Pilling in Cellulosic Fabrics, Part 2: A Study on Kinetics of Pilling in Alkali-Treated Lyocell Fabrics

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ABSTRACT: As pilling in textiles originates from many factors, the kinetic of pilling formation play an important role in the investigation and approaches of pilling. The single jersey-knitted lyocell fabrics were treated with different alkaline solution concentrations and submitted to Rapid Pilling Test—a wet-state Martindale test for cellulosic fabrics performed with increasing abrasion cycles. After each type of cycles, the pilling density was microscopically counted, and then pilling was visually rated. The changes in fiber properties were followed by water retention values (WRV), fibers swelling, fiber wet abrasion resistance (NSF), and fibers tenacity/elongation in wet and in dry states. The kinetics of pill formation—quantified by pills/cm^2—occurred in the following steps: pills are promptly formed at first abrasion cycles, reached the pill plateau cycles, and are self-removed from the fabric surface. The untreated and alkali-treated lyocell fabrics followed a similar trend of pill formation. However, the pilling propensity is distinct depending on the concentrations. The changes in the swollen state of fiber properties and fiber–fiber friction mainly determined the pill kinetics in lyocell fabrics. The kinetic model aims to figure out the pilling mechanism and the appropriate treatment for pilling resistance. © 2008 Wiley Periodicals, Inc. J Appl Polym Sci 109: 3696–3703, 2008

Key words: mechanical properties; modeling; polysaccharides; structure; fibers

INTRODUCTION

The pill formation in cellulosic fabrics is a complex combination of many chemical and mechanical effects as well as the inherent properties of each material. The fiber properties and their changes in the swollen state have great influence on the pilling mechanism. As lyocell fiber is prone to fibrillation because of the extension of cellulose chains and alignment of fibers axis during spinning process, the physical properties of lyocell fibers are significantly changed in the swollen state.[1,2] The high potential of fibrillation in lyocell fabric has intensive correlation with crystallinity and intrinsic viscosity that define mechanical resistance of fabrics, for example, the high-fiber tenacity but low lateral cohesion.[1] In lyocell fabrics, the pill formation in the wet state is defined by swelling and fibrillation together with high friction as suggested in the mechanism of pilling.[3] The massive fibrillation of lyocell fibers after wet abrasive treatment determines the pill structure and pilling growing rates.[4,5] Numerous studies have been done on predicting the pilling propensity,[6] modeling and computer simulation of pilling mechanism,[7,8] objective classification of pilling based on the wavelet transformation, and images analysis[9] in an attempt to characterize the pill formation in certain fabrics. The prediction of pilling propensity is an important factor for applying the suitable treatment and modifying the fabric final properties.

The pill formation was studied in alkali-treated lyocell fabrics. The caustic treatment has been applied for many years to change the swelling properties, crystallinity, and orientation of fibrils in cellulosic fibers.[10,11] These changes affect the physicomechanical properties of the material. The effects of alkaline treatment on fabrics depend on the alkali types and concentrations, physical state of material, and treatment parameters such as time and temperature. The high alkaline concentration is effective to change the fiber internal structure and rearrange the crystal and amorphous regions inside the fibers, whereas the low concentration is sufficient to improve the fibers surface and swelling behavior of fibers.[11] The sodium hydroxide concentrations (0.5, 1, 1.5, 2, 2.5, and 3*M*) were chosen based on the industrial mercerization process. In addition, cellu-

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losic fibers have limited swelling in aqueous solution as well as in nonaqueous media in the easily accessible regions or in the crystallinity regions.[12] At low concentrations, swelling increases with lye concentration, whereas after complete penetration of the whole structure, the decline in hydration number with increasing concentration is the decisive point leading to lower swelling values beyond the maximum.[12]

The kinetics model of pill formation in alkaline-treated lyocell fabrics is investigated with different sodium hydroxide concentrations to understand the pilling propensity as well as the motion and rate of pill formation in the fabrics, which may be helpful to further investigations of pilling and to optimize the suitable treatment for fabrics modification.

