

## HIGH DURABLE CELLULOSIC TEXTILES – STRATEGIES FOR HIGH RESISTANCE TO FIBRILLATION AND PILLING

### HOCHBESTÄNDIGE ZELLULOSE TEXTILIEN – STRATEGIE FÜR WIDERSTANDSFÄHIGKEIT GEGEN PILLING UND FIBRILLATION

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#### Abstract

The mechanism of pill formation in lyocell fabric including fuzz formation and fibrillation in wet state was studied. The fuzz was mainly generated by mechanical abrasion in dry condition while the fibrillation was induced by mechanical abrasion in wet condition. The pilling was formed only on the fabric treated with wash and dry treatments. The fiber/fiber friction was measured as counts of twist to open the yarn until it starts to slip in dry and wet states. The pilling was promoted by lower fiber/fiber friction in dry state and higher fiber/fiber friction caused by higher fiber swelling in wet state. The influence of fiber swelling on fibrillation tendency of lyocell fiber was also investigated. The critical degree of swelling for lyocell fiber with no fibrillation was  $0.45 \text{ cm}^3/\text{g}$  in ethanol/water mixture. The fibrillation was retarded with alkali treatment in aqueous NaOH and KOH solutions at concentrations between 3.0 and 7.0 mol/l, and minimized at 5.0 mol/l where the uniform reorganization of macrofibrils was observed with scanning electron microscope. The fibril number of lyocell fiber treated in trimethylammonium hydroxide was enhanced with increasing concentration and weight loss. The fibrillation was retarded by crosslinking with 1,3-dimethylol-4,5-dihydroxyethylene urea and by treatment with aminofunctional polysiloxane accompanying decrease in water retention capacity.

Keywords: *alkali treatment, crosslinking, fibrillation, fuzz, pilling, swelling, weight loss*

#### 1. Introduction

Man-made cellulosic fibers and natural fibers are comfortable to wear, and have a stable share of the fiber market. Currently, the lyocell is the most efficient large-scale alternative to the viscose process. The cellulose is dissolved in a tertiary amine oxide N-methylmorpholine-N-oxide (NMMO), spun into an aqueous spinning bath, and the filaments are run through several after-treatments, giving the desired textile features [1-3]. Fabrics produced from lyocell fibers are breathable, moisture absorbent [4] and have excellent dimensional stability. Lyocell fibers, alone or in blends, are used widely in apparel and other fashion articles [5].

In the swollen state lyocell has an extensive fibrillation tendency owing to linear high crystalline fibrillar morphology [6,7]. The fibrillation tendency of lyocell enables this fiber to be used in specific finishing effects such as peach skin, silk touch and soft denim. On the other hand, the fibrillations induce e.g. rope marking defect in hank finishing, graying of dyed fabrics and a change of handle of clothes that spoils garments features. Efforts to control the fibrillation tendency in lyocell fibers include dyeing with reactive dyestuffs and treating fabrics with crosslinking agents [8,9]. Some of the most important steps in fiber processing involve alkalis, such sodium hydroxide or sodium carbonate, and some fiber response to alkali treatments is an important criterion [10,11]. Many reports on the

morphological structure of man-made cellulosic fibers and their treatment with crosslinking agents have been published [9,12]. However, few studies of the fibrillation tendency of man-made cellulose have been conducted in alkali solution under different conditions.

Furthermore, fibrillation may lead to pilling and therefore spoil fabric appearance and touch [13]. Pill formation is a common problem mainly in knitted fabrics made not only from synthetic fibers but also from natural fibers, man-made cellulose and their blends because no consumers accept the undesirably pilled garments. There have been many studies about pilling mechanism for knitted fabrics, which described influences of selected fiber properties e.g. tensile strength, elongation, bending rigidity, fiber count, shape of fiber cross-section and friction on the pilling phenomenon. Those models were, however, established for dry conditions but not for processes including wet condition e.g. laundry. Man-made cellulosic fibers are hygroscopic materials and their structures of fiber, yarn and fabric dramatically change by swelling with polar solvent such as water [14].

In the present study, a pilling mechanism including fibrillation and fuzz formation in dry and wet states is discussed and concepts to achieve high durable lyocell textiles against fibrillation and pilling are suggested.

