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INFLUENCES OF ALKALI PRETREATMENT ON LYOCELL WOVEN FABRIC PROPERTIES AFTER ABRASION

ALKALI ÖN İŞLEMİN AŞINMA SONRASI LYOCELL DOKUMA KUMAŞ ÖZELLİKLERİNE ETKİSİ

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ABSTRACT

In this study, alkali pretreatment was applied to Lyocell woven fabrics at various concentrations to decrease the tendency of fiber fibrillation, and the influence of alkali pretreatment on the tensile and tearing properties of Lyocell fabrics after abrasion was investigated. Alkali pretreatment reduced fibrillation of Lyocell fibers. However, fabric shrinkage occurred because of the increased volume and damaged twisted structure of yarns due to the lateral fiber swelling. The warp/weft densities and crimp ratios increased as the alkali concentrations increased. The breaking and tearing loads of untreated Lyocell fabrics were higher than those of alkali pretreated fabrics since the strength loss caused by alkali pretreatment. The abrasive load caused fiber breakages and fiber entanglements on fabrics and decreased the both breaking and tearing loads. The weave types with long floating interlacements on the fabric surface were more severely damaged by exposing to abrasive load and resulted as higher strength reduction.

Keywords: Lyocell fabric, alkali pretreatment, weave types, tensile and tearing loads, abrasion

ÖZET

Bu çalışmada, Lyocell dokuma kumaşlara liflerin fibrilasyon eğilimini azaltmak için çeşitli konsantrasyonlarda alkali ön işlem uygulanmış ve alkali ön işlemin Lyocell kumaşların aşınma sonrası çekme ve yırtılma özelliklerine etkileri incelenmiştir. Alkali ön işlem, Lyocell liflerin fibrilasyonunu azaltmıştır. Bununla birlikte, yanal lif şişmesi nedeniyle artan hacim ve ipliklerin bükümlü yapısının zarar görmesinden dolayı kumaşta çekme meydana gelmiştir. Çözgü/atkı sıklıkları ve krimp oranları, alkali konsantrasyonları arttıkça artmıştır. Alkali ön işlemin neden olduğu dayanım kaybı nedeniyle, işlem görmemiş Lyocell kumaşların kopma ve yırtılma dayanımları, alkali ön işlem görmüş kumaşlara kıyasla daha yüksektir. Aşınma yükü, kumaşlarda lif kırılmalarına ve lif dolaşmalarına neden olmuş ve hem kopma hem de yırtılma dayanımlarını azaltmıştır. Kumaş yüzeyinde uzun atlamalara sahip dokuma türleri, aşınma yüküne maruz kalarak daha fazla hasar görmüş ve bu da daha fazla dayanım kaybı ile sonuçlanmıştır.

Anahtar Kelimeler: Lyocell kumaş, alkali ön-işlem, dokuma tipleri, çekme ve yırtılma yükleri, aşınma

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INTRODUCTION

The demand for renewable, sustainable and environmentally friendly biopolymers in both production and end-of-life has provided some improvements in the production of regenerated cellulosic fibers (Gindl et al. 2006; Klemm et al. 2005). The non-toxic N-methylmorpholine-N-oxide monohydrate solution is used in the production of Lyocell (Tencel®) fibres that prevents excessive environmental pollution of viscose fiber production (Broadbent, 2001; Franks and Varga 1980). Lyocell fibres, which are produced according to wet drawing process, consist of a combination of elementary fibrils partially separated by thin-long voids extending parallel to the fiber axis (Ozturk et al., 2009). Separation of hydrogen bonds that form lateral bonds between fibrils causes fibrillation (Zhang, Shao, and Hu, 2006; Zhang, Okubayashi, and Bechtold, 2005). Fibrillation negatively affects the structural and visual of fibers (Umur, 2010). Alkali treatment, enzymes, reactive dyes, cross-linkers, resin-based finishing agents and metal ions are used to control the fibrillation tendency of Lyocell fibres. However, alkali treatment is used as the simplest and most effective method (Renfrew and Phillips, 2003; Nechwatal et al., 1999). Mechanical, structural and surface properties, colours and fibrillation tendencies of Lyocell fibers are changed depending on the alkali types and concentrations, material forms, process temperatures and the auxiliary chemicals used. The alkali pretreatment improved the surface properties of Lyocell fibers and the fibrillation tendency of the alkali pretreated Lyocell yarns decreased (Yolacan, 2009). The fibrillation tendency of Lyocell fibers decreased by the increase in sodium hydroxide (NaOH) concentrations. At higher NaOH concentrations, there is no tendency of fibrillation because of the lateral fiber swelling (Ozturk, Okubayashi, and Bechtold, 2006). Siroky et al. (2011) examined the staining behaviors of reactive dyes by alkali pretreated Lyocell woven fabric with NaOH solution. The highest dye intake was provided in Cellulose II fibers treated with 2.53 and 3.33 mol/dm^{-3} NaOH solution. This concentration range increased color yield due to the increase in the pore volume accessible to the fiber. However, viscose fibers do not exhibit pore size changes on alkali treatments (Jaturapiree et al., 2011). Alkali pretreatments caused in lowered crease recovery, abrasion resistance, and tensile strength (Manian et al., 2006) while the reduction of fibrillation leads to better pilling resistance of Lyocell fabrics (Periyasamy, 2020). Manian et al. (2008) proposed a model related with changes occurred in Lyocell fiber structure depending on the alkali pretreatments that causes strength loss. There were no significant differences in crystallinity but, reduce in orientation degree among crystallites and amorphous regions can be caused strength loss. Rojo et al. (2013) reported an increase in amorphous regions of Lyocell fibers with a reduce in crystallinity. Swelling occurred in both Lyocell microfibers and fibers after alkali pretreatment.

