Pilling in Man-Made Cellulosic Fabrics, Part 1: Assessment of Pilling Formation Methods*

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ABSTRACT: Despite the pills appear similarly in fabric surfaces, their structure and mechanism of formation are distinct depending on material categories and handling conditions. The lyocell and viscose knitted fabrics were abraded with short cycles in the wet state as an assessment method for pilling formation called rapid pilling test (RPT). The samples were immersed in different wetting agents and subsequently padded before short abrasion in Martindale tester. The results were compared with long abrasion, besides 5–25 cycles of washing and drying (W-D). The samples were rated and these results were correlated with the changes in the physical parameters obtained in the dry and wet state. The correlation showed the feasibility of RPT in wet state with short-abrasion cycles, in lieu of long cycles and W–D cycles. Furthermore, the image analysis of single pills formed by different methods and the inner/outer pill structure may reflect the pilling mechanism and yield a comprehensive view of the whole process. Instead of conventional Martindale test, the easy-handling RPT can be applied for cellulosic fabrics, in which wet samples are abraded in short cycles fulfilling two important demanding factors during test performance: test of material in the wet state and reduced testing time.

Key words: hydrophilic polymer; mechanical properties; structure; swelling; fibers

INTRODUCTION

Pilling is defined as a surface flaw occurring in almost all fabrics. This undesirable phenomenon spoils fabric estheticism and reduces fabric service ability. Pilling is examined on many kinds of materials and became a debatable topic in man-made cellulosic fabrics as the fiber characters considerably vary in swollen state. The man-made cellulosic materials are widely used and fit the eco-friendly criteria. Among them, lyocell and viscose are typical representatives for the NMMO and xanthogenate fibers manufacturing process, respectively. Pilling has been induced by many tests and classified by many innovated methods using automated and informative technology such as wavelet transform and images analysis.[1–4] At present, the fabric pilling resistance is generally tested in laboratory by methods that simulate the wearing and using process.[4] Because of the fact that different pilling testers have different sensitivities for various fibers, yarn, and fabric parameters, using at least two pilling testers are recommendable to obtain reliable results.[4] To appraise the pill in cellulosic fabrics, the washing and drying (W–D) test is commonly used together with Martindale test (International Standard ISO 12945-Part 2).[5] The Martindale test runs in dry state with long-abrasion cycles (2000–7000 cycles, 40–140 min) and loading weight for knitted and woven fabrics. The test is effective to synthetic and woolen textile materials and normally gives the higher pilling in fabric surface due to the high abrasion when compared with other methods.[4,5] The Martindale is the adequate test to evaluate the pilling formation in fabric surface. However, the test may not reflect the pilling essence and the tendency related with the variation of fiber properties in wet state. The W–D test is performed by repeated long cycles of W–D. However, it demands high running time including the off-period (1–3 days), and it is difficult to operate in meticulous procedures with detergent and temperature control.[6,7] To cover the wet state in Martindale test and to perform one test instead of two parallel ones, as suggested for valuable results, we propose here a rapid pilling test (RPT) for man-made cellulosic-knitted fabrics.

The RPT consists of two basic steps. First, the samples are wetted, replacing the conventional dry samples. Then, the wet samples are abraded with short cycles of 50–500 (1–10 min) in lieu of long

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TABLE I
The Fabrics Specifications

Specifications	CLY	CV
Yarn count (Nm)	50/1	68/1
Fiber count (dtex)	1.3	1.3
Fibre length (mm)	39	39
Specific weight (g/m^2)	140	148
Yarn type	Ring	Ring
Knitting type	Single Jersey	Single Jersey
Loop density (loop/10 cm^2)	196	324

cycles (40–140 min) as recommended in the Martindale method. The measurement of changes in physical properties of fibers such as swelling diameter, strength, friction, and liquid retention value, and their correlation with pilling rates aims to establish the feasibility of the proposed RPT. The image analysis of single pills formed by different methods and the inner/outer pill structure may reflect the pill formation mechanism and give a comprehensive view of the whole process. In addition, RPT has been applied for our further investigation into a study on kinetic of pill formation in lyocell fabrics after different treatment.

EXPERIMENTAL

Materials

Fabrics

Tencel® Standard (CLY) and Lenzing Viscose® (CV) knitted fabrics supplied by Lenzing AG were used in this investigation. The material specifications are described in Table I.