MATERIALS AND METHODS

Materials

Lyocell (CLY) single jersey-knitted fabrics—Tencel® Standard—were supplied by Lenzing AG (Lenzing, Austria). The fabric specifications are as follows: 50/1-Nm ring yarn, 1.3-dtex count and 39-mm length fibers, 140 g/m^2 specific weight, and 280 loops/cm^2 loop density. The fabrics were cut into 80 cm $\times$ 40 cm pieces and kept 24 h in standard atmosphere room (20 $\pm$ 2°C, RH 65%). The samples were immersed in different sodium hydroxide concentration solutions (0.5, 1, 1.5, 2, 2.5, and 3*M*) for 5 min. The analytical grade sodium hydroxide (>98%) was purchased from Riedel-de Haën. The wet samples were padded once at 3.5-bar nip pressure and 1 m/min in Mathis padder laboratory module. The padded fabrics were batched for 4 h at room temperature, rinsed with hot and cold running tap water, neutralized with commercial 5% citric acid solution, rinsed with water and line-dried overnight.

Methods

Rapid pilling test

The samples were cut in 140/170-mm diameter for upper/lower sample holders, immersed 1 h in 1000 mL of deionized water, padded at 3.0-bar nip pressure and 1 m/min speed. The padded samples were immediately placed in the Martindale tester (James. H. Heal and Co). The right sides of two pairs of samples were abraded with rotation 50, 100, 300, 500, 700, 900, and 1000 cycles under 250-g loading weight. The samples were rated in a viewing cabinet under daylight illumination from 1 to 5, where 1 indicates the highest pilling according to the ISO12945: Part 2-Modified Martindale Method. A mean of eight rating values from two observers was recorded for each type of sample.

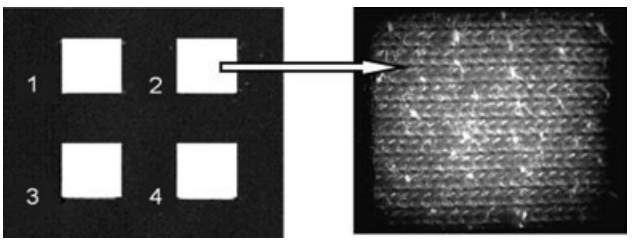

Figure 1 Photographic image of pill in 1 cm^2 derived from pill counting gauge.

Pill density counting

A gauge with 90-mm diameter for pill count was prepared in black carton with four areas of 1 cm^2 as referred in Figure 1. After each type of abrasion cycle, the upper holder samples were taken from Martindale abrasion tester and visualized with microscope (A. Krüss Optronic). The pill density was quantified by the number of pills in 1 cm^2, and four repetitions were done for each type of cycles. The obtained data of pill density (pills/cm^2) was analyzed by Table Curve 2D V5.0 software.

Water retention values

Dry pieces of 0.5 g of untreated and alkali-treated fabrics were immersed into 50 mL of deionized water for 24 h. The wet samples were centrifuged at 4000 $\times$ *g* for 10 min and weighted (W_w) with a mean value of four repetitions for each type of samples. The drying step was carried out at 105°C for 4 h and dry samples were weighted (W_d). The water retention values (WRV) were calculated as described in eq. (1).

$$\mathrm{WRV} = \frac{W_w - W_d}{W_d} \times 100\% \quad (1)$$

Fibers physical properties tests

For the series of tests to identify the fiber properties, the single fibers were untwisted from the yarns, which were unraveled following the wale direction in single weft-knitted untreated and alkali-treated samples. The performed tests are described later.

Fiber diameter measurement. The fibers were placed on a microscope slide, dropped with deionized water for 1–2 min to impulse the swelling and covered with a cover glass. The fiber diameter was obtained with mean value of 10 measurements from a Reichert Projection Microscope with 40/0.65 lens equipped with an illustration rule.

Wet abrasion resistance measurement. The fiber wet abrasion resistance (NSF) was measured using an abrasion tester (DELTA 100, Lenzing Technik Instruments). Single fibers attached to a clip with 50 mg

