	–3.37	1.5
A4 V2	91.85	–4.09	–0.96	8.9	8.27	13.71	18.32	–15.03	–5.86	**1**
A5 V3	96.26	–0.9	3.05	2.61	6.24	3.09	7.44	–3.96	–5.73	2
A4 V4	88.61	–3.77	–0.87	5.92	10.21	8.5	14.55	–12.97	–2.91	**1**
D5 V1	91.11	–2.22	–0.16	2.55	4.95	3.77	**6.72**	**–5.95**	–1.82	2
D4 V2	88.08	–3.21	–0.6	2.21	6.41	4.44	**8.11**	**–8.52**	–1.53	**2**
D5 V3	89.58	–3.1	–1.34	2.51	7.42	4.2	8.89	–8.51	–0.48	1.5
D4 V4	88.5	–2.54	–0.12	3.07	8.29	5.43	10.38	–9.63	–2.36	**1.5**
G5 V1	91.87	–2.62	0.6	3.29	6.09	5.31	8.72	–7.21	–3.65	1.5
G4 V2	88.67	–4.13	–1.03	6.86	10.53	9.58	15.81	–13.1	–2.31	**1**
G5 V3	90.47	–2.95	–0.51	2.69	5.86	4.09	7.64	–3.31	–1.69	2
G4 V4	88.85	–2.56	0.52	2.88	8.51	5.19	10.38	–9.4	–3.31	**1.5**

Table 6

EDX SPECTRUM OF THE MATERIALS COATED WITH TiO_2 AND TiO_2-Fe BY PADDING

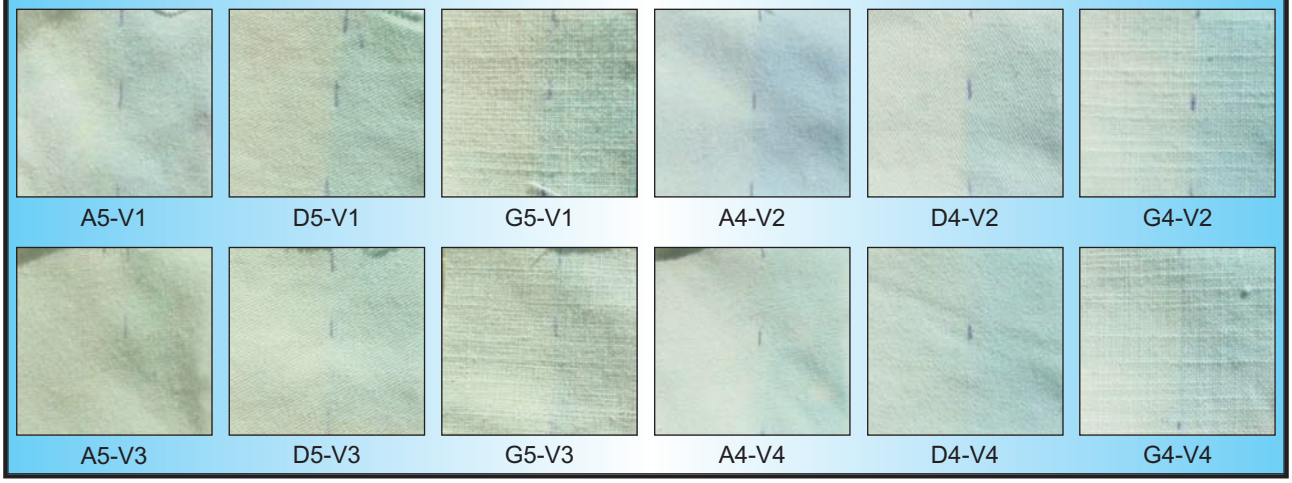


Table 7

CHROMATICITY COORDINATES OF THE TREATED MATERIALS BY PADDING (V1, V2) AND CATIONIZATION-PADDING (V3, V4) STAINED WITH $2x10^{-5}$ mol/L) MB EXPOSED 23 HOURS TO UV (254 nm) LIGHT

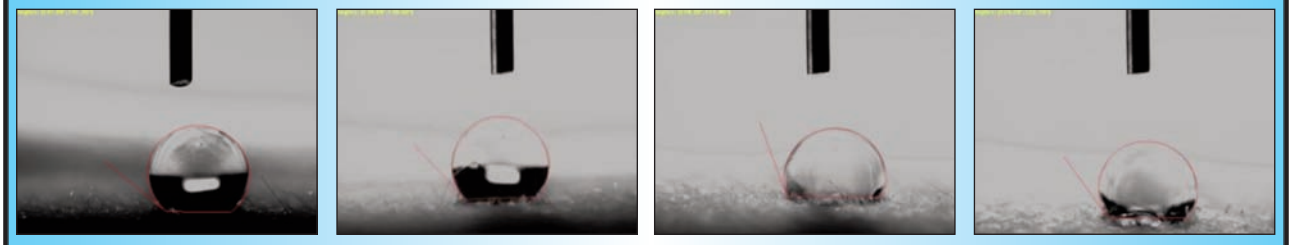
	L*	a*	b*	dL*	da*	db*	dE*	dC*	dH*	Note
A5 V1	85.52	–6.81	–7.06	1.8	2.41	5.86	6.59	–6.06	–1.84	2
A4 V2	86.45	–4.28	–6.20	**0.74**	**1.99**	**-0.20**	**2.13**	–1.14	–1.64	3.5
A5 V3	93.01	–1.71	0.38	0.74	1.67	2.28	2.92	–2.12	–1.86	3.5
A4 V4	92.21	–1.79	1.02	1.41	5.15	3.04	6.14	–5.17	–3.01	2
D5 V1	88.94	–4.02	–0.64	3.15	6.71	5.89	**9.47**	–8.49	–2.76	**1.5**
D4 V2	90.43	–1.91	1.63	**4.49**	**5.03**	**6.17**	**9.14**	–5.78	–5.48	**1.5**
D5 V3	87.15	–6.31	–2.28	1.75	3.73	3.97	5.72	3.42	3.88	2.5
D4 V4	88.25	–4.32	0.37	2.97	4.92	5.74	8	–6.18	–4.13	2
G5 V1	86.52	–7.71	–3.63	0.79	2.8	3.39	4.47	–1.42	2.58	2.5
G4 V2	86.65	–4.12	–6.03	**2.38**	**6.18**	**1.55**	**6.80**	–5.48	3.24	**2**
G5 V3	86.54	–5.67	–3.12	0.89	1.09	2.13	2.25	1	2.08	3.5
G4 V4	85.95	–7.4	–2.33	2.57	5.25	4.59	7.43	–2.11	0.17	1

