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Comparative Analysis of PET, PTT and PBT Yarns Hydrolyzed by Alkali

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ABSTRACT

Alkaline hydrolysis is a useful method for enhancing properties of polyesters. In this study, the alkali hydrolysis of three polyester filament yarns (poly(ethylene terephthalate) (PET), poly(trimethylene terephthalate) (PTT) and poly(butylene terephthalate) (PBT)) was comparatively investigated under various conditions such as alkali concentration, solvent mixture, process duration and temperature. The weight loss under different conditions was evaluated. Scanning electron microscopy was used for morphological evaluation of the samples. Tenacity loss was determined for hydrolyzed polyester yarns. Infrared spectroscopy was also applied to untreated and hydrolyzed polyester yarns. While the effect of the different conditions on weight loss of PET yarns was the highest, that of PBT yarns was the lowest. While PBT yarns lost nearly their half weight at 100 % ethanol, PTT yarns lost almost all of their weight and PET yarns were completely degraded. PBT yarns showed the fewest diameter loss percentages above 30 ºC. PTT yarns showed the highest tenacity loss for the similar weight loss values.

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KEYWORDS

Alkali hydrolysis, weight loss, tenacity loss, polyester yarns, surface morphology.

1. INTRODUCTION

Polyester fibers have many application areas such as apparel, sportswear, interior textiles, protective clothing and medical textiles due to their low cost production, easy-care properties, excellent mechanical properties and suitability in fiber blends' usage [1]. Although the most common polyester fiber is PET, there are other important types of polyester fibers used as commercially such as PBT and PTT. PBT fibers have lower tensile strength and degradation temperature, but similar surface characteristics compared to regular PET fibers [1-2]. PTT fibers, being a linear aromatic polyester have odd-numbered methylenes in the polymer chains and a bio-based origin. PTT fibers have higher resilience and elastic recovery than PBT and PET fibers. Therefore, PTT fibers are used widely in applications requiring good resilience particularly in carpets [3-5].

One of the major drawbacks of polyester fibers is their low moisture regain property that causes problems such as accumulation of static electricity and low comfort properties. Also, softness of polyester fibers is lower than that of natural fibers. Alkaline hydrolysis, which is a simple and cost effective method, is a useful tool for enhancing properties of polyester fibers such as improved hydrophility, lower static electricity charge, softer handle and better dyeability [6]. In alkaline hydrolysis, the electron-deficient carbonyl carbons of polyester are attacked by hydroxyl ions of aqueous sodium hydroxide, resulting in formation of carboxylate and hydroxyl end groups by chain scission and weight reduction [7-8]. This reaction is a topochemical degradation affecting only the surface polymer chains [9-10]. Alcohol as solvent [7, 11] and various additives in alkaline solution [10, 12] were used to increase the rate of the hydrolytic reaction on polyester.

Several studies about alkaline hydrolysis of PET reported that weight loss was a function of several variables such as temperature, duration and alkali concentration [7, 9, 13]. PET was used in different forms in the alkaline hydrolysis studies such as fabric [14], yarn [12], nonwoven [15], microfiber mat [16] and film [6]. The relationship between

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alkali hydrolysis and PET fiber spinning parameters such as spinning speed, drawing process and crystallinity were also investigated [6, 12, 17]. With alkali hydrolysis treatment of PET, while adding fluorocarbon and nano-silica particles made them super hydrophobic [18], adding surfactant and nano titanium dioxide enhanced their wettability and self-cleaning properties [19].

There are limited number of studies about alkali hydrolysis on PBT and PTT. Wang et al. reported that super hydrophobicity of melt blown PBT mats was obtained with alkali hydrolysis and subsequent fluorination [20]. Kotek et al, examined effect of spinning speed and heat setting temperature on alkali hydrolysis of PTT filaments. They reported that the weight loss clearly increased with heat-setting temperature, however the effect of spinning speed was only dominant at speeds lower than 4000 m/min [21]. Eberl et al., studied enzymatic hydrolysis of PTT fabric and film. They demonstrated that the T. fusca enzyme hydrolyzed both PTT fibers and films whereas the lipase from T. lanuginosus was only able to hydrolyze the fibers [22].

