

Thermal Stability Enhancement of Cotton and Cotton Polyester Blend Fabrics by Hyperbranched Poly Urethane-Urea Treatment

K.M.Haggag¹, A.I.Hashem², F.N.El Shall^{*1}

¹ Textile Research Division, National Research Centre and ² Faculty of Science, Ain Shams University, Cairo, Egypt.

DESPITE the importance of cotton fabrics and its blends in our lives, the lack of their thermal stability is one of the most important points to be studied. The ability of hyperbranched poly urethane-urea (HBPU) to improve the thermal stability of cotton and cotton (CO) polyester (CO/PET) fabrics was investigated. Different concentrations of HBPU were used for treatment of both CO and CO/PET fabrics. No significant effect on tensile strength properties was observed with CO fabrics, while tensile strength of treated CO/PET fabrics was affected by treatment with different concentrations of HBPU. TGA analysis shows enhanced char residue values with both treated CO and CO/PET fabrics. Polymer deposition on the surface of treated fabrics and structure changes were followed by SEM and FT-IR spectroscopy, respectively.

Keywords: Hyperbranched polymer, Thermal stability, Thermo-gravimetric analysis (TGA), Textile finishing.

Introduction

Textile Fabrics are one of the most important materials used in our daily life ever [1]. Improving all of their properties is a universal demand. Much of the research work has been done to produce fabrics possessing desired properties such as; resistance to microorganisms, resistance to ultraviolet light, flame resistance, improving their handle and comfort properties etc. [2-4]. Cotton fabrics are considered the top type in global consuming fabrics because of their required properties like; comfort, softness, biodegradation, warmness etc. The using of cotton blend fabrics, especially with synthetic fabric like polyester, is growing very fast because it combines the good properties of each component, such as the lower cost of polyester and comfort of cotton[3]. But the lack of thermal stability of cellulose fabrics and the ease of their fire combustion is the weak point for those fabrics [5], especially when producing high-performance and protective fabrics. Another important feature of blend fabrics is their being less ignitable with slow flame spread. But once ignition is started, synthetic fabrics melt with high rate of burning which double the risk that may lead to serious burning problems[2]. Therefore, the risk of combustion of blended fabrics is considered to be larger than that of natural or evensynthetic fabrics.

Moreover, there is a global trend to reduce the using of halogen compounds as fire retardant agents because of their environmental impact [6, 7]. There is a general care for synthesis of formulations that enhances the thermal stability and therefore the flame retardancy with low environmental impact. Different lines have been carried out in the textile field, for example; phosphorus species, polyhedral oligomeric silsesquioxanes and silica particles, have been studied to reach this aim in the finishing stage of the textile stuffs [3, 7].

On the other hand, new classes of organic polymers called hyperbranched polymers (HBP), extremely branched macro-molecules with large number of reactive end-groups, have been recently studied [8-10]. These types of polymers possess superior properties from being more reactive such as compact shape, lower viscosity, better solubility, retain great numbers of reactive end functions groups [11]. Because of previously mentioned features, these polymers found large application in characteristic fields like nano-materials, biomedical applications, photochemical molecular devices, coating additives and textile finishing etc. [12, 13]. However to the best of our knowledge, the effect of HBPU treatment finishing on the thermal properties of cotton fabric and blend cotton fabric has not been studied before.

*Corresponding author e-mail: ftm_alnady@yahoo.com
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The aim of the present work is to investigate the ability of the prepared hyperbranched poly urethane-urea (HBPU) to enhance the thermal stability of cotton and cotton polyester blend fabrics. Thermal stability of fabrics is studied using thermo-gravimetric analysis (TGA) and differential thermo-gravimetric analysis (DTG) to test the impact of polymers on fabrics stability. Also, the effect of different concentrations of HBPU on tensile strength value of fabrics is examined. The change in fabrics surface morphology before and after the treatment with HBPU were investigated by scanning electron microscope (SEM).

Experimental

Material

100% scoured bleached cotton (CO) fabric of 140g/m² and 50/50 cotton polyester blend (CO/PET) Knitted fabric of 170 g/m² are supplied by Weaving, Mehalla El – Kubra, Egypt. Synthesized Hyperbranched poly (urethane-urea). N, N-dimethyl formamide (DMF) from ACROS Chemical Co.

Method

Hyperbranched poly urethane-urea (HBPU) was synthesized by direct polymerization of isophoron diisocyanate and diethanol amine monomers according to procedure described [14, 15]. In brief, to a solution of 0.1mol of diethanol amine in 100mL of DMA, 0.1 mol of isophoron diisocyanet in 100 mL of DMA was added under vigorous stirring and nitrogen atmosphere. The mixture was kept at 80°C for 120min, then poured into diethyl ether solution. The precipitate was collected and dried under vacuum at 80 °C for 24 hr.