be made a correlation between the amount of TiO_2 founded on the materials and the degree of discoloration. Even if the amount of TiO_2 found on the materials are bigger in the case of cationization pretreatment, the obtained discoloration degree is smaller. In the case of the materials treated with TiO_2-Fe by padding without pre-cationization (V2) and exposed to UV light, according to difference in lightness (dL*) and total color difference (dE*), the most intense discoloration is obtained on cotton fabric (D4 V2), being followed by blended cotton/polyester (G4 V2), and, finally by the polyester fabric (A4 V2), who presents the lowest degree of discoloration. The cationization-padding treatment with TiO_2-Fe (V4) does not lead to improved discoloration degree, the dL* values being similar or smaller than those obtained in the case of padding treatment without cationization. Under the UV light, a better discoloration degree is noticed on 100% cotton and 50/50% polyester/cotton treated by padding with TiO_2-Fe than with TiO_2. In the case of 100% polyester the discoloration has low level being similar for both formulations. Overall, the degree of discoloration of the polyester and polyester/cotton fabrics stained with MB is higher under visible light as compared to the exposure to UV light. In the case of cotton, however, it can be seen a slightly higher discoloration when using the UV light spectrum.

From the point of view of efficiency of the tested fotocatalysts, TiO_2-Fe applied by pad-dry-cure without cationization, provides the most advanced photodegradation of MB dye under the visible light, especially when is applied on 100% polyester fabrics. Even under UV light, TiO_2-Fe based photocatalyst induces an intense discoloration though it is deposited in much smaller amounts on the materials than TiO_2. The better efficient photocatalytic effects of TiO_2-Fe than TiO_2 are determined by a more efficient separation of photogenerated electrons and holes, Fe^{3+} acting as collector of holes and/or electrons [14]:

Table 8

CONTACT ANGLES IMAGE ON THE MATERIALS TREATED WITH TiO_2-Fe (V4)



100% polyester (A) initial: 136.20; 136.90	A4-V4: 123.6; 122.88	D4-V4: 105.82; 106.2	G4-V4: 111.28; 110.42

$$Fe(III) + h^+_{VB} \rightarrow Fe(IV) \quad (1)$$

$$Fe(IV) + OH^- \rightarrow Fe(III) + {}^{\bullet}OH \quad (2)$$

$$Fe(III) + e^-_{CB} \rightarrow Fe(II) \quad (3)$$

$$Fe(II) + Ti(IV) \rightarrow Fe(III) + Ti(III) \quad (4)$$

Fe^{2+} ions being relatively unstable due to loss of energy by transition from d5 (half filled with electrons) to d6 orbital, tend to return to the valence 3+ (d5) by releasing the trapped electron. Since the energy level of Fe (II)/Fe (III) is very close to that of Ti (III) / Ti (IV) level, the electrons trapped by the Fe (II) can be easily transferred to the neighbouring surficial Ti (IV) [15], where they can combine with oxygen molecules generating reactive species, O°_2 and $^{\circ}OH$:

$$Fe^{2+} + O_2 \rightarrow Fe^{3+} + O^{\circ-}_2 \quad (5)$$

Consequently, due to promotion of the electrons and holes separation under the influence of light, Fe (III) improves the photocatalytic efficiency of TiO_2.

In addition, in such photocatalytic systems, may generate H_2O_2 on the surface of TiO_2 [16]. Simultaneous presence of Fe^{2+} and H_2O_2 in an acidic environment can produce $^{\circ}OH$ which act as oxidizing agents:

$$Fe^{2+} + H_2O_2 + H^+ \rightarrow Fe^{3+} + {}^{\circ}OH + OH^- \quad (6)$$

The effect of the photocatalytic treatment on the materials hydrophilic properties

The initial cotton and cotton/polyester fabrics are hydrophilic, while polyester fabric is hydrophobic. After padding, all the materials become hydrophilic.

By cationization-padding, the materials treated with TiO_2-Fe (V4) become hydrophobic (table 8).

In the variant V4, it is found that the value of the contact angle decreases for 100% polyester fabric from (138.2, 138.93) to (123.6, 122.88) showing a slight hydrophilization of the material. Instead, the material of cotton and polyester/cotton become more hydrophobic because cotton being negatively charged has a higher affinity to quaternary compounds than polyester. Consequently, on the cotton surface a larger amount of polyethylene polyamine resin is deposited whose hydrophobic alkyl chains are oriented toward the exterior of the fabric. What must be emphasized is that the same cationized materials under the same conditions but using TiO_2 instead of iron doped TiO_2 (V3) do not change their hydrophilic nature, contrary the polyester fabric becomes more hydrophilic after treatment with TiO_2. A hypothesis on this behaviour would be the lower acidity (pH = 5) of the TiO_2-Fe solution due to which secondary and tertiary amino groups of polyamine resin used are protonated and attract more hydrophilic carboxyl group of used polyacrylic binder. Consequently, due to electrostatic repulsion between the carboxyl groups of the acrylic binder coating the cotton surface and those found on the surface of TiO_2, the photocatalyst amount deposited on the material is lower. In addition, free quaternary groups involved in ionic bonds with the hydroxyl groups of cellulose can form ionic bonds with acrylic polymer (Itobinder AG), increasing the hydrophobicity of cotton fabric.