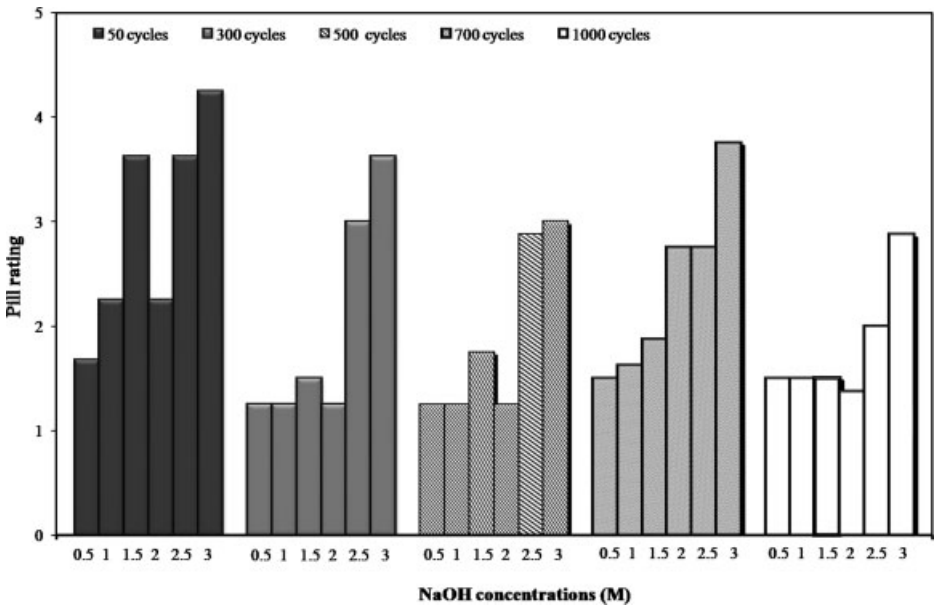

Figure 2 Effect of sodium hydroxide concentrations on pilling formation in lyocell fabrics with different wet abrasion cycles.

pretension were hanged on a frame (20 fibers per frame). An aluminum rough surface bar abraded the fiber by rotation in one direction at 100 rpm speed and DI water supply. The test gives the number of revolutions needed to break each fiber. A mean value from 60 fibers was recorded for each type of sample. Statistical analyses of results were conducted at a 0.05 level of significance.

Fiber tenacity and elongation. The tenacity/elongation of fibers was measured using Vibroskop integrated with Vibrodyn (Lenzing Technik Instruments) according to DIN 53816. A single fiber was individually attached with 70 mg pretension and hanged on the upper jaw. The lower jaw automatically clamped and strained the fiber at 100 mm/min speed. The initial distance between two jaws was 10 mm. The test was done in dry condition and in wet condition with DI water. The test provides the force necessary to break the fiber and the elongation at break. A mean value from 30 tests was recorded.

RESULTS AND DISCUSSION

Influence of alkaline treatment on pill formation

The pill formation in sodium hydroxide-treated fabrics after wet abrasive treatment was evaluated by visual pilling rating. The results are plotted in Figure 2.

The pill rates increased following the increase in the sodium hydroxide concentrations. The pill rates were lower when the abrasion cycles were higher. The fabrics were divided into two groups, which have similar rate and tendency of pill formation: fabrics treated with 0.5, 1, 1.5, and 2*M* sodium hydroxide and fabrics treated with 2.5 and 3*M* sodium hydroxide. The lower concentration treated fabrics group showed lower pilling rating (high pilling) and the higher concentration treated fabrics group showed the higher pilling rating (low pilling). The high concentration caustic treatment altered the fabric surface to brittle condition, which is analogous to the thermosetting of synthetic fibers. This setting effect can make the fabrics more lustrous and slippery to the surface–surface abrasion. The pill had the formation at first few cycles and reached the dynamic equilibrium level around 500 cycles with fabric treated with 0.5, 1, 1.5, and 2*M* sodium hydroxide. After the dynamic equilibrium level, the pill started to be removed from the fabrics surface and the pill rating slightly increased. The 2.5 and 3*M* sodium hydroxide reduced pilling tendency in fabrics. The alkaline treatment demonstrates the effectiveness on pilling resistance in lyocell fabrics. In general, the results showed that the concentrations of 1.5, 2.5, and 3*M* sodium hydroxide are useful for reducing pilling. However, due to the brittle surface of fabrics treated with the group of 2.5 and 3*M* sodium hydroxide, the fabric may not be comfortable for wearing and the 1.5*M* concentration may be preferred from the market point of view.

The dry-state starting point was at 1000 cycles where the water evaporation remarkably occurred and the fabrics were no longer in the wet state at the end of the pilling test. However, the formation rates and the influencing parameters such as treatment process and fiber properties are still demanding factors for investigation.

TABLE I
Physical Properties of Untreated and Alkali-Treated Lyocell Fibers and Fabrics