After the fabrics have been washed and dried, they were dipped in the DMF solution with varied (1, 3, 5 & 7 g/L) concentration of HPPU finishing bath for 15min, squeezed by a laboratory padder (Mathis HVF) with two dips and two nips to approximately wet pick up 100%. The samples were dried and cured at a specified temperature (160 °C) for 6 min in a laboratory curing oven (Wemer Mathis Co., automatic thermostatic oven, Switzerland). The fabric properties were measured after the treated fabrics were subjected to one washing cycle by tab water to remove any residue of solvent or unfixed HBPU particles followed by drying.

Analysis

Fabric performance evaluation

The treated samples tensile strength is evaluated by the ASTM standard Method D5035. Asano machine textile tensile strength tester was used. The tensile specimens were cut in rectangular shapes with dimensions of 20 cm in length and 5 cm in width. Three specimens for each treated fabric were tested in the warp direction. The results obtained were the average vales of three readings.

Weight add on were calculated according to equation (1):

$$\text{Add - on (\%)} = \frac{W_2 - W_1}{W_1} \times 100 \quad (1)$$

Stiffness properties

Stiffness of printed and unprinted samples was determined according to ASTM test method D 1388 – 96 using the cantilever apparatus (ASTM 1388 – 96, 2002).

Handle properties

The change in roughness values was measured for treated and untreated fabrics using a surface roughness measuring instrument SEL700 α . The presented results are average of three readings.

Thermo-gravimetric analysis (TGA)

The thermo-gravimetric analyses (TGA) of the fabrics samples are studied using a differential scanning calorimeter analysis tool. The test was performed to study the structural changes of the treated fabrics with thermal treatment. A 10 mg of sample was heated up to 700 °C, with a heating rate of 10 °C min⁻¹ in a nitrogen flow at a rate of 100 ml min⁻¹ on SDTQ-600 (TA-USA) thermo balance instrument.

FTIR spectroscopy analysis

FT-IR spectra of the treated fabrics were recorded on JASCO FT-IR 6100 spectrometer (Tokyo, Japan). The transmission measurements are carried out within the range of 4000-400 cm⁻¹.

Scanning electron microscope (SEM) analysis

SEM micrographs of samples were recorded on a Quanta FEG-250 microscope at a voltage of 10 kV. The samples were sputter-coated with gold before scanning to avoid charging.

Results and Discussion

Characterization of synthesized HBPU

The IR spectrum of HBPU is given in Fig.1. The spectrum reveals the appearance of HBPU

characteristic absorption broad band at 3382 cm^{-1} , which is corresponding to the overlapped -OH stretching and -NH stretching vibrations. The band at 2949 cm^{-1} is assigned to C-H stretching. The two peaks around 1618 cm^{-1} & 1581 cm^{-1} may be attributed to C=O of urethano unit (NHCOO) and ureido unit (NHCONH), respectively. The bands at 1411 cm^{-1} , 1264 cm^{-1} , 1238 cm^{-1} and 1039 cm^{-1} are corresponding to C-H bending, O-H bending, C-N bending and C-O stretching of ester group, respectively. Moreover, the disappearance of absorption band in the range between 2100 cm^{-1} - 2270 cm^{-1} , which is assigned to NCO isocyanate group, is an indication of complete consuming of isocyanate groups (from isophoro diisocyanate start) during the polymerization reaction.

The M_n (The number average molecular weight), M_w (the weight average molecular weight) and PDI polydispersity index (the weight average molecular weight divided by number average molecular weight) of synthesized HBPU found to be $1.93311 \times 10^4\text{ g/mol}$, $1.4319 \times 10^4\text{ g/mol}$ and 1.35×10^0 , respectively using Gel Permeation Chromatography (GPC). The suggested structure of HBPU is given in Fig. 2 [16].