Antifungal activity of the materials treated with TiO_2-Fe

The treated textile materials and controls were tested in duplicate against 2 pathogenic strains, *Candida albicans and Epidermophyton floccosum* by using modified **ISO 20743:2007** standard, absorption method, an evaluation method where the inoculum is inoculated directly onto the samples. Colony plate count method was used after 24 h incubation for quantification of Colony Forming Units (UFC), and percentage and logarithmic reduction rates were calculated against control sample for each material.

When tested against *Candida a.*, all samples yielded very good reduction rates, with reduction rates of over 90%, with maximum percent of 100% for samples A4-V2, D4-V2 and G4-V2. Highest inoculum concentration was of 24400 CFU/mL for control sample C (Aster).

When tested against *Epidermophyton f.,* the reduction rates of tested materials varied between 46.87% (D4-V2) and highest reduction rate of 90.58% for A4-V2. Highest inoculum concentration was of 15360 CFU/mL for control sample A (Dockers). It is important to notice that, due to the irregularity of textile materials surface, an uniform and homogeneous dispersion of photocatalytic particles on theirs surface is practically not possible to be achieved. More than that, the tested samples were taken randomly, so the antibacterial activity is difficult to be related to

Table 9

ANTIMICROBIAL ACTIVITY AGAINST *CANDIDA ALBICANS*

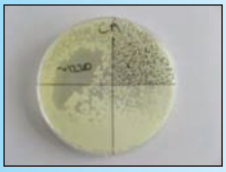
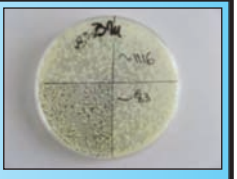
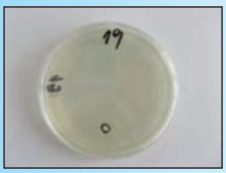
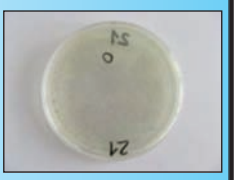
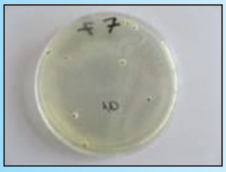
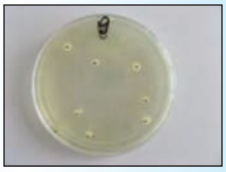
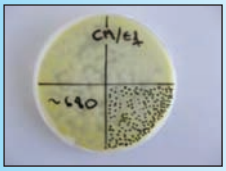
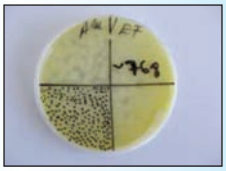
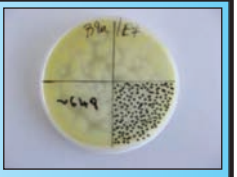
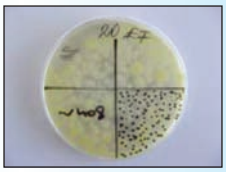
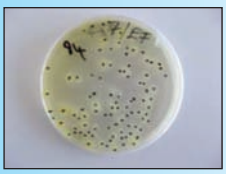
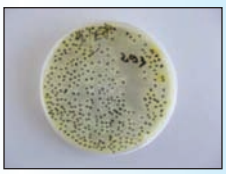
Sample/Results	Picture	Sample/Results	Picture	Sample/Results	Picture
(**CM**) ASTER C_i=2.440x10^4 UFC/mL Control		(**AM**) DOCKERS C_i= 2.312x10^4 UFC/mL Control		(**BM**) GOLF C_i=2.232x10^4 UFC/mL Control	
(**19**) A4-V2 C_i=2.440x10^4 CFU/mL C_{24h}=0 CFU/mL R%=100 Log_{10} red.=4.38		(**20**) D4-V2 C_i=2.312x10^4 CFU/mL C_{24h}=0 CFU/mL R%=100 Log_{10} red.=4.36		(**21**) G4-V2 C_i=2.232x10^4 CFU/mL C_{24h}=0 CFU/mL R%=100 Log_{10} red.=4.34	
(**7**) A4- V4 C_i=2.440x10^4 CFU/mL C_{24h}=2x10^2 CFU/mL R%=99.18		(**9**) D4-V4 C_i=2.312x10^4 CFU/mL C_{24h}=2x10^2 CFU/mL R%=99.13		(**8**) G4-V4 C_i=2.232x10^4 CFU/mL C_{24h}=6x10^1 CFU/mL R%=99.73	