There are also limited number of studies on comparison of polyester types with alkali hydrolysis. Shukla and Mathur studied the effect of alkali hydrolysis on the two polyester fabrics (PBT and PET) by using aqueous and alcoholic solutions at varying temperatures for different durations of time [11]. They reported that PBT was much less sensitive to the action of alkali than PET. Chen et al., compared biostability of PET and PTT filaments after exposed both alkali and enzymatic degradation [23].

In this study, alkali hydrolysis of three polyester filament yarns PET, PTT and PBT was comparatively investigated. Firstly the weight loss values of the polyester yarns were evaluated under various process conditions such as alkali concentration, solvent mixture, process duration and temperature. Secondly, morphological evaluation (surface properties and diameter loss) of the samples according to weight loss values were done by using scanning electron microscopy. Thirdly, tenacity loss percentages were evaluated for hydrolyzed polyester yarns. Lastly microstructural changes were investigated for untreated and treated polyester yarns by using FTIR spectrometer with ATR mode.

2. MATERIAL AND METHOD

2.1 Material

PET, PTT and PBT multifilament yarns were used as substrates. Linear density and filament numbers of the yarns are given in Table 1. Sodium hydroxide and ethanol (purchased from Sigma Aldrich) were used without further purification for alkali hydrolysis of the polyester yarns.

Table 1. Linear density and filament numbers of the yarns

Yarn type	Linear density [denier]	Number of filaments
PET	1200	188
PTT	1200	240
PBT	1200	272

2.2 Alkali Hydrolysis Process

The alkali hydrolysis process was applied to the polyester yarns under four different groups of conditions: alkali concentration, temperature, duration and solvent type (Table 2). All hydrolysis operations were carried out in sealed flasks at a bath ratio of 50:1 with mild mechanical agitation. After the hydrolysis, the polyester yarns were neutralized with 1 wt % acetic acid solution for 10 min, rinsed with distilled water and then dried at room temperature overnight. In solvent type study, ethanol was used with water as solvents for the purpose of acceleration of hydrolysis with alcohols [7, 11].

2.3 Weight Loss Measurement

The polyester yarn samples were conditioned at 70 °C for 1 hour in a drying oven then cooled in a desiccator at room temperature and weighed using an analytical balance before and after hydrolysis process. The percentage of the weight loss was calculated using Equation (1).

$$\text{Weight loss [\%]} = \frac{W_1 - W_2}{W_1} \times 100 \qquad (1)$$

Table 2. Four different groups of conditions applied in the alkali hydrolysis process

Group No	NaOH concentration [wt%]	Water/Ethanol [v/v]	Temperature [ºC]	Duration [min]
1	2.5-5-10-20-40	100/0	60	60
2	20	100/0	30-45-60-75-90	60
3	20	100/0	60	10-20-30-40-50-60
			97	10-20-30-40-50-60
4	10	100/0-75/25-50/50-25/75-0/100	45	30

where W_1 and W_2 are the weights of the samples before and after alkali hydrolysis, respectively. The weight loss percentages were determined at all conditions applied in the alkali hydrolysis process under four groups according to Table 2.

2.4 SEM Characterization

The micrographs of the treated polyester yarns according to group 2 conditions and the untreated polyester yarns as references were taken by using a field-emission scanning electron microscope (FE-SEM, ZEISS Gemini SEM 300) at different magnifications (500X and 5000X). An image processing software (Image J®) was used to measure of fiber diameters. From the SEM micrographs, the diameter of fibers was randomly measured for each condition and the average of forty measurements were calculated. The percentage of the average fiber diameter loss was also calculated using Equation (2).

$$\text{Diameter loss [\%]} = \frac{d1 - d2}{d1} \times 100 \qquad (2)$$

where d1 and d2 are the average fiber diameters of the untreated samples and alkali hydrolyzed samples, respectively.