Lyocell fibers	Untreated	Treated with NaOH (*M*)					
		0.5	1.0	1.5	2.0	2.5	3.0
Wet tenacity (cN/tex)	29.3 ± 5.4	30.5 ± 6.3	30.0 ± 5.0	30.7 ± 6.6	30.9 ± 7.6	30.7 ± 5.1	29.0 ± 5.1
Dry tenacity (cN/tex)	41.8 ± 6.9	38.7 ± 4.5	36.9 ± 4.3	36.8 ± 4.4	35.8 ± 5.1	34.2 ± 6.6	31.8 ± 5.6
Wet elongation (%)	14.8 ± 6.5	14.4 ± 3.7	13.4 ± 1.3	13.0 ± 7.6	10.8 ± 2.4	11.8 ± 2.2	12.1 ± 4.0
Dry elongation (%)	11.4 ± 1.9	9.0 ± 1.5	8.4 ± 2.1	9.2 ± 1.9	9.2 ± 1.6	7.99 ± 1.7	6.7 ± 1.5
Fibers diameter (μ)	13.2 ± 1.0	14.2 ± 0.7	14.1 ± 1.5	14.3 ± 0.8	14.7 ± 0.9	14.8 ± 1.3	14.0 ± 1.6
WRV (g/g)	0.61 ± 0.018	0.68 ± 0.010	0.71 ± 0.008	0.73 ± 0.018	0.7 ± 0.014	0.72 ± 0.007	0.68 ± 0.014
Abrasion resistance ±95% CI (counts)	37.3 ± 8.7	66.06 ± 15	60.94 ± 10.5	71.45 ± 13.4	69.69 ± 14.1	68 ± 12	57.05 ± 10.3

Influence of fiber properties on pill formation

The pill formation can be attributed to a number of factors. The changes of fiber properties caused by alkaline treatment can noticeably change the physicomechanical properties of fibers, while being responsible for the differences in pill formation during the wet abrasive treatment. The physical properties of untreated and alkali-treated fibers are shown in Table I.

Because of the typical shape of fibers, the tensile properties—which response to applied forces and deformations—are one of the most important mechanical properties. The tensile strength is as well closely related to the pill formation under abrasive treatment. The lyocell fibers show good molecular orientation, high tensile strength, and little extensibility and stiffness due to their fibrillar structure compared to other cellulosic fibers.

In comparison with untreated lyocell fibers in dry state, the tenacity of alkali-treated fibers decreased from ~ 7.5 to 24% depending on the alkali concentrations as shown in Table I. At low concentrations study, sodium hydroxide decreased strength and modulus of lyocell fibers, but without changing the crystalline orientation significantly.[2] Along with an increasing concentration of the sodium hydroxide solution, a decrease of the amorphous orientation was recorded, whereas the crystalline orientation remains almost constant.[2] This could serve an explanation for the decrease in dry tenacity with higher sodium hydroxide concentrations.

In the wet state, lyocell fibers lose a certain portion of their tensile strength as fiber tenacity depends on their molecular orientation.[2] In comparison with untreated lyocell fibers in wet state, the tenacity of alkali-treated fibers are almost stable and only slight variation was found among the different alkali concentrations as shown in Table I. The wet tensile strength of lyocell fibers shows positive correlation with the amorphous orientation.[2]

Regarding to tenacity, the elongation at break of alkali-treated fibers decreased in both wet and dry state from ~ 20 to 40% with increasing concentration of sodium hydroxide. At high sodium hydroxide concentrations, the low elongation made the fibers more rigid, less flexible, and easy to be pulled out from the yarn and fabric body. In addition, the pills that were already formed could be easily removed from the fabrics surface by abrasion force. The increase in fiber volume was indicated by changes in fiber diameter and WRV. In aqueous alkaline solution, the swelling of cellulose fibers generally occurred swiftly, and the fibril architecture on morphological level can drastically change.[12]

A noticeable phenomenon recognized in the fiber tests was the limitation of changing in fiber properties. The WRV and the NSF reached the maximum value at 1.5*M* sodium hydroxide concentration as shown in Figure 3. The high NSF values indicated the less fibrillation tendency and high abrasive resistance.

The changes in physical properties of lyocell fibers in wet state caused by alkaline treatment resulted in the changes of pill formation. The loss of pill occurred only in the wet state, whereas there was no record of pill loss in the dry Martindale test up to high 5000 abrasion cycles.[5] The pill formation and

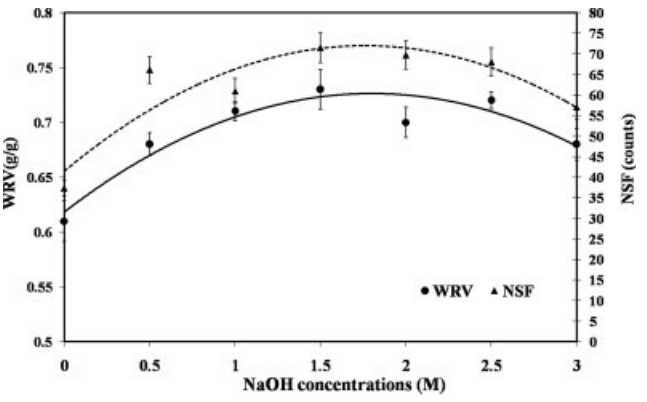

Figure 3 Effect of alkaline treatment on WRV and NSF of lyocell fabrics.