## 2. Experimental

### 2.1. Materials

Modal, viscose and three varieties of lyocell (lyocell 1, lyocell 2, lyocell 3) fabrics (knit single jersey, woven fabric) were supplied from Lenzing AG, Austria and used for experiments. Five staple fibers of lyocell (lyocell 1, lyocell 2, lyocell 3), modal and viscose were also provided by Lenzing AG. The titer of the fibers was 1.3 dtex and the fiber length was 38 mm. The fibers lyocell 2 and lyocell 3 were wet-crosslinked with different agents while lyocell 1 was untreated. Analytical grade ethanol (EtOH, 96 %), lithium hydroxide mono hydrate (LiOH; > 99 %), sodium hydroxide (NaOH; > 98 %), potassium hydroxide (KOH; > 99%), trimethylammonium hydroxide (TMAH; aqueous 25 % sol.) and

other chemicals were purchased from Fluka. Plane woven fabrics of lyocell treated with crosslinker and softeners were supplied by BASF (Ludwigshafen, Germany). Crosslinking agent, catalyst, softening agent and wetting agent were also provided by BASF.

### 2.2. Treatments

#### Wash and dry treatments [13]

The knit fabric was cut into a square with area of 15 x 15 cm<sup>2</sup> and sewed on a cotton woven fabric. The sample fabric was washed with a domestic washing machine using the mixed detergent of fatty alcohol ethoxylate and secondary alkane sulfonate with liquor ratio of 1:20 at 40 °C for 30 min (W treatment). Then the wet fabric was dried with a tumble drier at 60 °C for 30 min (D treatment). The treatments of washing and drying (WD) were repeated 5, 10, 20 and 25 times. Pill rating of the fabric was assessed after the treatments according to a description.

#### Alkali treatment [15]

The lyocell fiber was treated with alkali as follows; 0.5 g fibers were immersed in the aqueous alkaline solution at certain concentration for 2 hrs at room temperature. After the fibers were rinsed with hot water at 60 °C for 5 minutes, neutralized with an acetate buffer containing 0.1 mol/l acetic acid and 0.1 mol/l sodium acetate (pH 5.0), and rinsed with water sufficiently, they were dried in an oven at 60 °C.

#### Crosslinking and softening treatments [16,17]

The lyocell woven fabric was immersed in a solution containing a given amount of crosslinking agent Fixapret<sup>®</sup> ECO which main component is 1,3-dimethylol-4,5-dihydroxyethylene urea, 15 g/l mixture of metal salts (Condensol<sup>®</sup> FB), 1 g/l wetting agent (Kieralon<sup>®</sup> TX 1563) and 0.5 g/l acetic acid (60 %w/w aq.). Excess solution was removed by passing through squeeze rolls to obtain wet pickups of 75 %w/w. After the fabric was dried

at 110 °C, it was subsequently cured at 175 °C (air temperature) for 60 seconds.

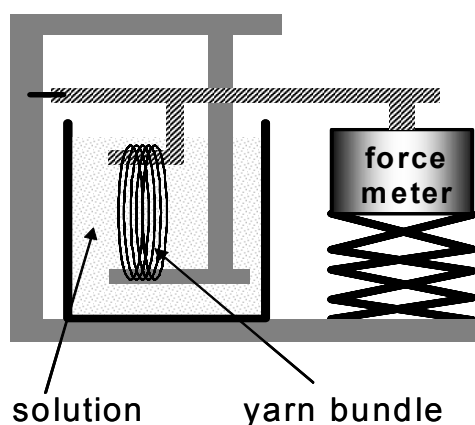
For softening treatment, a solution containing a given amount of softener Siligen<sup>®</sup> SIN which component is aminofunctional polysiloxane and 1 g/l acetic acid (60 %w/w aq.) was used. After excess solution was removed by using a vacuum filter or a centrifuge to obtain wet pickups of 100 %w/w, the fibers were dried at 60 °C.

### 2.3. Measurements

#### Fiber/fiber friction [18]

For the determination of interfiber friction an apparatus to determine the number of count (Zweigle D315) was used. A defined length of 50 mm of yarn was fixed between two clamps of the apparatus at defined pretension 2 cN. The number of reverse turns  $T_r$  required to open the yarn twist to the point of slipping is obtained as experimental result. The slipping experiments were performed at 20 °C and 65 % relative humidity. Each data point is the average from five measurements ( $T_{rdry}$ ). After the yarn was set between two clamps, 1 ml of distilled water was dropped on the yarn and  $T_r$  in wet state ( $T_{r\text{wet}}$ ) was measured by the same method for  $T_{rdry}$ .