2.5 Tensile Strength Measurement

Tensile strength tests of the polyester yarns were performed on a Titan Universal Strength Tester according to ISO 2062. Tensile strength values of untreated and treated polyester yarns according to selected weight loss values were determined.

2.6 FTIR Analysis

The Fourier transform infrared spectrometer (Shimadzu IRTracer-100) with an attenuated total reflectance (ATR) attachment was used. The FTIR spectra of untreated and treated polyester yarns with aqueous NaOH at 40 wt% of alkali concentration, 60 ºC of temperature and 60 min of duration were measured from 400 cm^{-1} to 4000 cm^{-1} at 2 cm^{-1} resolution at room temperature.

3. RESULTS AND DISCUSSION

3.1 Weight Loss Results

The weight loss results are given in four groups of conditions: NaOH concentration, process temperature, process duration and ethanol/water solvent mixture ratio.

3.1.1 Weight loss results according to alkali concentration

Table 3 shows the weight loss of PET, PTT and PBT filament yarns hydrolyzed with aqueous NaOH according to NaOH concentration. In this group, the temperature and the duration are kept constant as 60 ºC and 60 min, respectively.

The weight loss increases for all polyester types when NaOH concentration increases. Similar results are seen in alkali hydrolysis studies for PET substrates [7, 13]. In hydrolytic degradation of polyesters, hydroxyl groups of alkali determine chain scission [23]. Hence, weight loss increases with increasing amount of hydroxyl groups resulted from increasing of alkali concentration. However effect of alkali concentration on weight loss of polyesters is at different levels. While the effect of alkali concentration on weight loss of the PET yarns is the highest, that of the PBT yarns is the lowest. At the highest alkali concentration applied, the weight loss values of the samples are determined as 23.69 %, 7.74 %, and 4.31 % for PET, PTT, and PBT, respectively. The tendency of increase in weight loss seems similar for all polyester yarns up to 20 wt% of alkali concentration. However, the weight loss of PET yarns increases about fourth times, while those of PTT and PBT yarns increase about twice with the change of alkali concentration from 20 wt% to 40 wt%.

Table 3. Weight loss [%] of polyester yarns according to NaOH concentration

NaOH concentration [wt%]	PET	PTT	PBT
2.5	1.68	0.96	0.82
5	2.82	2.02	1.29
10	4.24	2.67	2.01
20	6.44	3.62	2.10
40	23.69	7.74	4.31

3.1.2 Weight loss results according to process temperature

Table 4 shows the weight loss of PET, PTT and PBT filament yarns hydrolyzed with aqueous NaOH according to process temperature. In this group, the alkali concentration and the duration are kept constant as 20 wt% and 60 min, respectively.

Table 4. Weight loss [%] of polyester yarns according to process temperature

Temperature [ºC]	PET	PTT	PBT
30	1.89	1.38	1.19
45	2.48	2.52	1.58
60	6.44	3.62	2.10
75	23.07	12.39	5.69
97	80.14	40.31	21.57

The weight loss exponentially increases for all polyester types with the rise of process temperature. Similar results are seen in the studies for PET [10, 11, 13] and PBT [11, 20] substrates. This can be explained by Arrhenius kinetics [24]. When temperature increases, amount of chain scission increases resulted from increasing of the kinetic energy. While the effect of process temperature on weight loss of the PET yarns is the highest, that of the PBT yarns is the lowest. The highest weight loss of PET yarns is determined as 80.14 % at 97 ºC. Those values for PTT and PBT yarns at that temperature are determined as 40.31 % and 21.57 %, respectively.

3.1.3 Weight loss results according to process duration

Table 5 shows the weight loss of PET, PTT and PBT filament yarns hydrolyzed with aqueous NaOH according to process duration at 60 ºC and 97 ºC. In this group, the alkali concentration is kept constant as 20 wt%.