Table 10

ANTIMICROBIAL ACTIVITY AGAINST *EPIDERMOPHYTON FLOCCOSUM*

Sample/Results	Picture	Sample/Results	Picture	Sample/Results	Picture
(**CM**) ASTER C_i=1.36x10^4 UFC/mL		(**AM**) DOCKERS C_i=1.536x10^4 UFC/mL		(**BM**) GOLF C_i=1.296x10^4 UFC/mL	
(**19**) A4-V2 C_i=1.36x10^4 CFU/mL C_{24h}=1.28x10^3 CFU/mL **R%=90.58** Log_{10} red.=1.03		(**20**) D4-V2 C_i=1.536x10^4 CFU/mL C_{24h}=8.16x10^3 CFU/mL **R%=46.87** Log_{10} red.=0.27		(**21**) G4-V2 C_i=1.296x10^4 CFU/mL C_{24h}=4.8x10^3 CFU/mL **R%=62.96** Log_{10} red.=0.43	
(**7**) A4- V4 C_i=1.36x10^4 CFU/mL C_{24h}=1.68x10^3 CFU/mL **R%=87.64** Log_{10} red.=0.91		(**9**) D4-V4 C_i=1.536x10^4 CFU/mL C_{24h}=5.86x10^3 CFU/mL **R%=61.84** Log_{10} red.=0.42		(**8**) G4-V4 C_i=1.296x10^4 CFU/mL C_{24h}=2.26x10^3 CFU/mL **R%=82.56** Log_{10} red.=0.76	

the content of TiO_2-Fe particles. As for example, even A4-V2 sample contains 1.13% TiO_2-Fe, the antibacterial reduction rate for *Epidermophyton floccosum* is higher than that of A4-V4 fabric which contains 2.55% TiO_2-Fe.

CONCLUSIONS

As results demonstrate, the largest amount of **TiO_2** is deposited on **cationised** materials, regardless the fabric composition while the largest amount of **TiO_2-Fe** is deposited by **pad-dry-cure** method except polyester fabric. By both methods, the greatest amount of photocatalyst is found on **pes/cotton** fabric.

The **highest discoloration under visible light** is produced by treatment with **TiO_2-Fe**, by both methods, especially on polyester and cotton/polyester. The **discoloration is stronger** on materials treated

by **pad-dry-cure** than by cationization-padding. Under the **UV light**, the most intense discoloration is noticed on the materials treated by **cationisation-padding** with **TiO_2-Fe** than with TiO_2.

All samples yielded very good reduction rates of *Candida a.*, with 100% reduction for samples treated by pad-dry-cure method. The reduction rates of *Epidermophyton f.* varies between 46.87% (D-V1-Fe) and to 99.18 % for A-V2-Fe, the best antibacterial activity being for the cationised materials.

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Elastic hybrid yarns for denim fabrics

HÜSEYİN GAZİ TÜRKSOY SÜMEYYE ÜSTÜNTAĞ

REZUMAT – ABSTRACT

Fire hibride elastice pentru materiale tip Denim

Îmbrăcămintea de tip Denim este purtată fără a ține cont de diferențele demografice, cum ar fi vârsta, sexul și statutul social. Domeniul de aplicare al îmbrăcămintei denim este tot mai larg de la an la an, iar producția și consumul acesteia au crescut imprevizibil în ultimele decenii. În scopul obținerii unor caracteristici de performanță diferite a țesăturilor tip denim, adesea sunt folosite diverse fire hibride. În acest studiu, au fost fabricate noi fire hibride elastice cu diferite metode de procesare și niveluri de răsucire. Ulterior, diferitele proprietăți fizice, cum ar fi neregularitatea, pilozitatea și tenacitatea, au fost evaluate comparativ. Suplimentar, au fost de asemenea comparate performanțele firelor hibride obținute, cum ar fi rezistența la tracțiune, alungire, proprietăți elastice, atunci când acestea au fost folosite pentru realizarea unor țesături tip denim. Concluziile studiului au arătat că metoda de procesare și nivelul de răsucire a firului hibrid au o influență semnificativă din punct de vedere statistic asupra pilozității, alungirii la rupere a firelor hibride și a proprietăților elastice ale țesăturilor denim în care firele hibride au fost folosite ca fire de bătătură.

Cuvinte-cheie: fire hibrid, denim, fire commingling, tenacitate, întindere maximă, întindere permanentă

Elastic hybrid yarns for denim fabrics

Denim wears are being worn irrespective of demographic differences such as age, gender and social status. The scope for denim wear is increasing tremendously every year; furthermore its production and consumption has increased unpredictably in the last few decades. In order to give different performance characteristics to denim wears, various hybrid yarns are often used in denim fabric production. In this study, new elastic hybrid yarns were manufactured with different production methods and twist levels. Then the various physical properties such as unevenness, hairiness and tenacity were evaluated comparatively. In addition, the performances of developed hybrid yarns in denim fabric form, such as tensile strength, elongation, stretch properties, were also compared. The findings of study revealed that the production method and twist level of the hybrid yarn have statistically significant influence on hairiness, breaking elongation values of the hybrid yarns and stretch properties of denim fabrics in which the hybrid yarns were used as weft yarn.

Keywords: hybrid yarn, denim, commingling yarn, tenacity, maximum stretching, permanent stretching

Denim is perhaps the most used fabric types in garment manufacturing. Through modern technology, denims have been improved in quality and provide a satisfactory level of comfort and durability to consumers [1]. Denim fabric is a laborious product for its design and production phase. Conventionally, denim fabric made from 100% cotton with indigo-dyed warp yarn and undyed weft yarn. Depending on growth and change in demand for denim products, denim fabric manufacturers constantly try to develop new kinds of denim fabrics to meet consumer demands [1, 2]. In order to give different performance characteristics to denim fabric, manufacturing companies use various hybrid yarns in their denim fabric production.