Fibrillation of Lyocell fibers increases the abrasion tendency and abraded fabrics have more tendency to failure under tensile or tear loads. There are many studies in the literature to decrease the fibrillation of Lyocell fibers by alkali pretreatments. However, they are mainly concentrated on the effects of alkali pretreatment on Lyocell fiber properties. For adopting the exposed deformations of fabrics during using, it is necessary to investigate the effects of alkali pretreatment on Lyocell fabric properties. In the present work, alkali pretreatment in various concentrations was applied to Lyocell woven fabrics to decrease the fibrillation tendency of fibers and the influences of alkali pretreatments on tensile and tearing properties of Lyocell fabrics after abrasion were investigated. Tensile and tearing properties of untreated and alkali pretreated samples were compared before and after the abrasion test. The novelty of this study is investigating the effect of alkali pretreatment on fabric properties after abrasion cycles which were varied by considering weave types.

MATERIALS AND METHODS

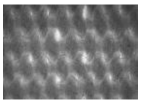
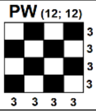
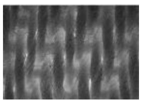
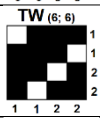
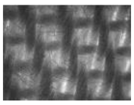
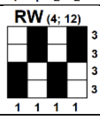
Lyocell Woven Fabrics

Lyocell woven fabrics were designed in different weave patterns as 1/1 plain (PW), 3/1 twill (TW) and 2/2 warp ribs (RW). Before alkali pretreatment, the fabric samples were washed with a non-ionic washing agent (1 g/l) at 60°C for 20 minutes at a liquor ratio of 1:100 and then rinsed and dried in the laboratory environment. Table 1 presents the specifications and microscopically views of fabrics after washing. Ne 30/1 Lyocell yarns were used in both the warp and weft directions of fabrics. The warp and weft densities of fabrics were 60 ends/cm and 20 ends/cm, respectively. The interlacements of fabrics affect many structural properties. The equivalent unit-cells of PW, TW and RW fabrics are different from each other. Therefore, the number of interlacements is also presented in Table 1 which was calculated for equivalent unit-cells of fabrics. As seen in Table 1, PW had a total of 24 interlacements (12 wefts and 12 warps), TW had a total of 12 interlacements (6 weft and 6 warp) and RW had a total of 16 interlacements (4 warp and 12 weft). PW, PT and PR fabrics were used since they had different interlacements to understand more clearly the effects of alkali pretreatment on tensile and tearing properties of Lyocell fabrics after abrasion.

Method

Sodium hydroxide (NaOH, 99%, Merck) was used for alkali pretreatment. The fabric samples were kept in alkali solution of 2, 5 and 7 mol/L at room temperature for 2 hours and stirred in every 30 minutes. The liquor ratio was used as 1:20. After that, the fabric samples were rinsed under tap water and then rinsed with distilled water. Acetic acid was used for adjusting the rinsing bath at pH 5. Rinsing with distilled-water was continued until the solution was neutralized and the fabric samples were dried in the laboratory environment.

Table 1. Specifications of Lyocell Woven Fabrics (Atıcı and Kaya, 2019)

Fabric type	Fabric view	Fabric interlacements	Crimp ratio (%)		Weight (g/m^2)	Thickness (mm)
			Warp	Weft		
PW			12.06 (±0.55)	10.02 (±0.81)	184.74 (±4.37)	0.44 (±0.02)
TW			9.54 (±0.78)	3.10 (±0.08)	182.53 (±3.23)	0.61 (±0.01)
RW			7.89 (±0.62)	4.44 (±0.00)	176.40 (±6.21)	0.58 (±0.01)

Fibrillation Tendency and Diameter Measurements of Lyocell Fibers

The Lyocell fibers' fibrillation tendency was evaluated by SEM images (ZEISS EVO® LS10). Diameter measurements of untreated and alkali pretreated Lyocell fibers were performed by using an image analysis software (BAB Bs200Doc) on SEM images.