#### Contraction force [18]



**Figure 1.** An apparatus for measurement of contraction force

The bundle of the yarn which length was 16 cm and consisting of 300 strings was mounted to the equipment shown in Figure 1 and the pretension of 300 g was given for 10 min. After the pretension was reduced to 50 g, the bundle was dipped into the distilled water solution for

15 min. The change of the contraction force was detected by a balance and recorded with a computer. The trials repeated four times to get a mean value.

#### Solvent retention capacity [19]

Fiber samples, 0.5 g in weight, were immersed in solution for 2 hrs at room temperature. The fibers were then centrifuged at 4000 G for 10 minutes and weighed ( $W_w$ ). The fibers were dried in an oven at 105 °C for 2 hrs and the weight was measured ( $W_d$ ). Solvent retention value in water (WRV) and in EtOH/water mixtures (ERV) were calculated by Equation 1.

$$\text{WRV(ERV)} = (W_w - W_d) / W_d \cdot D_{\text{alk.}} \quad (1)$$

Here,  $D_{\text{alk.}}$  is a density of the solution. The measurement was performed 4 times for each sample to obtain mean value.

#### Ball-bearings agitation [19]

A mass of fibers (0.5 g) was placed in a metal pot with 50 ml solution and 20 metal ball-bearings (0.5 cm diameter and 1 g weight). The pot was capped and tumbled end-over-end at 42 rpm and at fixed temperatures for 2 hrs. The fibers were then neutralized with a buffer solution containing 0.01 mol/l acetic acid and 0.01 mol/l sodium acetate (pH 5.0), rinsed with hot water at 50 °C and with cold water continuously. After the fibers were dried in an oven at 60°C for 1 hr, fibril numbers in fibers were counted on 0.38 mm segments using an optical microscope. Ten fibers from each sample were used to obtain mean values.

#### Weight loss [15]

Weight loss of the fiber during the alkali treatment was determined. 0.5 g of fibers was conditioned at 65 % relative humidity and 20 °C for 24 hrs and weighed ( $W_1$ ). The fiber was treated with the aqueous alkaline solution at certain concentrations by the same method for the alkali treatment. After the fiber was dried at 105 °C for 2 hrs, the fiber was conditioned at 65 % relative humidity and 20 °C for 24 hrs again

and weighed ( $W_2$ ). The weight loss (WL) was calculated by Equation 2.

$$WL = 100 \times (W_1 - W_2) / W_1 \quad (2)$$

**Scanning electron microscope [15]**

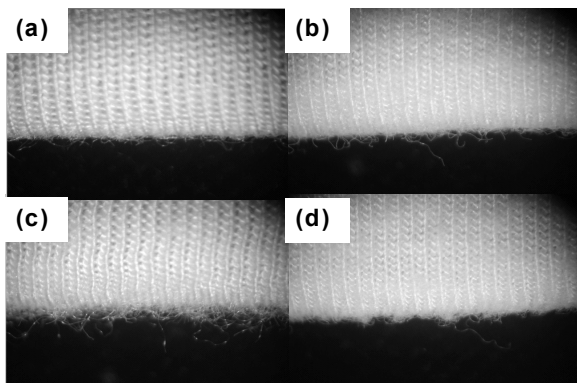
Images of the fiber cross sections were obtained using a scanning electron microscope (SEM; HITACHI S-2600H). The sample fiber after the pretreatment without fibrillation test was frozen in liquid nitrogen and broken apart by bending with hands.

**3. RESULTS AND DISCUSSION**

**3.1. Pilling mechanism**

Fuzz was observed on the fabric treated with only D, indicating that fuzz mainly forms in dry state. There are some pills neither on the fabric treated with W nor D, but on the fabric treated with WD. The individual treatment of W and D causes no pill formation while the combined treatment WD induces the pilling in the condition used for this work.

Photo images of the surface of knit fabrics after 25 times of WD, D and W treatment are shown in Figure 2.