Hybrid yarn is a yarn structure developed to benefit from the properties of two or more different components at the same time. Although hybrid yarn production methods can be divided into three main groups, namely core-spun, covering and twisting methods [3–20], core-spun hybrid yarns are commonly used for the production of denim fabric. Hard core filament (polyester-PET or poliamid-PA), soft core filament (elastane) and commingling yarns (PET or PA + Elastane) are used materials in the core part of core-spun yarns used in denim fabrics. Core-spun yarns containing PET or PA filament are often preferred for high strength in denim fabric production while core-spun yarns containing elastane are used to give elastic characteristics to denim fabrics. The commingling elastic filaments are also used in core part of core-spun yarn to overcome a number of problems resulting from the high recall property of elastane.

Production techniques and containing material are two important factors which affect the properties and performances of hybrid yarns. This study is aimed to develop new hybrid yarns containing commingling elastic filaments for denim fabrics. For this purpose, firstly elastic hybrid yarns were produced with innovative production techniques. Then, physical properties and performances of these hybrid yarns in denim fabric form were comparatively evaluated.

MATERIALS AND METHODS

Ne 8/1 and Ne 16/1 ring-spun 100 % cotton yarns and commingling elastic yarns were used for production of hybrid yarns. The commingling yarn was combined with 78 dtex elastane and 78/24 dtex poliamid using air covering process. Microscopic view of commingling yarn at the magnification of 20× is shown in figure 1.

The hybrid yarns were produced with 4 different methods, namely classical twisting with single yarn (TS), classical twisting with double yarn (TD), covering with twisted double yarn (CTD), covering with

Table 1

THE CODES AND PRODUCTION PARAMETERS OF HYBRID YARNS			
Code	**Production Method**	**Content**	**Tpm**
TS/250	Classical Twisting with Single Yarn	Ne 8 Co / C	250
TS/350	Classical Twisting with Single Yarn	Ne 8 Co / C	350
TS/450	Classical Twisting with Single Yarn	Ne 8 Co / C	450
TD/250	Classical Twisting with Double Yarn	Ne 16 x 2 Co / C	250
TD/350	Classical Twisting with Double Yarn	Ne 16 x 2 Co / C	350
TD/450	Classical Twisting with Double Yarn	Ne 16 x 2 Co / C	450
CTD/250	Covering with Twisted Double Yarn	Ne 16 x 2 Co / C	250
CTD/350	Covering with Twisted Double Yarn	Ne 16 x 2 Co / C	350
CTD/450	Covering with Twisted Double Yarn	Ne 16 x 2 Co / C	450
CND/250	Covering with Non-twisted Double Yarn	Ne 16 x 2 Co / C	250
CND/350	Covering with Non-twisted Double Yarn	Ne 16 x 2 Co / C	350
CND/450	Covering with Non-twisted Double Yarn	Ne 16 x 2 Co / C	450

Co: Cotton yarn, C: Commingling yarn

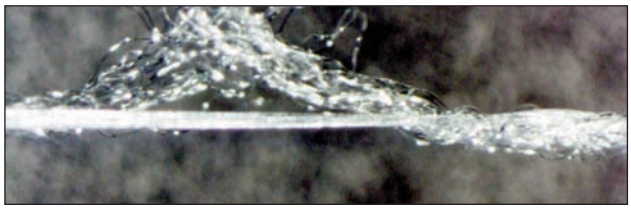

Fig. 1. Microscopic view of commingling yarn at the magnification of 20×

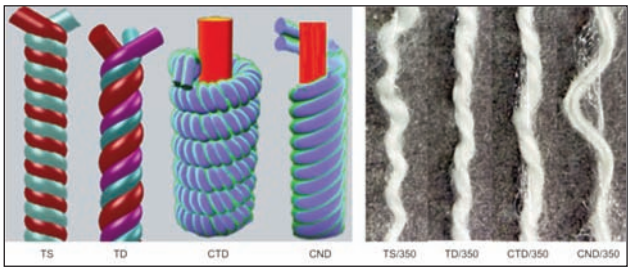

Fig. 2. Models of production methods and longitudinal views of hybrid yarns

non-twisted double yarn (CND) and with 3 different twist levels, namely 250 tpm, 350 tpm ve 450 tpm. Ağteks ‘‘Directwist-2B’’ machine was used for the production of hybrid yarns. Codes and production parameters of the hybrid yarn samples are summarized in table 1. The models and the photographs of the hybrid yarns produced with 4 different production methods at the twist level of 350 tpm are given in figure 2.

The unevenness and hairiness values of hybrid yarns were measured on Uster Tester 5. In addition, tenacity and elongation values of hybrid yarns were determined using Uster Tensojet instrument. The hybrid yarns were photographed with an Olympus SZ61 stereo microscope using Babsoft® digital image processing software.

For evaluating the performance of developed hybrid yarn in denim fabric form, 12 different denim fabric samples were produced with 3/1 Z twill structure using Picanol GT-Max Rapier weaving machine. The produced hybrid yarns were used as weft yarn with a density of 13 picks/cm while Ne 14/1 cotton yarns were used as warp yarn with a density of 32 ends/cm for all fabric samples.

In order to investigate how the production methods and twist levels of hybrid yarns changing the denim fabric properties; tensile strength, breaking elongation, maximum stretching, permanent stretching and dimensional change of denim fabric samples were analyzed. All tests were carried out after the fabric samples were conditioned in standard atmospheric conditions (temperature 20±2 °C and relative humidity 65±2%).

The tensile properties of denim fabrics in the weft direction were performed on an Instron 4411 tester according to TS EN ISO 13934-1 standard [21]. Five samples for each fabric were tested, and averages of the test results were calculated.

The stretching and dimensional change properties of denim fabrics were tested according to relevant standards [22, 23]. For each fabric sample, three tests were performed and the averages were reported. Before the stretching and dimensional change tests, all the fabric samples were washed at 85 °C for 60 minute three times.