Crimp and Density Measurements of Lyocell Fabrics After Alkali Pretreatment

The crimp and density measurements of Lyocell woven fabrics in warp and weft directions were performed after alkali pretreatment in accordance with ISO 7211-3 and TS 250 EN 1049-2 test standards, respectively.

Abrasion Test

The abrasion tests were performed using Martindale method according to ISO 12947-3 standard at 5000, 10000 and 15000 abrasion cycles. The tensile and tearing properties of abraded fabrics were examined. For this purpose, the sample dimension of abrasion test was adapted to the tensile and tearing tests. Fabric samples were placed on the holder and followed by polyurethane foam and metal coins with a diameter of 28 mm and a thickness of 2 mm instead of the conical piece (Bilisik and Yolacan, 2011). The excess parts of the fabric were folded, the sample holder was closed and made ready for testing. Lyocell fabrics were abraded in contact with standard wool fabrics.

Tensile and Tearing Tests

The tensile and tearing tests of the untreated and alkali pretreated Lyocell fabrics in warp and weft directions before and after abrasion were performed in a Hounsfield H5KS (UK) test device according to ISO 13934-1 and 13937-2 standards, respectively. After abrasion test, the width of samples was reduced to 32 mm by removing the yarn sets that were out of abraded region. The test speed was 100 mm/min for both tensile and tearing tests.

In this study, three different concentrations of alkali pretreatment were applied to three different weave types of Lyocell woven fabrics. The untreated fabric samples were mentioned as 0 mol/L. The tensile and tear strength results before and after abrasion of untreated and alkali pretreated Lyocell fabrics were compared by considering the both weave types of fabrics and alkali concentrations. In order to evaluate all these test results, analysis of variance (ANOVA) at 95% confidence interval was performed using SPSS V.24 package program. The Post-Hoc SNK (Student-Newman-Keuls) test was also performed to determine the change between subgroups.

RESULTS AND DISCUSSIONS

Fibrillation Tendency and Diameter Measurement Results of Lyocell Fibres

SEM images of Lyocell fabrics are presented in Figure 1. Diameter measurement results of untreated and alkali pretreated Lyocell fibers are given in Figure 2.