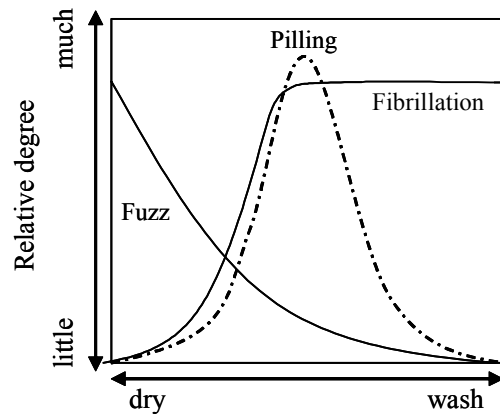


**Figure 2** Photographic image of knitted fabric (a), after 25 times of W (b), after 25 times of D (c) and after 25 times of WD (d)

On the contrary of fuzz formation, fibrillation appears on the fabric treated with W and WD. For further comprehension, the relation between fuzz, pilling, fibrillation, wash and dry treatments are schematically drawn in Figure 3. The y-axis is relative degrees of fuzz, pilling and fibrillation evaluated from Figure 2 and x-axis indicates the relative cycles of wash and dry treatments.

The degree of fuzz is relatively high after D and low after W. Contrarily, the degree of fibrillation is low after D and high after W and WD. There is no pill formation after D or W, and the pills appear only after WD. The pill formation is related to fibrillation more strongly than to fuzz formation.

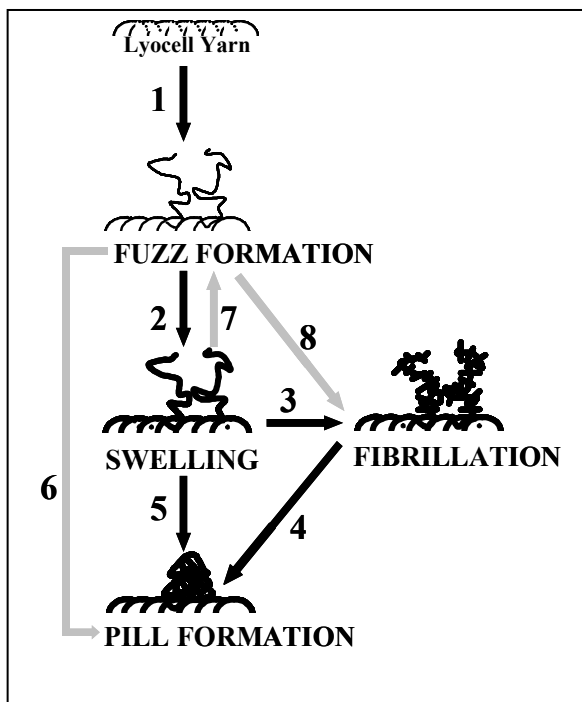
The critical number of count  $T_r$  where the fibers begin to slip in the yarn body and the applied force is sufficient to tear the yarn into two parts was obtained at  $F_R$  50 cN and summarized in Table 1. As the fiber length is equal, the higher values of  $T_{rdry}$  indicate increased fiber/fiber friction [18]. As compared to lyocell 1, lyocell 3 shows larger  $T_r$ , indicating high fiber/fiber friction. It is presumed that high fiber/fiber friction in dry state suppresses fuzz formation and consequently pill formation.



**Figure 3.** A schematic diagram of relationship among degrees of pilling, fuzz and fibrillation with wash and dry treatments.

**Table 1** Relationship among fiber/fiber friction, water retention capacity, contraction force and pilling.

Material	$T_{rdry}$ (counts)	$T_{rwt}$ (counts)	$T_{rwt}/T_{rdry}$	WRV ( $g \cdot g^{-1}$ )	Contraction force (g)	Pill rating
lyocell 1	30.5	>>	>>	-	175	-
lyocell 2	27.1	37.3	1.38	0.816	250	3
lyocell 3	34.2	37.8	1.11	0.799	230	4
modal	33.9	42.7	1.26	0.692	180	1
viscose	31.8	39.0	1.23	0.966	110	2



**Figure 4.** A schematic mechanism of pill formation during WD treatments. Arrows indicate the fuzz formation (1, 7), the swelling (2), the fibrillation (3, 7) and the pill formation (4, 5, 6, 8)

The fiber/fiber friction in wet state and contraction force are specific parameters that are expected to indicate the change of fiber properties by water uptake. The results are given as well as the values of water retention obtained by centrifugal method in Table 1.