Tensile strength performance of fabrics

The tensile strength of untreated and treated cotton and cotton polyester blend fabrics with (1, 3, 5 & 7g/L) of synthesized HBPU are listed in Tables 1&2 and Fig. 3&4, respectively. From these results It is evident that, the tensile strength of CO fabrics were not significantly affected by the treatment

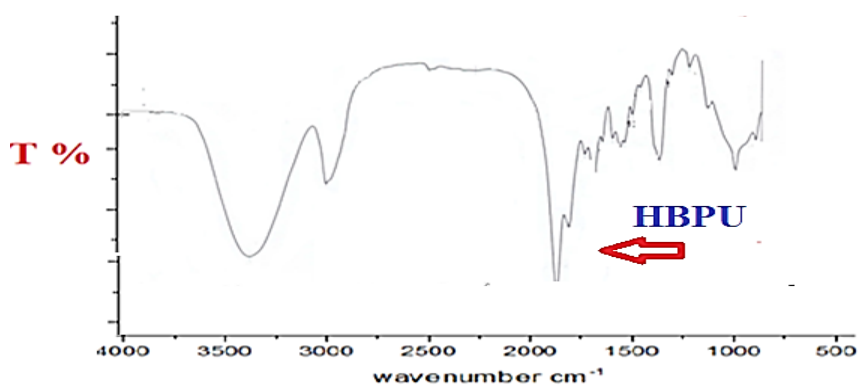


Fig. 1. FT-IR spectra of HBPU.

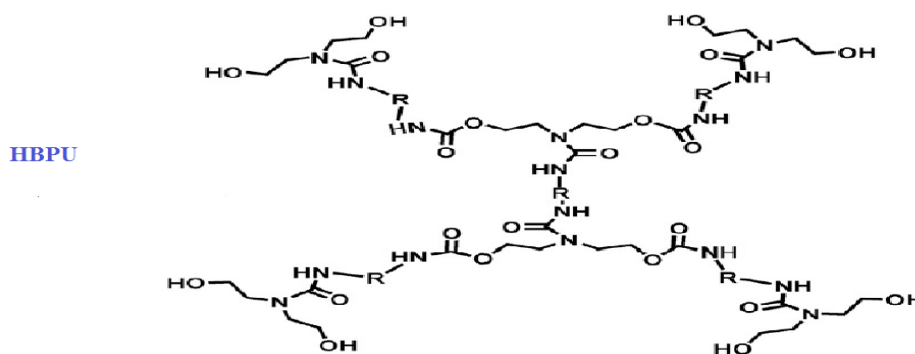


Fig. 2. HBPU structure.

with HBPU. However, when CO fabric was treated with 5 g/L of HBPU, a slight improvement in tensile strength was observed.

In the case of the CO/PET fabrics, the situation is somewhat different. The tensile strength decreased by increasing the concentration of HBPU in treatment bath, which may be due to the negative impact of HBPU treatment on the polyester fraction in CO/PET fabrics.

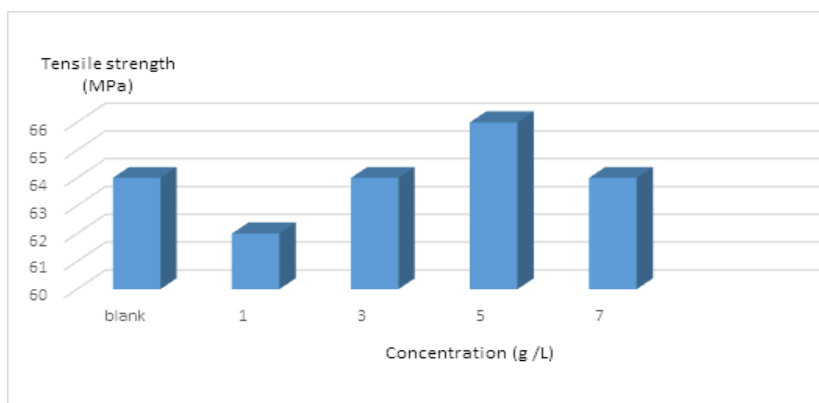
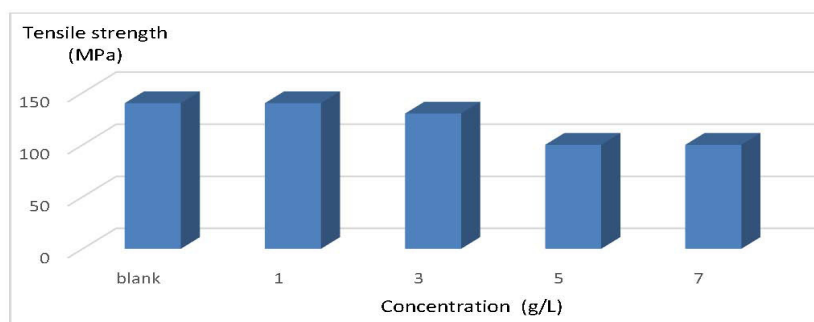
From Tables 1 and 2, It is noticed that there is an increase of the weight picked up by CO and CO/PET fabrics with increasing the concentration of HBPU in the treating bath. This could be related to the increase of HBPU deposits inside fabrics as the polymer concentration increases in treatment solution bath. Therefore, we can conclude that, the affinity of CO fabrics to retain polymer deposits inside them are less than CO/PET fabrics.