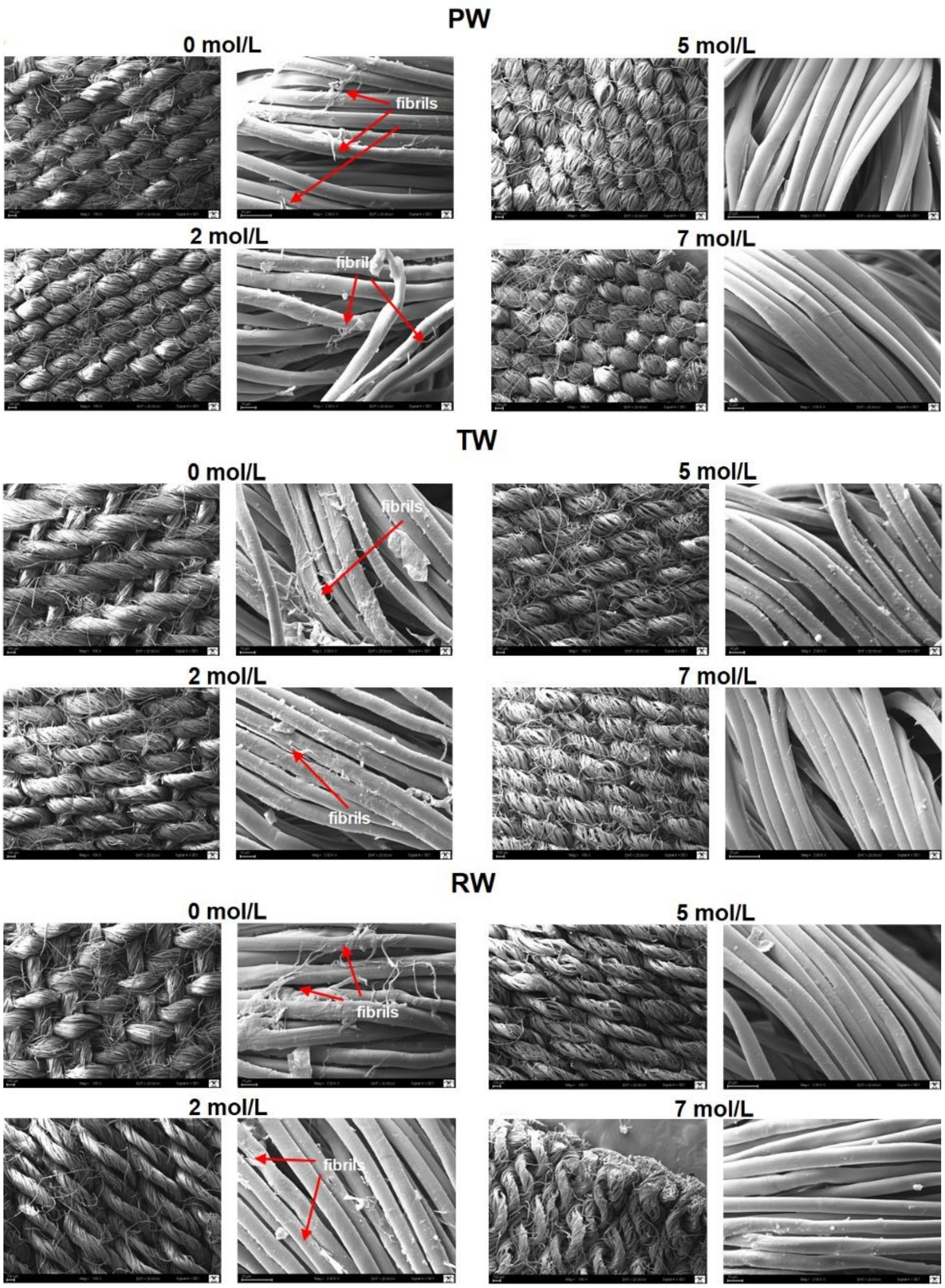


Figure 1. SEM Images of Lyocell Fabrics before and after Alkali Pretreatment (left images 100X, right images 2KX magnification) (Atıcı and Kaya, 2019)

A significant decrease was observed in fibrillation of Lyocell fibers at 2 mol/L alkali concentration, and fibrillation disappeared completely at 5 and 7 mol/L alkali concentrations. However, a noticeable change in the fabric appearance was observed as the alkali concentration increased. It was determined that the yarn sets got closer to each other and the yarn structure and fabric surface appearance were adversely affected, especially at 5 and 7 mol/L alkali concentrations.

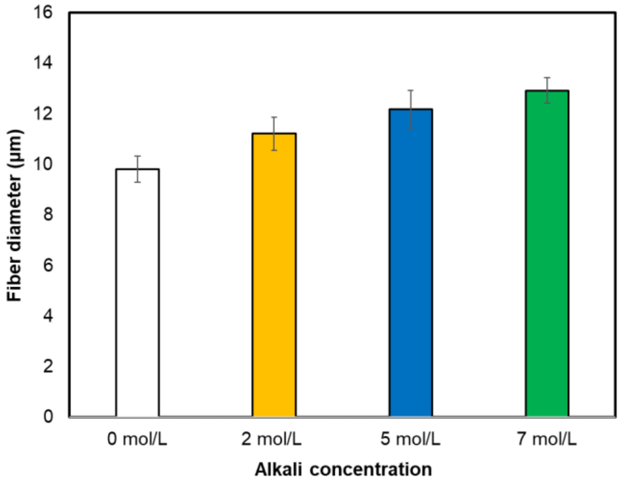


Figure 2. Diameter Measurement Results of Untreated and Alkali Pretreated Lyocell Fibers

The Lyocell fiber diameters increased by the increase in alkali concentrations because of the lateral swelling of fibers in alkali conditions (Manian et al., 2018; Xu et al., 2017; Atıcı and Kaya, 2019). The diameter of 7 mol/L alkali pretreated Lyocell fibres was about 25% higher than the untreated Lyocell fibers. Alkali pretreatment increased the volume of yarns in Lyocell fabrics due to the lateral fiber swelling and caused to damage on the twisted structure of yarns. The dense fibril appearance of untreated Lyocell fibers reduced at an alkali concentration of 2 mol/L. The fibrillation was not occurred at 5 and 7 mol/L NaOH concentrations due to lateral fiber swelling (Ozturk et al., 2006). More damages were occurred in the yarn/fabric structures of TW and RW fabric types with alkali pretreatment compared to the PW fabric due to the lower interlacements of TW and RW fabrics in which the yarns had more place to move and opening their twists.

Crimp and Density Measurement Results

The crimp and density results of untreated and alkali pretreated Lyocell fabrics in warp and weft directions are given in Figure 3.

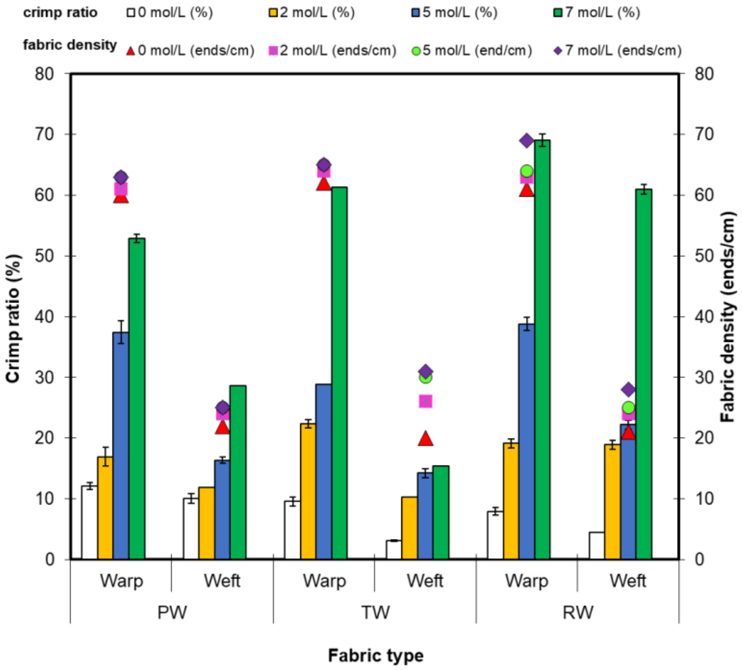


Figure 3. The Crimp and Density Results of Untreated and Alkali Pretreated Lyocell Fabrics