Considering the fiber properties in wet and dry states as shown in Figure 3 and Table 1, a mechanism of pill formation including the fibrillation step was detailed. An illustration of the mechanism is given in Figure 4.

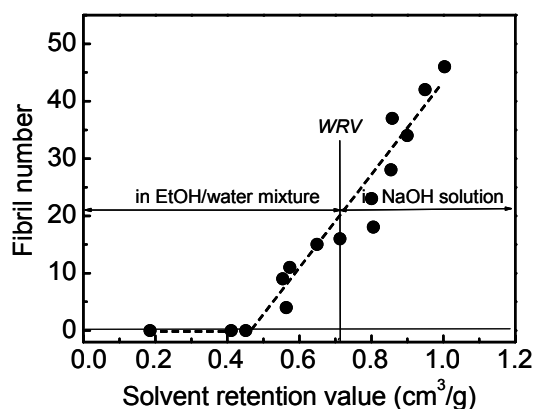
Firstly a fiber end comes out from the inside of a yarn by a mechanical abrasion during D treatment, which induces the fuzz (1). The fuzz fiber is swollen with W treatment and gets softer as shown in (2). The swollen soft fiber is easily fibrillated by mechanical abrasion during W and D treatments (3) and then tangled each other, which develops pilling (4). The fibrillation hardly occurs in dry state (9). Some swollen fuzz would lead to pilling without fibrillation as indicated in (5). The inducement of pill formation from fuzz is significantly hindered without wetting step as shown in Figure 2 (c) (6). Less degree of fuzz is formed when the fiber is swollen in wet state as shown in Figure 2 (b) (7). After a certain times of WD treatments, the fiber/fiber friction in dry gets

higher which results suppress of fuzz formation in dry state. Increase in fiber/fiber friction in dry state, decrease in degree of swelling might lower tendency of pill formation as well as fibrillation.

### 3.2. Fibrillation mechanism

As shown in Figure 4, the fibrillation plays an important role in pill formation that is significantly affected by fiber swelling. In order to clarify the effect of fiber swelling on not only pilling but also fibrillation, the fibrillation tendency in different degree of swelling was investigated. Figure 5 exhibits the plots of fibril numbers against the solvent retention values of fibre lyocell 1 in different solutions at room temperature.

The fibrillation of lyocell fiber occurs when the solvent retention value in EtOH/water mixture is higher than 0.45 cm<sup>3</sup>/g, and the fibril number is increased with increase in the retention value. The value 0.45 cm<sup>3</sup>/g characterizes a critical degree of swelling to obtain no fibrillation. The result implies that a lyocell fiber showing less than 0.45 cm<sup>3</sup>/g of water retention capacity should exhibit no fibrillation in water.



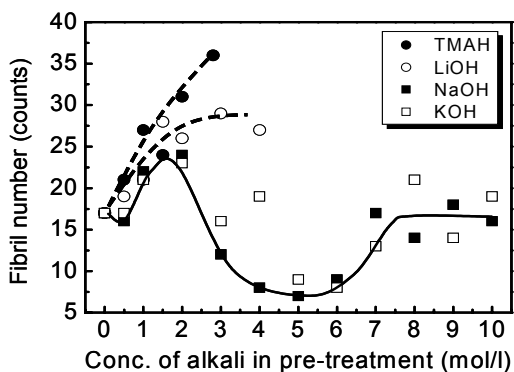
**Figure 5.** Fibril number as a function of solvent retention value of lyocell 1 at room temperature.

Alkali treatments are conventional process applied to textile such as mercerization, dyeing and resin-finishing. It is of great interests to study influence of alkali treatment on fibrillation tendency of lyocell

fibers. The influence of the alkaline concentration in the alkali treatment of the

The influence of the alkaline concentration in the alkali treatment of the lyocell on the fibril number ( $FN_{pre}$ ) was investigated. The results are shown in Figure 6. The lyocell fiber was treated with an alkali at a given concentration and then the fibrillation was induced in water by the method using metal balls with tumbling.