A stretch testing instrument, consisting of a frame with separate clamps fixed at the top and at the bottom, was implemented to determine the stretch properties of the fabrics. Sample strips from weft direction were hung on the apparatus after marking a 250 mm distance in the central part of each specimen. A 1360 g load, which was hung according to the fabric weight in the bottom hanger, was applied to the sample three times with 5 second intervals and after the fourth

application the samples were released for hanging 30 minutes. After 30 minutes, the marked distance was measured once again. For permanent stretching, the marked distance after 1 hour relaxation time was also measured again. Maximum and permanent stretching values were calculated from these measured outcomes. For maximum and permanent stretching values, the formulas (1, 2) were used.

$$\text{Maximum stretching in \%} = (B - A)/A \times 100 \quad (1)$$

$$\text{Permanent stretching in \%} = (C - A)/A \times 100 \quad (2)$$

A: The distance marked between the upper and bottom parts of the fabric (250 mm).
B: The distance between the marked points after hanging the sample for 30 minutes with the load (mm).
C: The distance between the marked points after 1 hour of relaxation.

Fabric sample were marked from weft direction with a 50 cm index in 3 different central part of each specimen using ruler for dimensional change test. After washing, the dried fabrics were conditioned in standard atmospheric conditions for 4 hour duration. Dimensional change values (%) of the fabric samples were determined by using sanfor ruler from weft direction [23].

All of the outcomes related to the physical properties of hybrid yarns and denim fabrics were evaluated statistically for significance in differences using two-way replicated analysis of variance (ANOVA) and the mean differences subgroups were also compared by the post hoc Duncan test at 95 % significance level with use of SPSS statistical package. In this way, the effects of factors (production method and twist level) on the physical properties and performances of hybrid yarns were analyzed.

RESULTS AND DISCUSSIONS

In this study, 12 different types of elastic hybrid yarns were manufactured with 4 different production methods and 3 different twist levels. The various physical properties of these hybrid yarns were evaluated comparatively. In addition, the performances of these hybrid yarns in denim fabric form were also compared.

Assessment of hybrid yarn properties

The average values of yarn count, unevenness, hairiness, tenacity and breaking elongation of elastic hybrid yarns produced in the scope of this study are given in table 2.

ANOVA test results of the hybrid yarns for all tested properties are shown in table 3. The mean differences of subgroups were compared and interpreted for only statistically significant factors by using Duncan post hoc test. Duncan test results of the hybrid yarns for hairiness, tenacity and breaking elongation values are given in table 4, 5 and 6 respectively.

Based on ANOVA results, it is clear that the unevenness of hybrid yarns doesn't depend on production method and twist level. On the other hand, the intersection of production method and twist level is found

Table 2

THE CODES AND PHYSICAL PROPERTIES OF HYBRID YARNS

Yarn code	Yarn count (tex)	CVm (%)	Hairiness (H)	Tenacity (cN/Tex)	Breaking elongation (%)
TS/250	88.72	10.51	10.38	12.32	7.54
TS/350	84.99	10.11	9.67	12.12	8.03
TS/450	84.38	10.91	8.83	13.85	9.83
TD/250	91.49	10.44	8.92	17.38	6.50
TD/350	89.68	10.10	8.13	16.26	7.18
TD/450	87.54	10.91	7.89	15.43	8.33
CTD/250	92.62	11.00	9.19	17.14	7.29
CTD/350	93.41	10.95	8.28	15.82	7.06
CTD/450	94.93	9.78	8.10	16.07	7.12
CND/250	83.67	11.16	8.96	15.50	5.49
CND/350	89.54	10.85	9.06	13.96	4.99
CND/450	95.72	10.73	9.11	12.95	5.64

Table 3

ANOVA TEST RESULTS FOR YARN PROPERTIES

		Production method	Twist level	Intersection of the factors
CVm (%)	F	1.453	0.982	3.151
	Significance	0.252	0.389	0.020
Hairiness (H)	F	55.332	43.121	7.877
	Significance	0.000	0.000	0.000
Tenacity (cN/Tex)	F	20.820	3.142	1.922
	Significance	0.000	0.061	0.118
Breaking elongation (%)	F	36.825	9.475	2.917
	Significance	0.000	0.001	0.028

to be statistically significant in terms of unevenness values.

According to ANOVA results, it was found that all factors (production method and twist level) and intersection of these factors are statistically significant in terms of hairiness values of the hybrid yarns. According to Duncan test results, it was found that the hybrid yarns produced by TS method show significantly higher hairiness values than other production methods. This relatively higher hairiness values is attributed to the fact that hybrid yarns produced by TS method have two components, Ne 8 cotton yarn and commingling yarn, unlikely for the rest of the production methods. In addition, the differences between hairiness values of hybrid yarns are found to be statistically significant for all twist levels according to Duncan results. It is well known that yarn twist reduces hairiness value of yarn. The hairiness values of hybrid yarns produced by TS, TD and CTD methods decrease by increasing twist level. This situation can also be seen in table 2. Table 2 reveals that the highest hairiness value is obtained from the hybrid yarn produced by TS method and twist level of 250 tpm.

The production method has a significant effect on the tenacity values of hybrid yarns as seen in table 3. However, the twist level and intersection of the factors are statistically insignificant in terms of tenacity value.

Duncan test results show that there isn't a significant difference between tenacity values of hybrid yarns produced by TS and CND methods. The same situation is also valid for the hybrid yarns produced by TD and CTD methods. As shown in table 5, the tenacity values of hybrid yarns produced by TS and CND methods are lower than that of other production methods. This result can be related with the components and the placement of components in hybrid yarns' structure. In TS production method, Ne 8 cotton yarn and commingling yarn were twisted. In addition, in CND production method, two Ne 16 cotton yarn just covered commingling yarn as seen in figure 2. On the other hand, Ne 16 yarns are used as twisted in TD and CTD methods.