As the alkali concentration increased, both the warp and weft crimp ratios and fabric densities increased in all weave types. Alkali pretreatment increased the volume of yarns in fabrics due to the increase in Lyocell fiber diameter and opened the twist that caused shrinkage (Poongodi et al., 2021). Due to this shrinkage, the warp and weft crimp ratios and fabric densities increased.

The higher increase in warp/weft crimp ratios with alkali pretreatment occurred in RW weave type, followed by TW and PW because of the RW had the lowest warp interlacements (4), followed by TW (6) and PW (12). The yarns of fabric that had long floating interlacements were more crimped caused by the shrinkage with alkali pretreatment. The higher number of interlacements prevented the yarn movements and the intersection points resisted the shrinkage caused by alkali pretreatment.

Tensile Test Results

The breaking loads and breaking elongations of untreated and alkali pretreated Lyocell fabrics in warp and weft directions before and after abrasion are given in Figure 4.

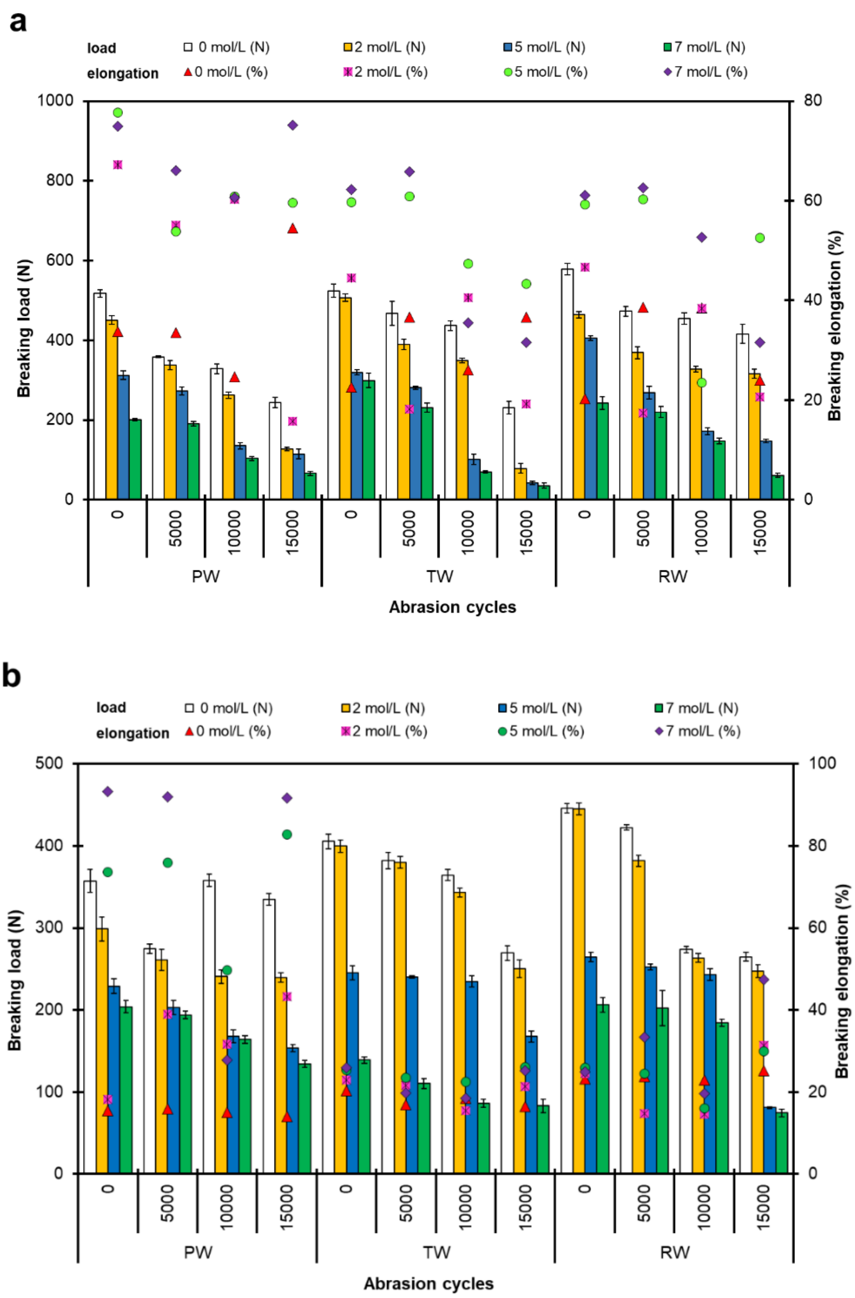


Figure 4. The Breaking Loads and Breaking Elongations of Untreated and Alkali Pretreated Lyocell Fabrics in Warp (a) and Weft (b) Directions before and after Abrasion