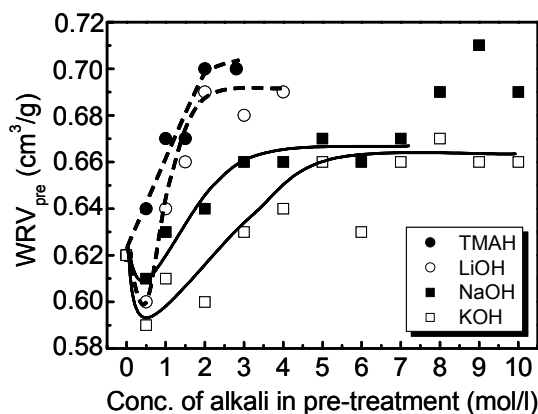
The  $FN_{pre}$  at 0 mol/l of alkaline concentration is the fibril number of the lyocell treated with water. As the fiber was treated with TMAH and LiOH, fibril number is increased with increasing the alkaline concentration. The  $FN_{pre}$  of the lyocell treated in NaOH sol. decreases, increases and decreases to 7 counts continuously with increasing the alkaline concentration to 5.0 mol/l. Further, the  $FN_{pre}$  increases again and reaches constant value at 7.0 mol/l. The curve of the  $FN_{pre}$  with KOH is comparable to that with NaOH.



**Figure 6** Plots of fibril number of lyocell fiber treated in alkaline solution against concentration of TMAH (■), LiOH (●), NaOH (▲) and KOH (▼)

The WRV in lyocell fiber treated with alkalis ( $WRV_{pre}$ ) was measured and plotted against the alkaline concentration in Figure 7. The  $WRV_{pre}$  with TMAH and LiOH increase up to approximately  $0.70 \text{ cm}^3/\text{g}$  with increasing concentration of alkaline solution in the range of concentration between 0.5 and 4.0 mol/l. The  $WRV_{pre}$  in fiber treated with NaOH solution increases to  $0.66 \text{ cm}^3/\text{g}$ , the value is unchanged at the concentration between 3.0 and 7.0 mol/l, and then increases again. For the pretreatment with KOH solution the  $WRV_{pre}$  increases to  $0.66 \text{ cm}^3/\text{g}$  at the concentration of 5.0 mol/l and the value is constant until the concentration increases to 10.0 mol/l. At concentration of 5.0

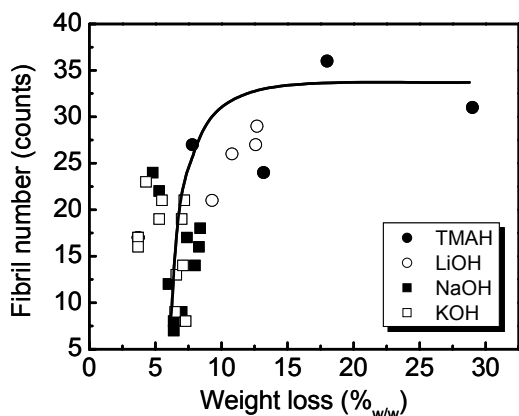
mol/l where the minimal  $FN_{pre}$  is obtained the  $WRV_{pre}$  with both NaOH and KOH is the equal value of  $0.66 \text{ cm}^3/\text{g}$  though alkali retention value in NaOH is larger than that in KOH [19]. The  $WRV_{pre}$  with NaOH at concentration between 3.0 and 7.0 mol/l where the  $FN_{pre}$  decreases is unchanged at  $0.66 \text{ cm}^3/\text{g}$ . These results suggest a consecutive structural change of the fibrils in NaOH solution at concentration between 3.0 and 7.0 mol/l. The change of fibril structure improves the fibrillation tendency of the lyocell fiber.



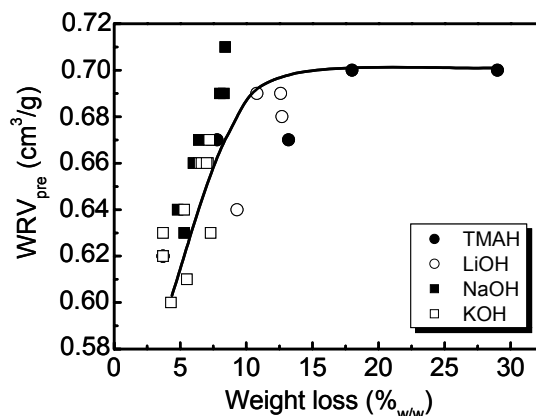
**Figure 7** Plots of water retention value of lyocell fiber treated in alkaline solution against concentration of TMAH (■), LiOH (●), NaOH (▲) and KOH (▼)

The weight loss is one of the important factors affecting the fiber property. The  $FN_{pre}$  and  $WRV_{pre}$  are plotted against weight loss in Figures 8 and 9.