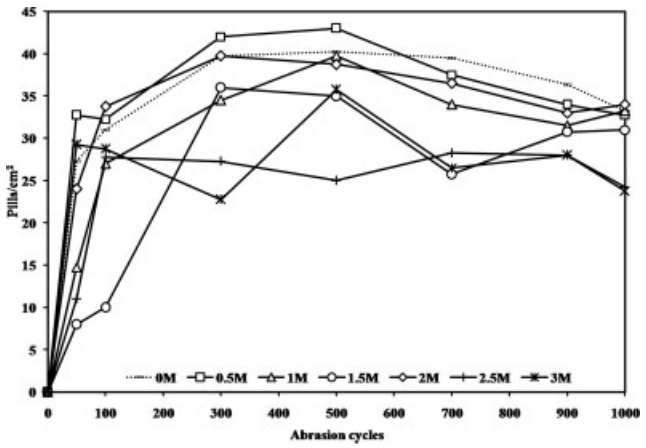

Figure 4 Pill density in alkali-treated lyocell fabrics.

pill loss proceeded at different levels depending on the sodium hydroxide concentration. However, the similar tendency of pill formation and pill loss after different concentrations alkaline treatment suggested that the kinetics of pill formation follow the same mechanism.

Kinetics of pill formation in alkali-treated lyocell fabrics

To understand how the pills form and depend on which parameter, the kinetics of pill formation is investigated here using a chemical kinetic equation. The rate equation for a chemical reaction is an equation that links the reaction rate with concentrations of reactants and constant parameters. To apply chemical kinetic equation on pilling, pill density—quantified by pill/cm^2—is considered as the ''concentration'' of pill formation reaction. The pill density results obtained by microscopy counting are plotted in Figure 4 showing that the pill density reduced with increasing sodium hydroxide concentrations. The plotting exhibited similar trends at different starting points. The fabrics treated with high alkali concentrations showed less variation in pill formation/pill loss than the ones treated with low alkali concentrations. In comparison with pill rating, pill density results displayed the medium level of correlation with the correlation coefficient of 0.57. It can be attributed by the effect of samples size and pill size. Pill rating is evaluated from 140/180-mm diameter samples, whereas pill density is counted in 1 cm^2 of samples. The size of the pills can be slightly differenced from untreated and alkali-treated fabrics due to the fibers swelling and the changes in fabric structure. However, pill rating and pill density followed the same trend, where high pilling rating (less pill density) are recorded in 1.5, 2.5, and 3*M* alkali-treated fabrics.

The kinetics model equation is suggested in eq. (2).

$$\mathrm{A} \xrightarrow[k_1]{} \mathrm{B} \xrightarrow[k_2]{} \mathrm{C} \qquad (2)$$

where,

Substrate A: virtual pilling propensity or the initial concentration of virtual pill in specific fabrics. The pilling propensity at the time $t = 0$ is A_0. The pilling propensities are calculated data from the software Table Curve 2D V5.0 when applying chemical kinetic equation with the input data are pilling density.

Substrate B: pilling formed during wet abrasive treatment counted at certain time (certain abrasion cycles). The formed pills are expressed by pilling density (pill/cm^2). This parameter is the experimentally measured value.

Product C: removal pills or pilling loss occurring during wet abrasive treatment and over the plateau point of pill formation, expressed by pills/cm^2.

The pill rating and pill density displayed in Figures 2 and 4 showed that the rate of pill formation is higher than the rate of pilling loss. Because of that, the first order intermediate equation, where $k_1 > k_2$, is chosen here and described in eq. (3).

$$\frac{dB}{dt} = y_B = \frac{A_0 \times k_1((\exp(-k_1 t) - \exp(-k_2 t))}{k_2 - k_1} \qquad (3)$$

where A + B + C = A_0.

The four key parameters are: (1) the given pilling or pilling propensity of the treated fabrics: A_0 (pills/cm^2); (2) the reaction rate of pill formation (b): k_1 (s); (3) the reaction rate of pill removal (c): k_2 (s); (4) the pills at the certain time (or certain cycles of abrasion) expressed by pill/cm^2 : dB/dt.

The proposed kinetic model is represented in Figure 5.