The warp directional breaking loads of untreated Lyocell fabrics were higher than the weft directional breaking loads before abrasion. This was because the warp density was higher than the weft density. As the alkali concentration increased the breaking loads decreased, although the warp and weft densities increased. The reduced carboxyl groups and crystallinity by alkali pretreatment in the Lyocell fiber caused to strength loss (Rojo et al., 2013; Ozturk et al., 2009; Yolacan, 2009). The breaking loads in the warp and weft directions decreased as the abrasion cycle increased in PW, TW and RW fabrics since the abrasive load caused fiber breakages and fiber entanglements. The difference between the warp directional breaking loads before and after abrasion was higher compare to the weft direction in TW fabric. The warp yarns of TW fabric had long floating interlacements on the fabric surface that were more severely damaged by exposing to abrasive load (Bilisik and Yolacan, 2011).

The warp directional breaking elongations of untreated Lyocell fabrics were higher than the weft directional breaking elongations since the warp crimp ratio was higher than the weft crimp ratio. Both the warp and weft directional breaking elongations increased by the increase in alkali concentrations since the increased crimp ratios due to the fabric shrinkage with alkali pretreatment. The warp and weft directional breaking elongations generally decreased with the increase in abrasions cycles since the abrasive load caused fiber breakages.

The ANOVA results for breaking loads, breaking elongations and tearing loads of Lyocell fabrics in warp and weft directions are given in Table 2. The alkali concentration, weave type and abrasion cycles were statistically significant on the warp/weft directional breaking loads and breaking elongations of Lyocell fabrics according to ANOVA results. The effects of interaction between alkali concentration and weave type, alkali concentration and abrasion cycles, weave type and abrasion cycles were statistically significant. The effects of interaction between all three variables were also significant for warp/weft breaking loads and breaking elongations. The alkali concentration had the highest effect on both the warp and weft directional breaking loads by considering F values of ANOVA results. Besides, the alkali concentration had the highest effect on warp directional breaking elongations while it was weave type for weft directional breaking elongation according to F values of ANOVA results.

The SNK results of breaking load, breaking elongation and tearing loads in the warp and weft directions are presented in Table 3. A significant difference was determined between the warp/weft directional breaking loads of the untreated and alkali pretreated fabrics in 2, 5 and 7 mol/L alkali concentrations. The highest warp directional breaking load was obtained in untreated fabrics while the lowest warp directional breaking load was obtained in 7 mol/L alkali pretreated fabrics. A significant difference was also determined between the warp/weft directional breaking loads of PW, TW, RW weave types. The PW fabric showed the lowest warp/weft directional breaking loads while RW showed the highest warp/weft directional breaking loads. The warp/weft directional breaking loads decreased with the increase in abrasions cycles in which the abrasive load caused damage on the yarn sets of fabrics.

Table 2. ANOVA Results of for Breaking Loads, Breaking Elongations and Tearing Loads in Warp and Weft Directions

Direction		Breaking load (N)		Breaking elongation (%)		Tearing load (N)	
	Source	**F**	**P**	**F**	**P**	**F**	**P**
Warp	**Alkali concentration (A)**	3696.805	0.000	1093.123	0.000	4002.540	0.000
	Weave type (B)	388.919	0.000	629.163	0.000	682.210	0.000
	Abrasion cycles (C)	2942.813	0.000	259.184	0.000	638.605	0.000
	A * B	50.250	0.000	34.839	0.000	19.870	0.000
	A * C	54.286	0.000	194.163	0.000	130.680	0.000
	B * C	98.834	0.000	20.689	0.000	47.967	0.000
	A * B * C	33.670	0.000	80.882	0.000	21.640	0.000
Weft	**Alkali concentration (A)**	4872.412	0.000	2651.729	0.000	9757.389	0.000
	Weave type	149.940	0.000	5769.804	0.000	2712.538	0.000
	Abrasion cycles (C)	1325.300	0.000	814.345	0.000	5790.028	0.000
	A * B	207.633	0.000	1596.608	0.000	197.897	0.000
	A * C	31.351	0.000	204.057	0.000	305.276	0.000
	B * C	182.222	0.000	227.076	0.000	457.212	0.000
	A * B * C	55.983	0.000	136.961	0.000	159.236	0.000

Table 3. SNK Results of Parameters on Breaking Load, Breaking Elongation and Tearing Load