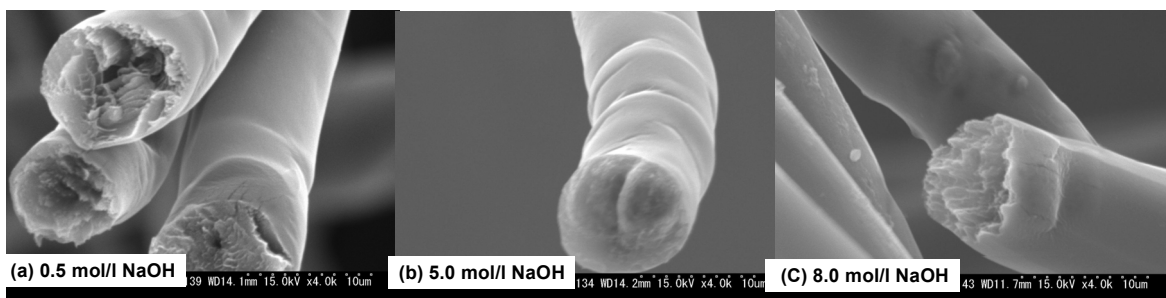
Both the  $FN_{pre}$  and the  $WRV_{pre}$  increase up to the weight loss of 15 %w/w and reach at constant value of 35 counts and  $0.70 \text{ cm}^3/\text{g}$ , respectively, regardless of the type of alkalis. Consequently,  $WRV_{pre}$  and weight loss of lyocell fiber treated with alkali are increased with increasing alkali concentration except in  $FN_{pre}$ , though the degrees of the increases are different among the alkali. The  $FN_{pre}$  of fiber treated with TMAH and LiOH increases with increasing alkaline concentration. Contrarily, the  $FN_{pre}$  is minimized in 5.0 mol/l NaOH and KOH solutions. The similarities in the relation of alkaline concentration,  $FN_{pre}$  and  $WRV_{pre}$  between in NaOH and in KOH solutions as shown in Figure 6 and 7 indicate that the mechanism of fibrillation after pretreatments in NaOH and KOH is similar but different from those in TMAH and LiOH.



**Figure 8.** Plots of fibril number of lyocell fiber treated in alkaline solution against weight loss of lyocell fiber treated with TMAH (■), LiOH (●), NaOH (▲) and KOH (▼)



**Figure 9.** Plots of water retention value of lyocell fiber treated in alkaline solution against weight loss of lyocell fiber treated with TMAH (■), LiOH (●), NaOH (▲) and KOH (▼)



**Figure 10.** SEM images of lyocell fiber treated with 0.5 mol/l (a), 5.0 mol/l (b) and 8.0 mol/l of NaOH (c)

In order to clarify the structural change during alkaline treatments, image analysis was performed using SEM. Figure 10 gives the images of cross section of fiber treated in NaOH solution.

The large bundles or layers of macrofibrils are clearly observed on the cross section of the fibers treated in 0.5 mol/l and 8 mol/l NaOH solutions. The fiber treated with aqueous solution containing 5.0 mol/l of NaOH shows a smooth cross section without any bundle and layer of macrofibrils. The fiber surface treated with 0.5 mol/l of NaOH is less rough than that treated with 8.0 mol/l of NaOH.

The less fibrillation was observed on the fiber with the smooth cross section without the bundle or the layers of macrofibrils. The formation of the bundle and the layers of the macrofibrils clearly elevate the fibrillation.