According to ANOVA results, it was found that all factors and intersection of these factors have significant influence on the breaking elongation values. According to Duncan test results, there are significant difference between all but TD and CTD methods in terms of the breaking elongation value. As can be seen in table 2 and 6, the breaking elongation values of the hybrid yarns produced by TS method is higher than that of the other methods. Also, TS/450 hybrid yarns produced with TS method at the twist level of 450 tpm show highest breaking elongation values.

Assessment of hybrid yarn performances in denim fabric form

On the grounds that elastic hybrid yarns produced in the scope of this study were used in the weft direction of denim fabrics, assessments of denim fabric samples were carried out only in the weft direction. The average values of tensile strength, breaking elongation, maximum strength, permanent stretching and dimensional change of denim fabric samples are given in table 7.

ANOVA test results for all measured properties of denim fabric samples are shown in table 8. Duncan

Table 4

DUNCAN TEST RESULTS OF HYBRID YARNS FOR HAIRINESS VALUE

Process	N	Subset		
Production Method		1	2	3
TD	9	8.3133		
CTD	9	8.5189		
CND	9		9.0422	
TS	9			9.6256
Twist Level		1	2	3
450	12	8.4792		
350	12		8.7833	
250	12			9.3625

Table 5

DUNCAN TEST RESULTS OF HYBRID YARNS FOR TENACITY VALUE

Process	N	Subset	
Production Method		1	2
TS	9	12.7656	
CND	9	14.1344	
CTD	9		16.3411
TD	9		16.3578
Twist Level			
350	12	14.5400	
450	12	14.5742	
250	12	15.5850	

Table 6

DUNCAN TEST RESULTS OF HYBRID YARNS FOR BREAKING ELONGATION VALUE

Process	N	Subset		
Production Method		1	2	3
CND	9	5.3722		
CTD	9		7.1600	
TD	9		7.3367	
TS	9			8.4667
Twist Level		1	2	3
250	12	6.7075		-
350	12	6.8142		-
450	12		7.7300	-

Table 7

THE PHYSICAL PROPERTIES OF DENIM FABRICS					
Yarn/ fabric codes	**Tensile strength (N)**	**Breaking elongation (mm)**	**Maximum stretching (%)**	**Permanent stretching (%)**	**Dimension changes (%)**
TS/250	647.67	25.37	72.2	9.2	–43.6
TS/350	687.90	26.26	71.8	8.8	–42.4
TS/450	709.02	29.36	60.2	7.2	–39.8
TD/250	802.80	20.79	66.2	10.8	–43.0
TD/350	768.76	22.28	58.4	8.8	–40.9
TD/450	763.50	25.53	53.4	7.2	–39.0
CTD/250	564.26	27.48	68.4	10.0	–43.8
CTD/350	717.34	33.74	67.4	10.0	–42.3
CTD/450	681.10	35.68	62.8	8.8	–40.0
CND/250	702.92	15.55	79.4	10.8	–50.5
CND/350	593.06	14.44	72.6	10.0	–46.2
CND/450	603.38	16.54	68.2	8.8	–45.3

Table 8

ANOVA TEST RESULTS OF THE DENIM FABRIC SAMPLES FOR ALL MEASURED PROPERTIES				
		Production method	**Twist level**	**Intersection of the factors**
Tensile strength (N)	F	135.229	1.866	41.371
	Significance	0.000	0.166	0.000
Breaking elongation	F	668.715	89.812	14.008
	Significance	0.000	0.000	0.000
Maximum stretching (%)	F	10126.556	11008.000	624.889
	Significance	0.000	0.000	0.000
Permanent stretching (%)	F	1422721	5353201	326641
	Significance	0.000	0.000	0.000
Dimensional changes (%)	F	1361.710	964.409	25.069
	Significance	0.000	0.000	0.000

test results of the denim fabric for tensile strength, breaking elongation, maximum strength, permanent stretching and dimensional change values are given in table 9, 10, 11, 12 and 13 respectively.

When table 8 is examined, it is seen that the production method and intersection of the production method and twist level factors have significant effect on tensile strength value of denim fabric samples. However, the twist level is statistically insignificant in terms of tensile strength.

According to Duncan test results, there are significant differences between tensile strength values of all denim fabric samples. It was found that the highest tensile strength value was obtained from denim fabric woven with TD hybrid yarns.

The ANOVA results reveal that the production method, twist level and intersection of these factors have a significant effect on the breaking elongation values of denim fabric samples. As seen in table 10, the maximum breaking elongation value was associated with CTD production method. In addition, the maximum breaking elongation of denim fabrics is observed for the hybrid yarns produced by CTD methods and at twist level of 250 tpm. Duncan test also reveals the significant difference between breaking elongation values of denim fabrics regarding twist level. As the twist level of hybrid yarns increases, the breaking elongation value of denim fabric increases in the manner.

The ANOVA results show that the production method, twist level and intersection of these factors have a significant effect on the maximum stretching values of denim fabric samples.

According to Duncan test results, the difference between maximum stretching values of denim fabrics which were woven with the hybrid yarns produced by different production methods and twist levels are statistically significant.