Parameters		Breaking load (N)		Breaking elongation (%)		Tearing load (N)	
		Warp	Weft	Warp	Weft	Warp	Weft
Alkali concentration	**0 mol/L**	419.05 a	346.05 a	32.45 d	18.96 d	31.75 a	46.85 a
	2 mol/L	331.68 b	312.49 b	36.99 c	24.88 c	18.88 b	29.57 b
	5 mol/L	214.46 c	206.79 c	54.91 b	39.64 b	15.12 c	21.11 c
	7 mol/L	155.54 d	148.44 d	56.66 a	43.26 a	11.97 d	16.71 d
Weave type	**PW**	251.39 c	238.32 c	54.60 a	48.68 a	16.33 c	21.85 c
	TW	272.72 b	256.25 b	40.68 b	21.25 b	19.43 b	30.15 b
	RW	316.43 a	265.77 a	40.48 b	25.11 b	22.54 a	33.68 a
Abrasion cycles	**0**	401.76 a	303.26 a	52.48 a	32.78 b	23.04 a	41.23 a
	5.000	321.49 b	275.25 b	47.42 b	33.41 b	21.01 b	30.09 b
	10.000	240.68 c	243.61 c	42.42 c	22.65 c	18.72 c	26.55 c
	15.000	156.81 d	191.66 d	38.69 d	37.90 a	14.95 d	16.37 d

A significant difference was determined between the warp/weft directional breaking elongations of untreated and alkali pretreated fabrics in 2, 5 and 7 mol/L alkali concentrations. As seen in Table 3, the warp/weft directional breaking elongations increased as the alkali concentrations increased due to the increased crimp ratios caused by alkali pretreatment. The warp/weft directional breaking elongations of PW fabric were significantly higher than that of the TW and RW fabrics while the warp/weft directional breaking elongations of TW and RW were close to each other and were not statistically different. The warp/weft directional breaking elongations decreased with the increase in abrasions cycles.

Tearing Test Results

The tearing loads of untreated and alkali pretreated Lyocell fabrics in warp and weft directions before and after abrasion are given in Figure 5. The weft directional tearing loads of untreated Lyocell fabrics were higher than the warp directional tearing loads before abrasion. This was because the warp densities of fabrics were higher than the weft densities. The higher fabric density prevented the shear of the yarns under tearing force, and thus it could be hard to resisting of the yarns against the tearing force together. The warp/weft tearing loads Lyocell fabrics decreased as the alkali concentration increased. Alkali pretreatment caused the both decreased fiber strength and increased fabric density. The increase in the warp/weft directional fabric density also caused a decrease in the tearing loads of PW, TW and RW fabrics. The tearing loads in warp and weft directions also decreased as the abrasions cycles increased. Abrasive load caused fiber breakages and fiber entanglement that greatly reduced the tear strength of Lyocell fabrics.

The alkali concentration, weave type and abrasion cycles were statistically significant on warp/weft directional tearing loads of Lyocell fabrics according to ANOVA results in Table 2. The effects of interaction between alkali concentration and weave type, alkali concentration and abrasion cycles, weave type and abrasion cycles were statistically significant on warp/weft directional tearing loads. The effects of interaction between all three variables were also significant. The alkali concentration had the highest effect on both warp and weft directional tearing loads by considering F values of ANOVA results. As seen in Table 3, a significant difference was determined between the warp/weft directional tearing loads of the untreated and alkali pretreated fabrics in 2, 5 and 7 mol/L alkali concentrations according to the SNK results. The highest warp/weft directional tearing load was obtained in untreated fabrics while the lowest warp/weft directional tearing load was obtained in 7 mol/L alkali pretreated fabrics. A significant difference was also determined between the warp/weft directional breaking loads of weave types. The PW showed the lowest warp/weft directional tearing load while RW showed the highest warp/weft directional tearing load. The warp/weft directional tearing loads decreased with the increase in abrasions cycles in which the abrasive load caused damage on the yarn sets of fabrics.

CONCLUSIONS

The influences of alkali pretreatment on tensile and tearing properties of Lyocell woven fabrics after abrasion were examined. Lyocell woven fabrics were designed in different weave patterns as 1/1 plain, 3/1 twill and 2/2 warp ribs. NaOH was used for alkali pretreatment in 2 mol/L, 5 mol/L and 7 mol/L concentrations.