Table 5. Weight loss [%] of polyester yarns according to process duration

Temperature [°C]	Duration [min]	PET	PTT	PBT
60	10	2.19	1.87	0.26
	20	3.17	2.60	0.46
	30	3.40	2.70	0.67
	40	4.97	2.88	0.82
	50	6.28	3.47	1.51
	60	6.44	3.62	2.10
97	10	19.91	8.33	1.51
	20	34.69	13.42	5.89
	30	43.61	16.29	6.74
	40	60.79	19.79	14.46
	50	70.01	22.39	16.19
	60	80.14	40.31	21.57

The weight loss increases for all polyester types at applied temperatures when process duration increases. Similar results are seen in the studies for PET [10-13], PBT [11, 20] and PTT [21] substrates. In chain scission, terephthalic acid and ethylene glycol are separated and the terephthalic acid is neutralized to disodium terephthalate [25]. The process duration increases the weight loss because the disodium terephthalate dissolves in water and that needs time. While the effect of process duration on weight loss of the PET yarns is the highest, that of the PBT yarns is the lowest. The weight loss of PET yarns is determined as 6.44 % for 60 min duration at 60 °C. Those values of PTT and PBT yarns for the same conditions are determined as 3.62 % and 2.10 %, respectively. The effect of duration on weight loss of the samples at 97 °C is higher.

3.1.4 Weight loss results according to solvent mixture

Table 6 shows the weight loss of PET, PTT and PBT filament yarns hydrolyzed with NaOH according to solvent mixture. In this group, different ratios of water/ethanol solvent mixtures are used. The alkali concentration, the temperature and the duration are kept constant as 10 wt%, 45 °C and 30 min, respectively.

Table 6. Weight loss [%] of polyester yarns according to water/ethanol ratio

Water/ethanol [v/v]	PET	PTT	PBT
100/0	1.62	1.43	0.83
75/25	3.34	2.91	1.10
50/50	16.89	13.80	2.01
25/75	73.21	49.73	13.48
0/100	100.00	92.52	56.67

The effect of alkaline hydrolysis on the weight loss of the polyester yarns is the lowest when pure water is used. Increase in ethanol concentration in the solvent mixture nonlinearly increases the weight loss for all polyester types. That means the same hydrolysis effect can be obtained using lower amount of alkali, lower temperature and shorter duration when ethanol is used in the solvent mixture. Similar results are seen in the studies for PET [7, 11] and PBT [11] substrates. Efficiency of alkali hydrolysis of polyester fibers increases when solvent polarity decreases [10]. Hence, the weight loss of the polyester yarns increases when ethanol concentration in the mixture increasesdue to decreasing of solution polarity. Especially, above 50 % of ethanol concentrations there are dramatically increases in the weight loss values of all polyester types. While PBT yarns lose nearly their half weight, PTT yarns lose almost all of their weight at 100 % ethanol. Particularly, PET yarns are completely degraded at 100 % ethanol.

According to the weight loss results, the resistance of three polyester yarns to alkali hydrolysis is quite different under various conditions such as NaOH concentration, temperature, duration, and ethanol/water solvent mixture ratio. The resistance of PBT yarn to alkali hydrolysis under same conditions is the highest, while that of PET is the lowest. The main reason is attributed to the difference in the chemical structure of PET, PTT and PBT yarns. The polyester yarns chemically differ only on the number of methylene groups of the aliphatic chain: two (PET), three (PTT) and four (PBT) methylene groups. In alkali hydrolysis, chain scission is occurred on the ester linkages and the methylene groups are located near ester linkages that improves resistance to hydrolysis. Also the distance between two ester linkages increases in the order: PET<PTT<PBT. That may reduce contact of hydroxyl ions.