Table 9

DUNCAN TEST RESULTS OF THE DENIM FABRIC SAMPLES FOR TENSILE STRENGTH					
Process	**N**	**Subset**			
Production Method		1	2	3	4
CND	15	633.1200			
CTD	15		654.2333		
TS	15			681.5300	
TD	15				778.3533
Twist Level		1	2	3	4
250	20	679.4125	-	-	-
450	20	689.2500	-	-	-
350	20	691.7650	-	-	-

Table 10

DUNCAN TEST RESULTS OF THE DENIM FABRIC SAMPLES FOR BREAKING ELONGATION					
Process	**N**	**Subset**			
Production Method		1	2	3	4
CND	15	15.5067			
TD	15		22.8653		
TS	15			26.9953	
CTD	15				32.2987
Twist Level		1	2	3	4
250	20	22.2965			-
350	20		24.1785		-
450	20			26.7745	-

Table 11

DUNCAN TEST RESULTS OF THE DENIM FABRIC SAMPLES FOR MAXIMUM STRETCHING					
Process	**N**	**Subset**			
Production Method		1	2	3	4
TD	9	59.3333			
CTD	9		66.2000		
TS	9			68.0667	
CND	9				73.4000
Twist Level		1	2	3	4
450	12	61.1500			-
350	12		67.5500		-
250	12			71.5500	-

Table 12

DUNCAN TEST RESULTS OF THE DENIM FABRIC SAMPLES FOR PERMANENT STRETCHING					
Process	**N**	**Subset**			
Production Method		1	2	3	4
TS	9	8.3989			
TD	9		8.9333		
CTD	9			9.6000	
CND	9				9.8667
Twist Level		1	2	3	4
450	12	8.0000			-
350	12		9.4000		-
250	12			10.1992	-

As seen in table 11, the maximum stretching values of denim fabrics which were woven from the CND hybrid yarns are higher than that of other fabric samples. Also, it can be seen from Duncan test results, maximum stretching value of denim fabrics increases with the decreasing twist level of the hybrid yarns. The inversely effect of twist level on maximum stretching of denim fabrics can be attributed that increasing the twist level prevents movement of commingling yarn in the structure of the hybrid yarns. Moreover, the highest maximum stretching value is obtained from the CND/250 denim fabrics.

From the ANOVA results, all factors and intersection of these factors are statistically significant in terms of permanent stretching values of the denim fabric samples. Duncan test proves that there are significant differences between permanent stretching values of all denim fabrics which were woven with the hybrid yarns produced by different production methods and twist levels. It was found that the best elastic recovery can be obtained, if the hybrid yarns produced by TS methods are used in weft direction of denim fabrics. On the other hand, the highest permanent stretching value is obtained with CND methods.

Table 13

DUNCAN TEST RESULTS OF THE DENIM FABRIC SAMPLES FOR SHRINKAGE				
Process	**N**	**Subset**		
Production Method		1	2	3
CND	9	–47,3333		
CTD	9		–42,0222	
TS	9		–41,9444	
TD	9			–40,9667
Twist Level		1	2	3
250	12	–45,2250		
350	12		–42,9500	
450	12			–41,0250

In other words, the denim fabrics which were woven with the hybrid yarns produced by CND methods are deformed quicker than that of other denim fabrics. As expected, the permanent stretching values of denim fabrics increase with the decreasing twist level of the hybrid yarns.

The term 'dimensional change' can simply be defined as a change in the dimensions of a fabric under conditions of washing, drying, steaming and pressing. This dimensional change may be in a positive (growth) or negative (shrinkage) direction for fabric length, width, and thickness. For a denim fabric, shrinkage relates to the loss of the length and/or width dimensions [24].

According to ANOVA test results, all factors have a significant effect on the shrinkage of denim fabric samples.

From the Duncan test results, the difference between shrinkage values of denim fabrics which were woven with hybrid yarns produced by CTD and TS methods is statistically insignificant whereas the difference between shrinkage values of others is statistically significant. The highest shrinkage value belongs to the denim fabric samples which manufactured by CND hybrid yarns. This is followed by the denim fabrics woven with hybrid yarn produced by CTD, TS and TD methods, respectively.

Also, it is clear that the difference between shrinkage values of denim fabrics woven with hybrid yarns produced by using different twist levels is statistically significant. As in maximum and permanent stretching, shrinkage value of the denim fabrics decreases with increasing twist level. The reason of this is related to prevented movement of commingling yarn in the structure of the hybrid yarns with increasing the twist level.

CONCLUSIONS

Within the scope of this study, 12 different elastic hybrid yarns and denim fabrics by using these hybrid yarns in the weft direction were manufactured. Various physical and performance tests were applied on the hybrid yarns and denim fabrics, and the test results were comparatively evaluated.

According to the test results, the production method is a significant factor for hairiness, tenacity and breaking elongation values of hybrid yarns. Also, the twist level has a significant effect on the hairiness and breaking elongation values of hybrid yarns.

The findings of study revealed that the production method and twist level of the hybrid yarns have statistically significant influence on breaking elongation, stretch properties (maximum and permanent stretching) and shrinkage values of denim fabrics. According to test results that the lowest tensile strength and breaking elongation values are obtained from denim fabrics woven with the CND hybrid yarns, as in tenacity and breaking elongation values of the hybrid yarns. The highest maximum stretching value is obtained from fabrics woven with the hybrid yarns produced by CND and TS methods, respectively. Furthermore, the highest permanent stretching value is obtained from denim fabric samples manufactured by CND hybrid yarns while denim fabrics woven with TS hybrid yarns have the lowest permanent stretching value. As in stretch properties, denim fabrics woven with CND hybrid yarns have the highest shrinkage values. In addition, maximum stretching, permanent stretching and shrinkage values of the denim fabrics have been seen to go through a definite decrease with increasing twist level. This is believed to be stemming from the fact that movement of the commingling yarn in the structure of the hybrid yarns is limited because of twist increasing.

For further studies, it is though that physical properties of elastic hybrid yarns produced within the scope of this study will also be compared with their core-spun counterpart.

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