The numerical results of pilling kinetics model obtained from the software are listed in Table II, and the computed fit curves from model equation are

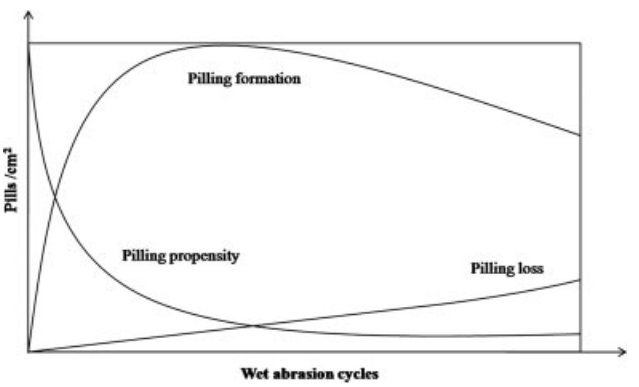

Figure 5 Kinetic model of A (pill propensity), B (pill formation), and C (pill loss) in alkali-treated lyocell fabrics.

TABLE II
Numeric Results of Pilling Kinetics Model

NaOH (*M*)	r^2	a (A_0) (pills/cm^2)	b (k_1) (10^{-3}) (s^{-1})	c (k_2) (10^{-3}) (s^{-1})	Ratio of k_1 to k_2
0	0.97	42.36	17.298	0.190	91
0.5	0.94	44.61	20.678	0.285	72
1.0	0.98	41.47	9.723	0.285	34
1.5	0.86	45.45	4.201	0.609	7
2.0	0.99	43.17	16.164	0.277	58
2.5	0.91	29.39	15.162	0.143	106
3.0	0.93	34.14	34.974	0.273	128

plotted in Figure 6. The r^2 values were higher than 0.9 showing the high correlation between the suggested model and the obtained results.

The numerical results displayed in Table II indicate that the concentrations of sodium hydroxide treatment affected the pilling propensity (A) and the pill formation rate (k_1) and pilling loss (k_2). The final appearance of pilling in lyocell fabrics after the treatment resulted from the equilibrium between three factors: A, k_1, and k_2.

The noteworthy influence of alkaline treatment was on the pill formation rate (k_1) and the pilling loss rate (k_2). The alkaline treatment retarded k_1 and slightly intensified k_2. However, this is valid only for fabrics treated with 0.5, 1, and 1.5*M* sodium hydroxide. Contrasting, the fabrics treated with 2 and 2.5*M* sodium hydroxide have similar k_1, and the fabrics treated with 3*M* sodium hydroxide had a twofold increasing k_1 when compared with untreated fabrics.

The relation between k_1 and k_2 is shown in Table II. Although in general $k_1 > k_2$, the ratio between k_1 and k_2 decreased following the increasing of sodium hydroxide concentration until 1.5*M*. At 2*M* sodium hydroxide, the significant difference between k_1 and k_2 increased furthermore and reached the highest ratio value at 3*M* sodium hydroxide ($k_1/k_2 = 128$) because of the steady k_2 and the increasing k_1.

The reactions rates (k_1 and k_2) relation plotted in Figure 7 defined the final results of pilling rating in alkali-treated lyocell fabrics. Figure 7 indicates that at 1.5*M* sodium hydroxide, the pill formation rate is the lowest and the pill removal rate is the highest,