TABLE 1. Tensile strength, elongation and weight add on of CO fabrics treated with varied HBPU concentrations.

HBPU (g/L)	Tensile strength (MPa)	Elongation (%)	Add-on (%)
0	64±5.98	24±1.05	--
1	62±2.52	22±1.68	2.38
3	64±7.00	22±3.04	4.27
5	66±4.25	19±0.78	5.46
7	64±5.03	22±2.15	7.84

TABLE 2. Tensile strength, elongation and weight add on values of CO/PET fabrics treated with varied HBPU concentrations.

HBPU (g/L)	Tensile strength (MPa)	Elongation (%)	Add-on (%)
0	140±10.52	22±1.36	--
1	140±9.53	26±1.93	1.95
3	130±7.26	27±2.50	2.82
5	100±4.68	18±1.25	7.53
7	100±7.41	22±0.96	10.14

**Fig. 3.** Effect of HBPU treatment at varied concentration on the tensile strength of treated CO fabric.**Fig. 4.** Effect of HBPU at varied concentration on the tensile strength values of treated CO/PET fabrics.

Stiffness performance of fabrics

Table 3 shows the stiffness values of treated and untreated CO and CO/PET fabrics. From the stiffness values corresponding to CO fabrics It was observed that, both 3 and 5 g/L HBPU treated fabrics show improved stiffness values and have good handling when compared to 1 and 7 g/L HBPU treatment. However, in the case of CO/PET fabrics both of 5 and 7g/L HBPU treated fabrics possess superior stiffness& handling character compered to neat CO/PET.

Handling properties of fabrics

From Table 4 it is clear that, all the roughness values of treated CO/PET fabrics reflect an improvement when compared with neat CO/PET at all treatment concentration. However, in case of CO fabrics the roughness characters are slightly changed by treatment with HBPU.

Thermal behavior of fabrics

TGA and DTG analyses were carried out to investigate the thermal decomposition behavior of treated and untreated fabrics (Fig.5&6).

TABLE 3. Stiffness properties of treated and untreated CO and CO/PET fabrics with varied HBPU concentrations.

HBPU g/L	Stiffness (mg.cm)	CO	CO/PET
	Blank		1174.8
1		1317.2	1886.8
3		569.6	2670.8
5		1068.0	1103.0
7		1673.2	996.0

TABLE 4. Roughness properties of treated and untreated CO and CO/PET fabrics with varied HBPU concentrations.

HBPU g/L	Roughness	CO	CO/PET
	Blank		15.7±0.12
1		16.03±1.31	12.74±0.67
3		14.74±0.80	13.25±0.60
5		18.53±2.23	13.65±1.69
7		15.84±0.98	13.48±2.18

When cotton cellulose is exposed to thermal decomposition the pyrolysis process takes place in two steps. The first step occurred at lower temperature is corresponding to adsorbed water evaporation and char formation. The second step takes place at a higher temperature and is believed to be responsible for cellulose polymer depolymerization. The depolymerization of cellulose results in the formation of levoglucosan which in consequence leads to the creation of highly flammable volatile products [3, 17-20].

On the other hand, the thermal degradation of polyester, takes place through random scission of the ester bonds along polymer chains followed by pyrolysis of small esters formed [21].

The thermal stability of treated and untreated CO and CO/PET fabrics are studied by TGA analytical tool, the data are illustrated by Fig. 5 and 6, respectively. The mathematical explanations of TGA & DTG profiles are listed in Tables 5 and 6.

Blank CO fabric and treated CO fabric with 1 g/L HBPU demonstrate comparable decomposition performance. From DTG profiles, one degradation step with maximum degradation temperature (T_{max}) in range between (303-383) °C and (299-392) °C were observed, respectively. The appearance of one degradation step during the thermal decomposition of blank and 1 g/L treated CO fabrics at the temperature ranges clarified above, demonstrate the degradation of the samples by the formation of char from decomposition of the glycosyl units at lower degradation temperatures. As HBPU concentration increases (\gg 1g/L) in the finishing bath, the treated CO fabrics exhibit two stages of decomposition with two T_{max} of degradation. (254&297);(316&394)°C, (214&284);(304&382)°C and (241&282);(306 &380)°C. These represent the temperature ranges corresponding to the two degradation stages of CO fabrics treated with 3, 5 and 7 g/L of HBPU, respectively. From the above results it is clear that, the thermal stability of treated CO fabrics is affected by HBPU treatment. The values of T_{o2} and T_{max} of treated fabrics decreases as the concentration of HBPU increases during treatment process.