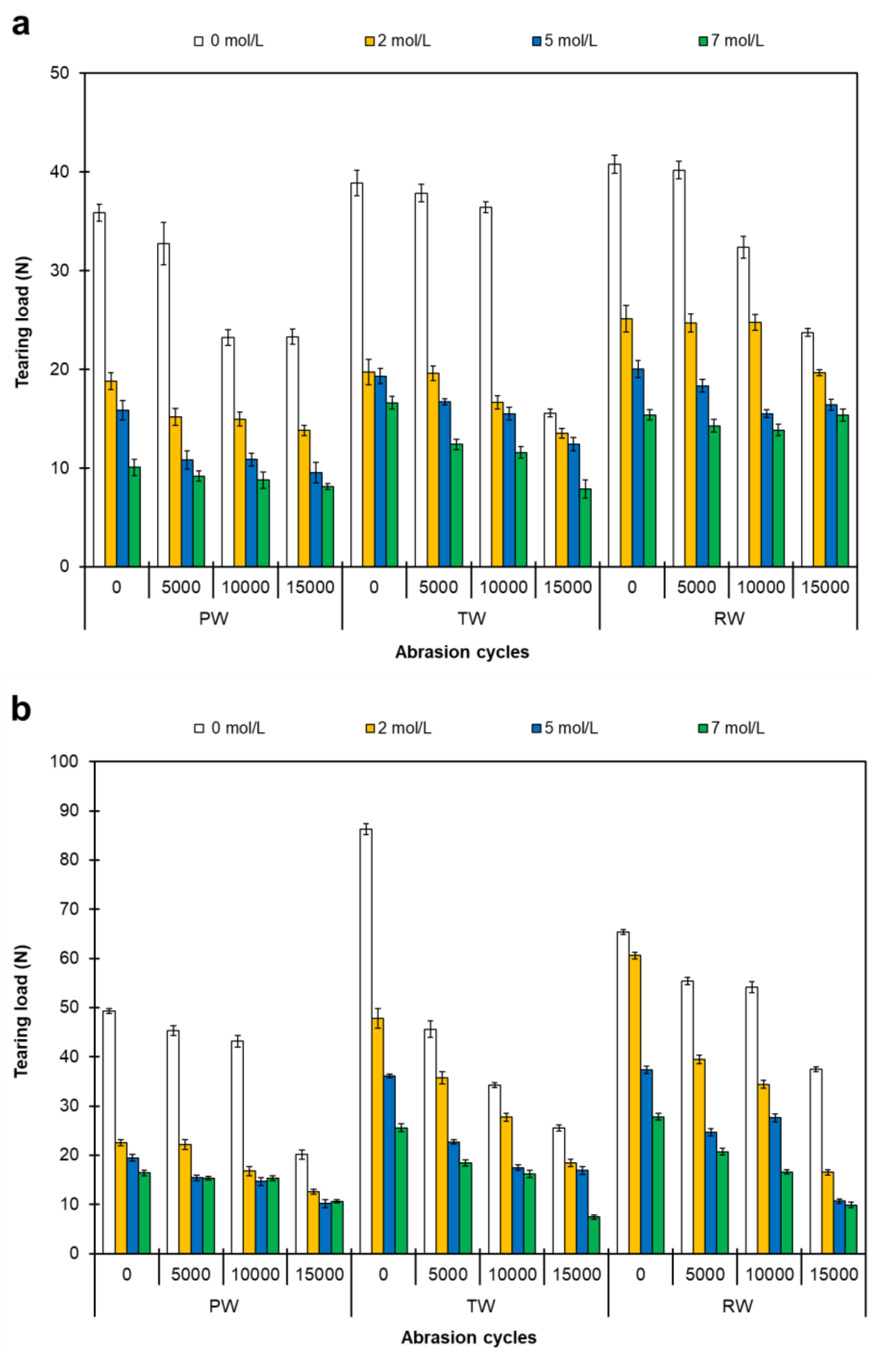


Figure 5. The Tearing Loads of Untreated and Alkali Pretreated Lyocell Fabrics in Warp (a) and Weft (b) Directions before and after Abrasion

The following conclusions are:

- Alkali pretreatment reduced fibrillation of Lyocell fibers. The dense fibril appearance of untreated Lyocell fibres greatly reduced at an alkali concentration of 2 mol/L and the fibrillation was not occur in 5 and 7 mol/L NaOH concentrations due to lateral fiber swelling. The lateral fiber swelling also caused the shrinkage because of the increased volume and damaged twisted structure of yarns (Poongodi et al., 2021).

- Both the warp and weft densities and crimp ratios increased as the alkali concentration increased in all weave types. The increase in warp/weft crimp ratios of ribs fabric was higher than that of the twill and plain fabrics because of the less number of interlacements in ribs fabric. The breaking and tearing loads of untreated Lyocell fabrics were higher than those of alkali pretreated fabrics since the reduced carboxyl groups and crystallinity by alkali pretreatment in the Lyocell fibers caused to strength loss (Rojo et al., 2013; Ozturk et al., 2009; Yolacan, 2009). The abrasive load caused fiber breakages and fiber entanglements on fabrics and decreased the both breaking and tearing loads. The breaking elongations increased by the increase in alkali concentrations since the increased crimp ratios due to the fabric shrinkage with alkali pretreatment. The breaking elongations generally decreased with the increase in abrasion cycles since the abrasive load caused fibre breakages.

- The weave types that had long floating interlacements on the fabric surface were more severely damaged by exposing to abrasive load and resulted as more decreased strength.

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