3.2 SEM Analysis Results

FESEM images of the treated polyester yarns according to group 2 conditions (see Table 2) and the untreated polyester yarns as the references are given in Figure 1. Alkali hydrolysis affects the surface morphology of the polyester filaments. The surface of the untreated polyester filaments are relatively smooth, while the surface of the treated filaments are textured. The surface roughness increases with the process temperature. Similar results are seen in the studies for PET [13, 15, 16, 25] and PBT [20] substrates. NaOH interacts with the surface of the polyester fibers and etches the fiber surface as temperature increases. Hence, some pits on the fiber surface are seen when etching goes on. When weight loss increases with process temperature, surface roughness, number and depth of the pits increases. PBT filaments show the fewest pores and roughness on the surface for the same process conditions. Particularly, the surfaces of PET and PTT filaments turn to sponge-like texture at 97 °C of process temperature. Table 7-9 show the average fiber diameter results of the untreated and treated polyester yarns according to group 2 conditions.

Alkali hydrolysis also causes to decrease average diameter of the polyester filaments. Similar results are seen in the studies for PET [15, 16, 23], PBT [20] and PTT [23] substrates. The outermost layers of the filaments are degraded because alkali hydrolysis is a topochemical reaction. Hence the filaments' diameters decrease caused by weight loss. Diameter loss percentages of three polyester filaments increase with the process temperature due to increase in weight loss, whereas those percentages are different for each polyester type. For instance, average diameters of PET, PTT and PBT filaments decrease 55.29 %, 32.17 % and 24.66 %, respectively at 97 °C of process temperature. PBT filaments show the fewest diameter loss percentages for the same process conditions above 30 ºC.

SEM micrographs and the diameter loss values support that PET yarns are the most susceptible to alkali hydrolysis process. PTT yarns show more resistance to alkali hydrolysis process while PBT yarns show the highest resistance. This results are related to different weight loss values of three polyester yarns for the same process conditions.

3.3. Tensile Test Results

Table 10 shows the tensile strength values of untreated and treated polyester yarns according to the selected similar weight loss values. Also, the tenacity loss percentages of the treated samples are given in Figure 2.