The value of char residue at different decomposition temperatures is a very distinctive signal wich distinguishes the thermal stability of the fabrics. The higher its residual value is, the greater the thermal stability of certain fabrics

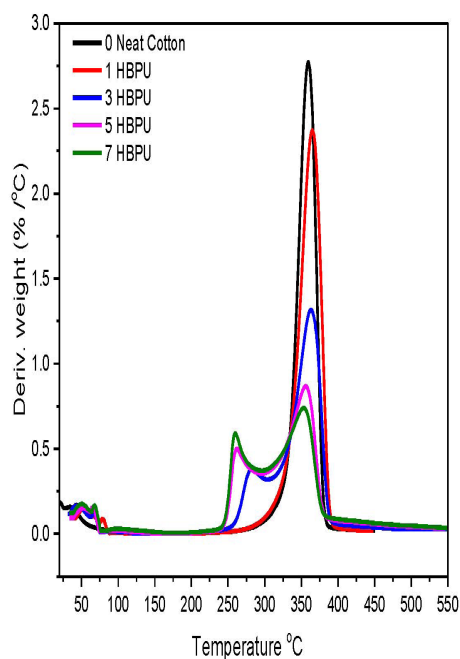
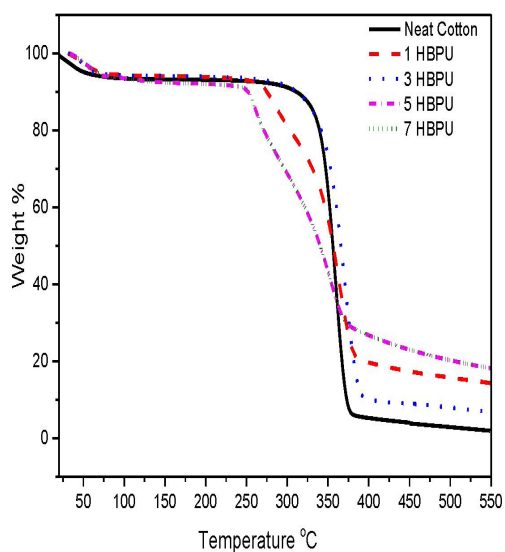


Fig. 5. TGA & DTG curves of blank and treated CO fabrics at varied concentrations (g/L) of HBPU.

will be found. The results showed that, the residual char wt% of blank CO fabric at T_{max1} and 500°C are about 42.78 and 2.72 %, respectively. A very distinctive char residue values were obtained through thermal degradation of treated of CO fabric with 5g/L HPBU at different decomposition temperatures, the values were 89.08, 42.98 & 21.85 % from the total weight of treated fabrics at T_{max1} , T_{max2} and 500°C, respectively. The higher char residue values after treatment with HBPU could be attributed to the

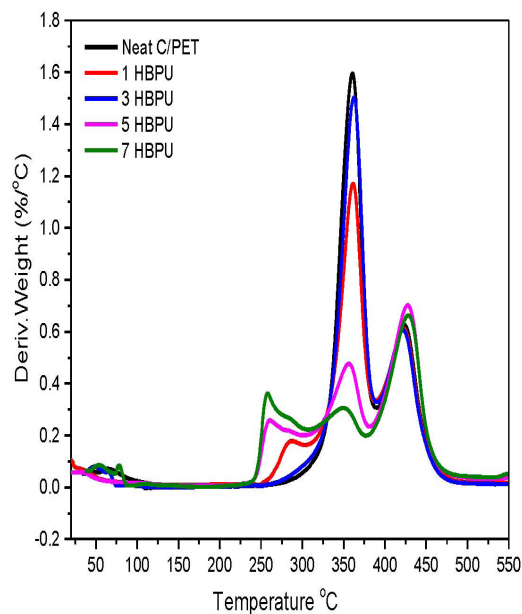
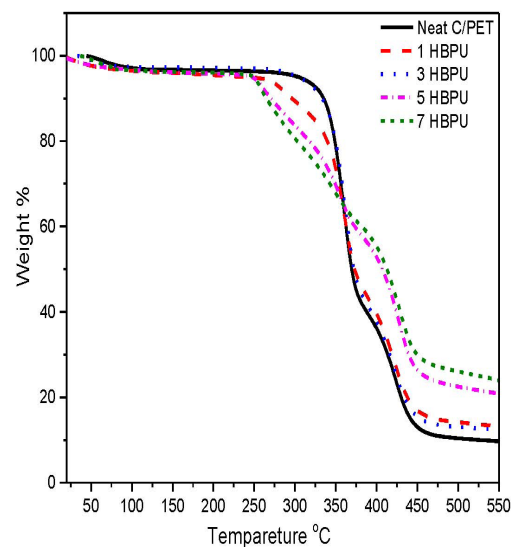


Fig. 6. TGA & DTG curves of blank and treated CO polyester blend fabrics at varied concentrations (g/L) of HBPU.

increasing ability of fabrics towards the creation of less volatiles and nonflammable products at lower temperature range, which in turn delay the thermal decomposition and increase their thermal stability. From the above results, it could be concluded that the treatment of CO fabrics with HBPU polymer enhances their thermal stability and the recommended HBPU concentration for treatment of CO fabric is 5g/L HBPU at which the remaining char residual value is about 10 times greater than the char value of blank CO fabric at

500 °C.