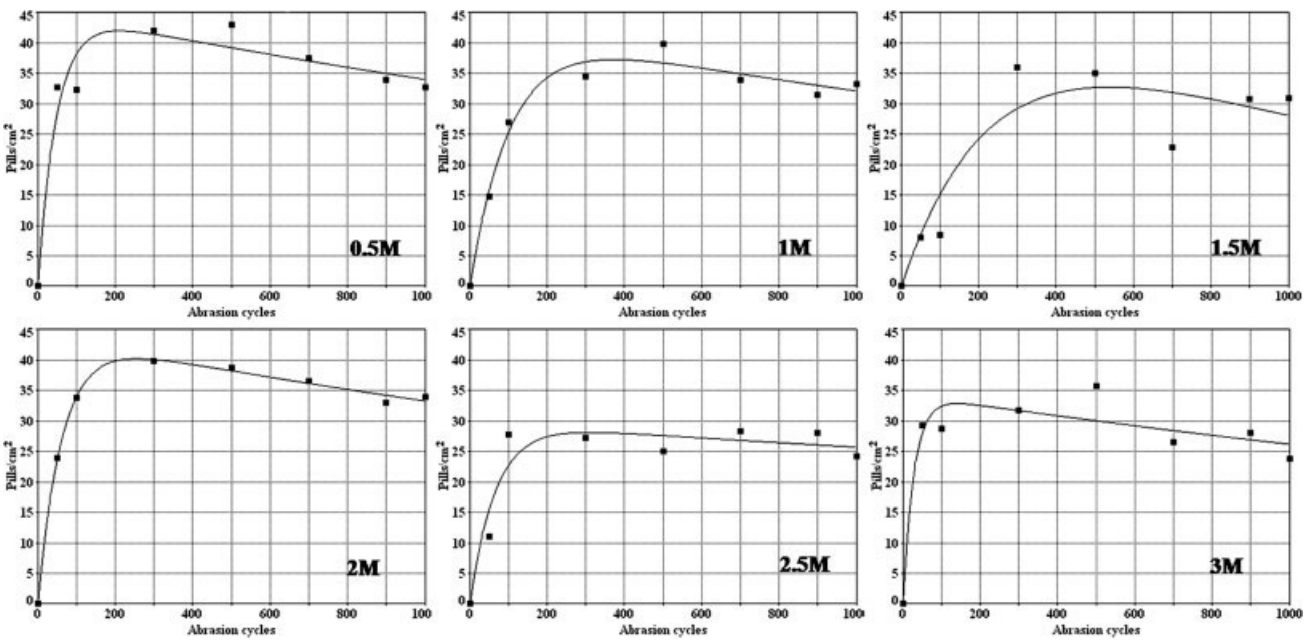

Figure 6 The computed curves from model equation of pilling kinetics in alkali-treated lyocell fabrics with different sodium hydroxide concentrations.

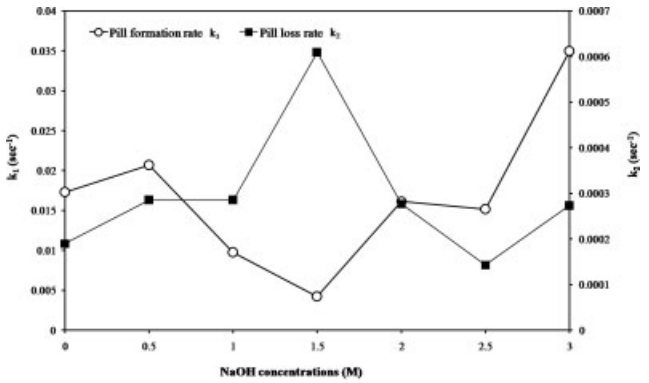

Figure 7 The relation between the pill formation rate (k_1) and pill loss rate (k_2) with sodium hydroxide concentrations.

leading to the self-cleaning phenomenon. The lowest ratio of k_1 and k_2 is responsible for the less pilling existence in the fabrics. The rates k_1 and k_2 showed the correlation with the fibers character in different sodium hydroxide concentrations treatments. When the NSF and WRV increased, the pill formation rate k_1 decreased and the pill removal rate k_2 increased. The highest NSF and WRV obtained for fabrics treated with 1.5*M* sodium hydroxide illustrate the critical rates of pill formation and pill loss. The pilling propensity (A) was clearly reduced when sodium hydroxide concentrations reached 2*M*. Therefore, the final pill rates of fabrics were higher (less pilling) when treated with sodium hydroxide 2.5 and 3*M*, although k_1 increased.

According to the kinetics model, there are three concentration values where the pilling is reduced on fabric. For fabrics treated with 1.5*M* sodium hydroxide, the pill formation rate was retarded approaching the pilling loss rate. This concentration was the most effective to improve the NSF and WRV. The NSF and WRV are important fiber properties in determining fabrics behavior when submitted to wet abrasive treatment. The high WRV indicates fiber reorganization and the high NSF may attribute to the fibers force to resist fibrillation tendency. As fiber fibrillation is one of the major factors in pilling mechanism, the higher fibrillation resistance can retard the formation rate k_1. In the case of fabrics treated with 2.5 and 3*M* sodium hydroxide, although the ratios between k_1 and k_2 are higher, the lower pilling propensity can be identified as the reason for lower pill formation in fabric surface.

The schematic diagram of relation between degree of pilling propensity (A_0), pill formation rate (k_1), and pilling loss rate (k_2) in alkali-treated lyocell fabric is shown in Figure 8, illustrating the relations among A_0, k_1, and k_2 for each situation of pill formation.