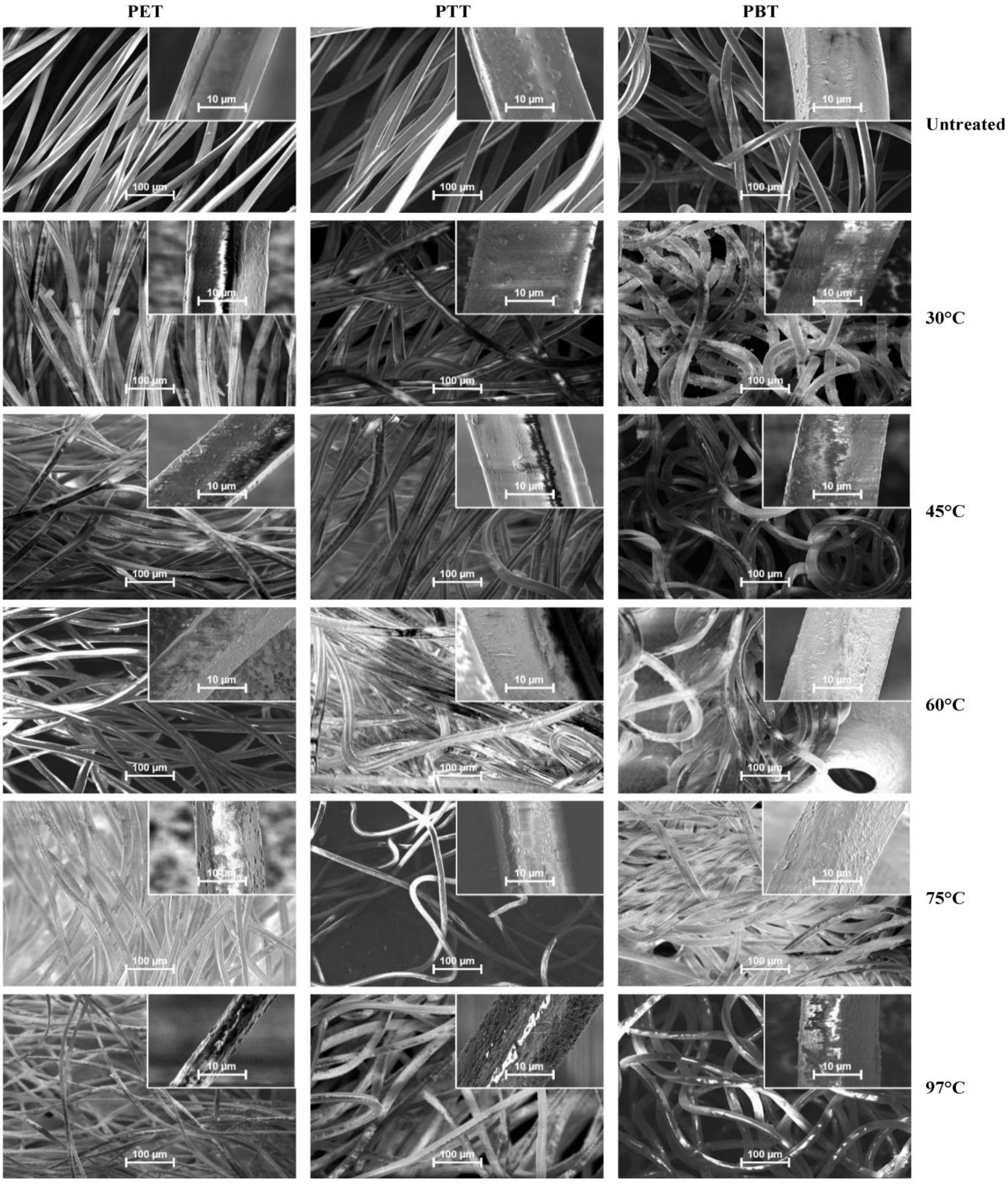


Figure 1. FESEM images of the polyester yarns according to group 2 conditions

Table 7. Fiber diameter and diameter loss of PET yarns according to process temperature

Temperature [ºC]	Weight loss [%	Fiber diameter [µm]	Diameter loss [%]
Untreated	-	22.19 ± 1.96	-
30	1.89	20.83 ± 2.34	6.13
45	2.48	18.90 ± 2.39	14.84
60	6.44	16.61 ± 2.17	25.17
75	23.07	14.80 ± 2.17	33.30
97	80.14	9.92 ± 1.07	55.29

Table 8. Fiber diameter and diameter loss of PTT yarns according to process temperature

Temperature [ºC]	Weight loss [%	Fiber diameter [µm]	Diameter loss [%]
Untreated	-	26.02 ± 2.11	-
30	1.38	22.22 ± 1.88	14.62
45	2.13	19.89 ± 1.59	23.56
60	3.62	18.41 ± 2.80	29.25
75	12.39	18.01 ± 1.43	30.79
97	40.31	17.65 ± 1.47	32.17

Table 9. Fiber diameter and diameter loss of PBT yarns according to process temperature

Temperature [ºC]	Weight loss [%]	Fiber diameter [µm]	Diameter loss [%]
Untreated	-	23.58 ± 1.97	-
30	1.19	21.43 ± 1.53	9.11
45	1.58	20.38 ± 0.98	13.55
60	2.10	19.48 ± 1.39	17.40
75	5.69	18.98 ± 1.02	19.48
97	21.57	17.76 ± 1.62	24.66

Table 10. Tensile strength values of untreated and treated polyester yarns according to the selected weight loss values

PET		PTT		PBT	
Weight loss [%]	**Tensile strength [cN/tex]**	**Weight loss [%]**	**Tensile strength [cN/tex]**	**Weight loss [%]**	**Tensile strength [cN/tex]**
0	31.04	0	15.65	0	23.41
1.89	28.09	1.87	10.23	1.9	19.47
6.28	20.12	8.33	8.61	6.74	17.86
16.89	15.00	16.29	3.71	16.19	14.20
23.65	9.20	22.39	2.03	21.57	11.71