In case of CO/PET fabrics, the following observations were detected; all samples showed two independent stages of thermal decomposition, the first stage (at lower temperature) is due to the CO fiber degradation while the later stage (at higher temperature) is due to the decomposition

of polyester fiber [1, 22]. Also, T_{max1} and T_{o1} values decrease as HBPU concentration increases during treatment process. Highest drop in T_{max1} is observed with CO/PET fabrics treated with 7g/L HBPU. While the values of T_{max2} are almost unchanged with the change of polymer concentrations in the finishing bath.

TABLE 5. Thermo-gravimetric analytical data of untreated and treated CO fabrics at varied HBPU concentrations.

HBPU g/L	T_{o1} °C	T_{max1} °C	CR_{max1} wt%	T_{o2} °C	T_{max2} °C	CR_{max2} wt%	$CR_{500°C}$ wt%
Blank	342	358	42.78	--	--	--	2.721
1	302	360	48.42	--	--	--	3.311
3	257	282	87.54	317	364	39.68	15.571
5	239	260	89.08	308	354	42.98	21.85
7	243	258	86.87	303	353	39.81	20.27

TABLE 6. Thermo-gravimetric analytical data of untreated and treated CO polyester blend fabrics at varied HBPU concentration.

HBPU g/L	T_{o1} °C	T_{max1} °C	CR_{max1} wt%	T_{o2} °C	T_{max2} °C	CR_{max2} wt%	$CR_{500°C}$ wt%
Blank	325	361	62.11	391	424	24.91	10.14
1	321	362	60.85	393	423	27.24	14.12
3	316	360	69.28	395	422	22.52	13.06
5	312	355	66.66	387	426	37.68	22.53

Furthermore, the char residue value increases as polymer concentration increases during the treatment of CO/PET fabrics. The highest char value was observed at 7g/L of HBPU treatment. The highest thermal stability detected with CO/PET fabrics were observed at 7 g/L of HBPU, whereas the T_{max1} value drop from 361°C to 349°C and the $CR_{500°C}$ values increases from 10.14 % to 25.97 % at 500°C. Thus it can be said that, the treatment of CO/PET fabrics with synthesized HBPU increases their thermal stability and char residue values when the fabric is subjected to higher temperature atmosphere.

From the above TGA and DTA results of CO and CO/PET fabrics, It could be concluded that, the treatment of these fabrics with synthesized HBPU improves their thermal stability. Furthermore, the thermal stability enhancements in the case of CO fabrics were higher than that in the case of CO/PET fabrics. This result is in agreement with the results obtained earlier for tensile strength

measurements. However it appears that the polyester fibers do not respond well to HBPU treatment as CO fiber portion.

FT-IR analysis

The effect of treatment of CO and CO/PET fabrics with 5 and 7 g/L HBPU on the chemical composition of CO and CO/PET fabrics were studied by FT-IR spectroscopy. In general, it is clear that, HBPU treatment has a significant effect on the structure of both CO and CO/PET fabrics. From IR spectra it was noticed that, all the treated fabrics (CO and CO/PET) exhibited a change in peaks position and intensity (Fig.7&8).

All the Peaks of neat CO and CO/PET appeared after the treatment with HBPU. The intensity and shapes of FTIR peaks after treatment changed considerably. HBPU peaks are clearly overlapped with the absorption bands of CO and CO/PET fabrics.

The broad band in the range 3750 to 3000 cm^{-1} is corresponding to inter and intra hydrogen bonding stretching vibration of $-\text{OH}$ from cotton cellulose. The peaks appeared at 2920 cm^{-1} , 1650 cm^{-1} and 1440 cm^{-1} are corresponding to CH stretching, $-\text{OH}$ and $-\text{CH}_2$ bending vibrations, respectively.