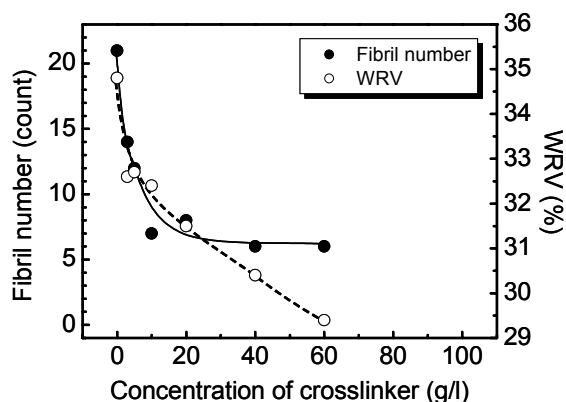
These results suggest that the treatments with alkalis cause the re-orientation of fibril structure and the fibrillation tendency is related to re-orientated microfibril structure. When the macrofibrils are re-orientated unequally, the bundles or the layers of the macrofibrils generate and the fiber shows high fibrillation tendency. Contrarily, the fibrillation is retarded if the uniform re-orientation of fibrils is induced by the treatments. In this study, the uniform reorganization of the macrofibrils was obtained by the alkaline treatment with 5.0 mol/l NaOH and KOH.

The chemical modification of fibrillar structure using crosslinking agent and additive polymers are of great interest regarding change in fibrillation resistance. Figure 11 and Figure 12 show the effect of concentration of crosslinking agent and softener on FN and WRV of lyocell fabric.

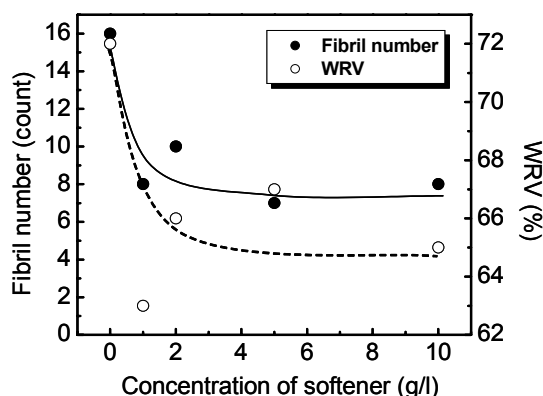
The FN and WRV considerably decrease by crosslinking with 10 g/l agent. The fibrils are crosslinked and the force among

fibrils is increased, resulting in lower degree of fibrillation.

The FN is strikingly decreased from 16 counts to 8 counts by adding 2 g/l softener. The result indicates that the fibrillation resistance is enhanced by the addition of aminofunctional polysiloxane and the addition of small amount of softener decreases 50 % fibrillation. The reduction in fibrillation is caused by coverage of fiber surface with the polymer, resulting in the decrement in fiber/fiber friction.



**Figure 11.** Plots of fibril number (●) and water retention value (○) of lyocell fabric treated with crosslinking agent against concentration of crosslinking agent



**Figure 12.** Plots of fibril number (●) and water retention value (○) of lyocell fiber treated with softener against concentration of softener

#### 4. CONCLUSIONS

The mechanism of pill formation including fibrillation process was proposed

taking into account the effects of consecutive wash and dry treatments,  $T_r$  corresponding to the fiber/fiber friction in dry and wet, the water retention capacity and contraction force indicating softness of the swollen fibers on degree of fibrillation, fuzz formation and pilling. The model suggests that the fibers come out from yarn by mechanical abrasion due to low fiber/fiber friction, they are fibrillated and tangled owing to the softness and high fiber/fiber friction in wet condition. The pilling was greatly accelerated by a combination of fuzz formation caused in dry state and fibrillation in wet state.

The fibrillation tendency is directly related to degree of swelling of fibers. The critical degree of swelling, that is the maximum retention capacity of fibers in the solution with no fibrillation, is  $0.45 \text{ cm}^3/\text{g}$  in ethanol/water mixture. The fibrillation is retarded by alkali treatment in 3.0-7.0 mol/l NaOH or KOH solution and minimized at 5.0 mol/l where SEM images shows smooth cross section with small and uniform macrofibrils of fiber. The weight loss of lyocell fiber enhanced fibrillation. The fibrillation resistance was enhanced by crosslinking with 1,3-dimethylol-4,5-dihydroxyethylene urea and by treatment with small amount of aminofunctional polysiloxane accompanying the decrement in water retention capacity of fibers. The fibrillation is inhibited by not only prevention of fibril separation but also modification of fiber surface, resulting in decrease in surface friction and water accessibility.

Consequently, the pilling of lyocell fabric maybe retarded by high fiber/fiber friction in dry state but low in wet state, low degree of fiber swelling and less extent of fibrillation caused by re-orientation of fibril structure, decrease in weight loss, enhancement of inter-fibril force and reduction in fiber surface friction.

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