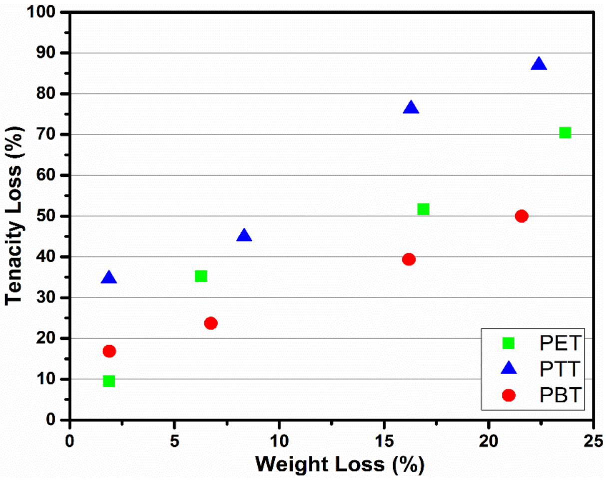


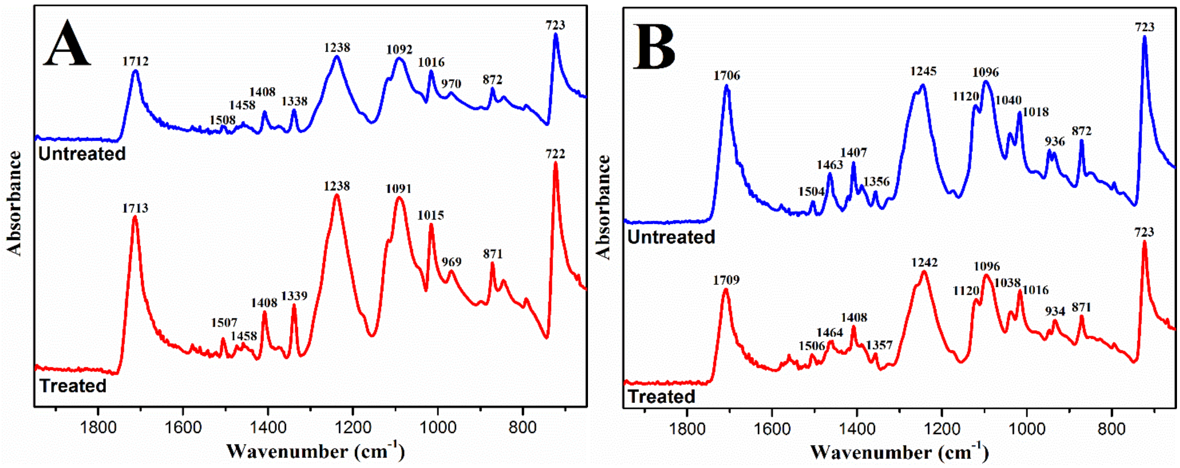
Figure 2. Tenacity loss values of the polyester yarns according to the selected weight loss values

Tensile strength of three polyester yarns decreases when the weight loss increases. Similar results are seen in the studies for PET substrates [9, 13, 14, 16]. It is related to degradation of the macromolecule chains in the hydrolysis reaction. Tenacity loss percentages of three polyesters are different from each other. PTT yarns have the highest tenacity loss percentages for the similar weight loss values. This can be related to the lowest initial tenacity of PTT yarns caused by molecular chain orientation. The amorphous regions interact more readily with hydroxyl ions than crystalline regions [15, 20]. The hydrolysis reaction goes deeper from surface in PTT filaments produces deeper cavities (Figure 1). Hence higher tenacity loss is seen in PTT yarns with smaller change in diameter and weight loss. Also, PBT yarns have the lowest tenacity loss percentages for the similar weight loss values above 5 % of weight loss.

3.4. FTIR analysis results

Figure 3 shows the FTIR spectra of untreated and treated polyester yarns with aqueous NaOH at 40 wt% of alkali concentration, 60 ºC of temperature and 60 min of duration. The weight loss values of PET, PTT and PBT yarns under those conditions were measured as 23.69 %, 7.74 % and 4.31 %, respectively.