In case of CO fabrics, the peak attributed to str-OH located at 3427 cm^{-1} within neat CO fabric was shifted to 3425 cm^{-1} and 3421 cm^{-1} (lower frequency) with samples treated with 5 and 7 g/L HBPU respectively. The CO cellulose C-H peak was also shifted to a lower wavenumber from 2911 cm^{-1} (neat CO) to 2901 cm^{-1} (5 g/L HBPU treated CO) and 2899 cm^{-1} (7 g/L HBPU treated CO). This shift may be attributed to the possible HBPU influence on inter and intra hydrogen bonding of cellulose OH groups and formation of new bonds.

Conversely, in case in CO/PET fabrics, the shifting in position of str-OH and str-C-H bands to lower frequency from neat to treated CO/PET fabrics were not observed. However, a significant shift in position of str-C-O from 1090 cm^{-1} to and 1082 cm^{-1} of neat and 7 g/L treated CO/PET, respectively occurred. One possible reason may be the inert chemical structure of polyester fibre in CO/PET fabric which may restrict the hydrogen bonding formation ability between CO/PET & HBPU.

Moreover, all bands intensities in case of treated CO fabrics showed increase in intensity as compared with neat CO, while reverse situation occurred with treated CO/PET fabrics (the treated fabrics show a decrease in bands intensity when compared with neat CO/PET fabric). In general, the reduction of the bands absorption intensities means that a physical change has been occurred, while the increase in the band intensities means a change in the morphology and chemical composition of that band [23].

From all the above results, it seems that, CO fabrics showed a different response from CO/PET fabrics when treated with HBPU polymer.

Scanning electron microscopy

Figures 9 and 10 illustrate the surfaces morphology changes of fabrics respectively before and after HBPU treatment as investigated by SEM analysis. The 8A and 9A images represent SEM micrographs of surface of untreated CO and CO/PET fabrics respectively, while 8B&9B

images represent SEM micrographs of treated CO and CO/PET fabrics with 5 and 7g/L HBPU, respectively. It is clear that, were formed polymer coating deposits on the surface of fabrics treated with HBPU (5 & 7g/L), which is in contrast to the clean, smooth and clear surfaces of untreated fabrics. Although there are polymer coating deposits on the surface of treated fabrics, these deposits are not affected on fabrics fine structures and still appeared retaining their properties.

Conclusion

Enhancements in tensile strength properties were achieved by treating CO fabrics with 5 g/L HBPU polymer, while CO/PET fabrics showed a reduction in tensile strength properties upon HBPU treatment. Also, the treated samples showed good handling. The thermal stability enhancement in the case of CO fabrics was higher than CO/PET fabrics. From TGA & DTG results, both of treated CO and CO/PET fabrics with 5g/L and 7 g/L of HBPU, respectively, have improved thermal stability and may be utilized in high temperatures fabrics application. The type of interaction exists between HBPU and fabric (CO and CO/PET) after treatment were investigated by the FT-IR analysis. The peaks intensity of CO fabrics were enhanced after HBPU treatment, while CO/PET peaks intensity followed a different pathway. Also FT-IR spectral data revealed that, the interaction between CO fabrics and HBPU occurred by a different mechanism from CO/PET fabrics.

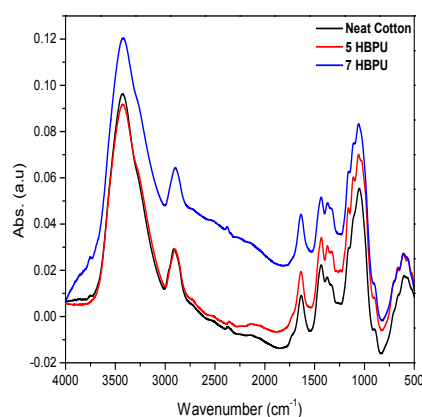


Fig. 7. FT- IR spectra of CO fabrics before and after treatment with 7 and 5 g/L HBPU.

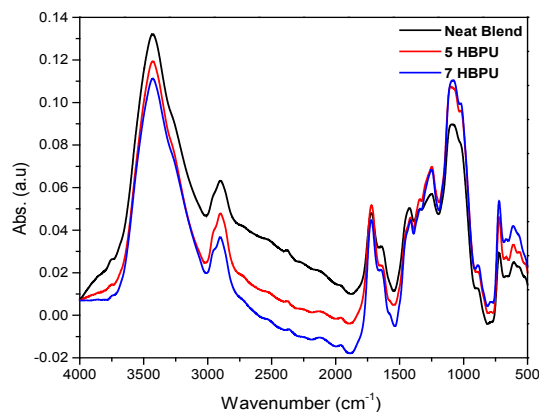


Fig. 8. FT- IR spectra of CO/PET fabrics before and after treatment with 7 and 5 g/L HBPU.