Figure 8(a) corresponds to untreated and 0.5*M* sodium hydroxide-treated lyocell fabrics, Figure 8(b) corresponds to 1, 1.5, and 2*M* sodium hydroxide-treated fabrics, and Figure 8(c) corresponds to 2.5 and 3*M* sodium hydroxide-treated fabrics. The variation of the three key parameters k_1, k_2, and A_0 depending on treatment concentrations is clearly defined the development of pilling in alkali-treated lyocell fabrics.

CONCLUSIONS

A study on the kinetics of pill formation was carried out to understand the pilling propensity and pill formation rates in alkali-treated lyocell fabrics. The swelling of fibers in alkali leads to a rearrangement and modification of fibers in yarn and fabrics. In lyocell fabrics, the two-sided effect of high fibrillation and changing of physical properties in the swollen

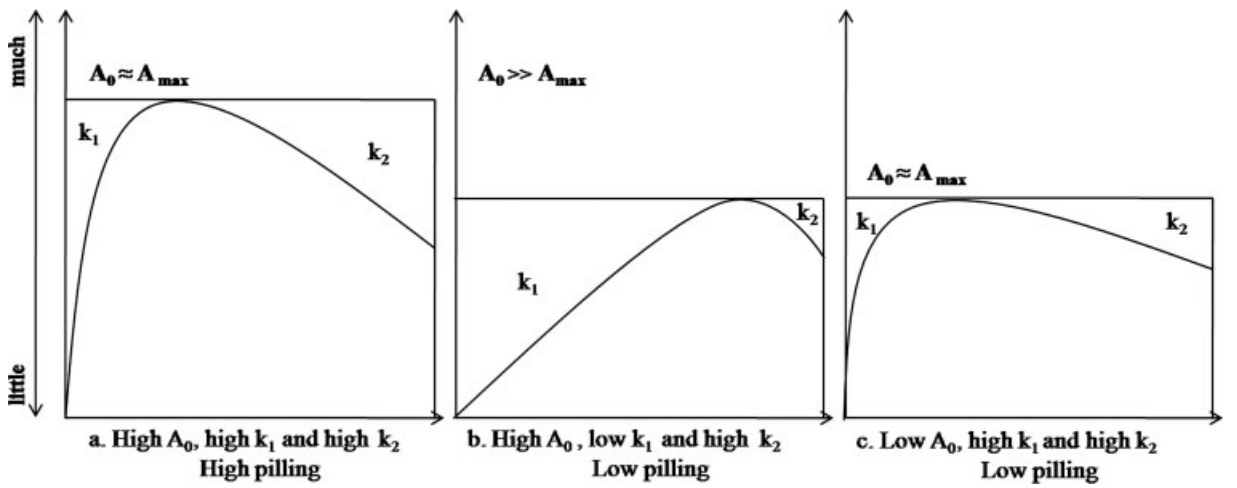

Figure 8 A schematic diagram of relation between degrees of A_0, k_1, and k_2 in alkali-treated lyocell fabrics.

state on the fabric quality is a challenge. The sodium hydroxide treatment on lyocell fabrics was expected to improve the pilling resistance and experimental results showed the actual benefit. The different concentrations of sodium hydroxide caused the specific changes in fibers properties, even at small intervals of alkali concentrations. The weaker tenacity in swollen state resulted in the fiber breakage and faster pill removal from fabrics surface. With gradual increase in sodium hydroxide concentrations during alkaline treatment, the critical minimum concentrations for improvement of pilling could be identified. However, this value will be dependent on the characteristic of cellulosic fibers and yarn and fabric structure. When the formation rate and removal rate of pilling grow correspondingly, the self-cleaning effect of pilling occurs and the appearance of the fabric surface improves. This improvement was clearly recorded in lyocell fabrics treated with 1.5*M* sodium hydroxide—the most effective concentrations to improve the fibers physical properties, which are strongly related to the pill formation. In consequence of low pilling propensity, fabrics treated with 2.5 and 3*M* sodium hydroxide exhibited low pilling. However, the brittle surface and the stiffer touch may cause inconvenience to customers. The relation between slow formation rate, fast removal rate, and low propensity of pilling resulted in the final pilling existence in the lyocell fabric surface. The simple model could be used to fit the experimental results with high correlation. The kinetics model proved that the suggested kinetics equation is the appropriate tool to understand the pilling mechanism in alkali-treated lyocell fabrics. The kinetics model can be used to track down the suitable alkaline concentration to improve the fabrics properties. The pilling kinetics study will be extended to other types of man-made cellulosic materials and to establish a useful tool for systematic investigations on the pill formation in fabrics.

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