FTIR spectra of the polyester yarns show some common peaks for PET, PTT and PBT (Figure 3). The important common bands for three polyester types shown in Figure 3 are the band at 1712 cm^{-1} corresponding to the ester groups; the band at 1458 cm^{-1} corresponding to bending vibrations of gauche methylene; the band at 1408 cm^{-1} corresponding to the ring C-C stretching vibration; the band at 1016 cm^{-1} corresponding to the ring C-H bending vibration and the band at 723 cm^{-1} corresponding to the ring C-H out of plane bending vibration [6, 26, 27]. There are also specific peaks for each polyester types in Figure 3. The band at 1338 cm^{-1} corresponding to the O-C-H bending vibration is seen as typical PET trans conformers for only PET yarns [6, 27]. The band at 1040 cm^{-1} shown in only Figure 3B associates with the C-C stretching of three methylene units of PTT in gauche conformation [26, 28]. The intensities at all major bands of PET, PTT and PBT yarns are changed after alkaline hydrolysis treatment. While the intensities at the bands for PET yarns are increased, those for PTT and PBT yarns are decreased. The changes of intensities at the bands representing ethylene glycol conformer, trimethylene glycol conformer and butylene glycol conformers indicate the change of ratio of gauche and trans conformers. The increase of intensity at the band of 1338 cm^{-1} corresponding to ethylene glycol conformers points to an increase in trans conformers of O-C-H bonds. The decrease of the intensity at the band of 1040 cm^{-1} points to a decrease in gauche conformers of $-CH_2-CH_2-$ bond [23, 28].



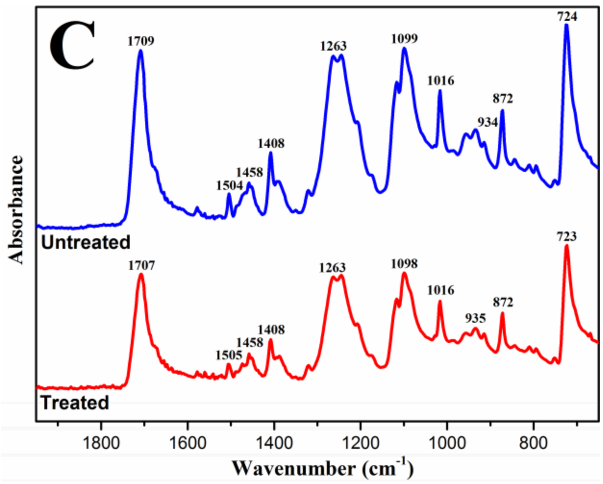


Figure 3. FTIR spectra of untreated and treated polyester yarns. A) PET yarns, B) PTT yarns, C) PBT yarns.

4. CONCLUSION

In this study, the alkali hydrolysis of three polyester filament yarns (poly (ethylene terephthalate) (PET), poly (trimethylene terephthalate) (PTT) and poly (butylene terephthalate) (PBT)) was comparatively investigated under various conditions such as alkali concentration, solvent mixture, process duration and temperature. The weight loss of the polyester yarns under various conditions were evaluated. Scanning electron microscopy was used for morphological evaluation of the samples according to weight loss values. Tenacity loss percentages were also determined for hydrolyzed polyester yarns. FTIR spectroscopy was also applied to untreated and treated polyester yarns. According to the weight loss results, the resistance of three polyester yarns to alkali hydrolysis is quite different under various conditions such as NaOH concentration, temperature, duration, and ethanol/water solvent mixture ratio. The resistance of PBT yarn to alkali hydrolysis under same conditions is the highest, while that of PET is the lowest. SEM micrographs and the diameter loss values support that PET yarns are the most susceptible to alkali hydrolysis process. This results are related to different weight loss values of three polyester yarns for the same process conditions. The PBT yarns showed the fewest diameter loss percentages for the same process conditions above 30 ºC. The PTT yarns showed the highest tenacity loss for the similar weight loss values. The intensities at all bands in FTIR spectra of PET, PTT and PBT yarns are changed after alkaline hydrolysis treatment.

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