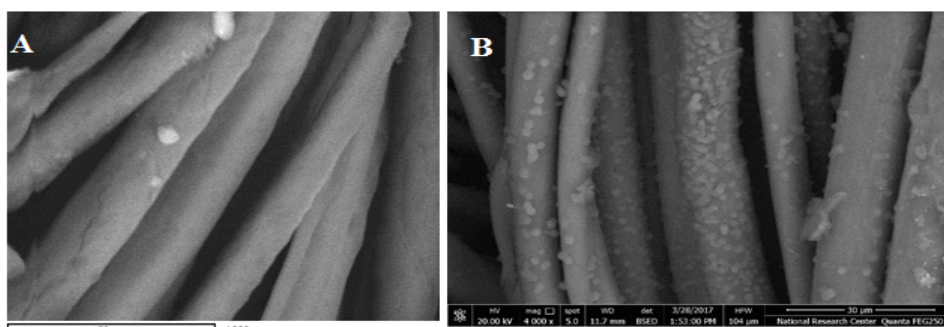


Fig 9. SEM micrographs of untreated (A) and treated CO fabric at 5 g/L HBPU (B).

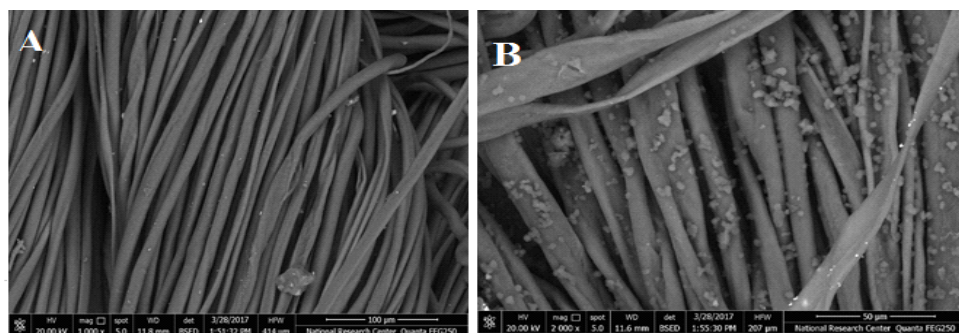


Fig. 10. SEM micrographs of untreated (A) and treated CO/PET fabrics at 7 g/L HBPU (B).

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تحسين الثبات الحراري لكلا من اقمشه القطن ومخلوط القطن البولوي استر بواسطة المعالجه بمركب البولوي يوريثان عديد التفرع

كريمه محمد منير حجاج¹، احمد اسماعيل هاشم² و فاطمة نادي الشال¹
اشعبة بحوث النسيج - المركز القومي للبحوث و²كلية العلوم - جامعة عين شمس - القاهرة -مصر.

علي الرغم من اهميه اقمشه القطن ومخاليطها في حياتنا اليوميه الا انها لا تتميز بالثبات الحراري العالي وتكون عرضه لسرعة الاحتراق عند تعرضها للحرارة مما يترتب عليه مخاطر عديدة وبالتالي فان تحسين الثبات الحراري لتلك الاقمشة يعد مطلب مهم جدا . تحسين الثبات الحراري لاقمشة القطن ومخلوط القطن والبولوي استر قد تمت دراسته بواسطة معالجة تلك الاقمشة بمركب البولوي يوريثان عديد التفرع المحضر معمليا. النتائج التي تم الحصول عليها من اختبار قوة الشد والاستطاله للعينات قبل وبعد المعالجة اوضحت ان اقمشة القطن احتفظت بقوة الشد الخاصه بها بينما اقمشة المخلوط اظهرت تناقص في قوة الشد مع زياده تركيز البوليمر في محلول المعالجة. نتائج التحليل الحراري للعينات اشارت الي زياده الثبات الحراري لكل العينات المعالجة بدليل زيادة قيمه الرماد المتبقي من العينات مقارنة بقيمة الرماد التي تم الحصول عليها من الاقمشه غير المعالجه . التحليل الحراري اوضح ايضا ان استجابة اقمشة القطن للمعالجة بالبولوي يوريثان عديد التفرع اعلي منه في حالة مخلوط القطن والبوليستر. تأثير المعالجة علي التركيب الكيميائي للاقمشة تم دراسته بواسطة الاشعة تحت الحمراء والذي بدوره اثبت حدوث تغير ملحوظ في مخطط الاشعة تحت الحمراء للاقمشة بعد المعالجة. المسح الميكروسكوبي الالكتروني اثبت وجود ترسيبات من جزيئات البولوي يوريثان عديد التفرع علي سطح الاقمشة المعالجة .