Wetting solutions

Solution (2 mL/L) of commercial detergent from Persil-Color-Gel from Henkel Austria GmbH (CD) was used. The commercial available grade glycerine 86.5% (K. Deuring GmbH) was mixed with deionized water in the volume ratio of 10 to 1 to obtain glycerine/water wetting solution (GW). Deionized water was also used as a wetting solution (DI).

METHODS

Fiber physical character measurements

To perform the fiber tests described below, each single fiber was withdrawn from a bundle of fibers untwisted from the yarn. The yarn was unraveled following the wale direction in single weft-knitted samples.

Fiber diameter measurement

The fibers were placed on a microscope slide and allowed to swell in different solutions (immersion oil, glycerin, GW, CD, and DI water), and the swollen fibers were covered with a cover glass. As the equilibrium swelling of cellulosic fibers is attained within 1–2 min,[8] measurement was taken at 2-min swollen fibers. The fiber diameter was obtained with a mean value from 10 results measured at a Reichert projection microscope with 40/0.65 lens equipped with illustration rule.

Fiber wet abrasion resistance measurement (NSF values)

Abrasion resistance of fibers in wet state was measured using an abrasion tester (DELTA 100, Lenzing Technik Instruments). A single fiber was individually attached with 50 mg pretension and hanged on the sample-holding frame (20 fibers/frame) prior to place in the tester. An aluminum rough surface bar attached to the tester abraded the fiber by rotation in one direction at 100 rpm and DI water supply. The rotation numbers necessary to tear the fiber were recorded. A mean value from 60 fibers was taken for each type of samples. Statistical analyses of results were conducted at a 0.05 level of significance.

Fiber tenacity measurement

The tenacity and elongation of fibers were 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. The initial distance between two jaws was 10 mm. The test was individually done both in dry state and in wet state with DI water. The force necessary to break the fiber and the elongation at break was recorded. A mean value from 30 tests was recorded.

Pill inducement methods

Washing and Drying

The fabrics were cut into the pieces of 300 × 500 mm. The washing and drying (W–D) tests were performed using a Miele T5212 washing machine and a Miele Electronic WS 5405 drying machine. The liquor ratio was 1 : 20 (material/washing liquid) with commercial detergent. The washing condition was 30 min at 40°C, easy-care mode, without prewashing, with three rinses and one centrifugation. The drying condition was 30 min at 60°C in extra dry mode. The repetitive test was performed with 5, 10, 15, 20, and 25 W–D cycles.

Martindale test in dry state

The fabrics were cut in 140 of 170-mm diameter for upper/lower sample holders. The samples were

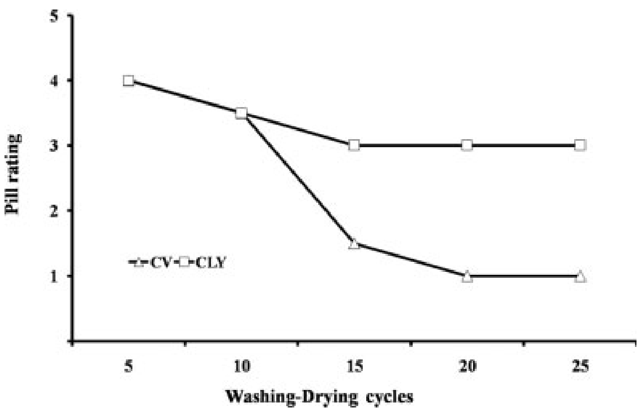


Figure 1 Effect of W-D cycles on pill formation in CLY and CV fabrics.

kept in a standard atmosphere room (20°C ± 2°C, RH 65%) for 24 h and weighed. The right sides of the fabrics were abraded under a normal load of 250 g. Short-abrasion cycles (50, 200, and 500) and long-abrasion cycles (1000 and 5000) were applied with four repetitions for each type of cycles.

Martindale test in wet state

The samples were cut as for dry-state Martindale, kept in a standard atmosphere room for 24 h and immersed in 1000 mL of each different wetting solvents (DI water, CD, GW) for 1 h, padded at 3.0-bar nip pressure and 1 m/min. The padded samples were weighed and immediately placed in the Martindale-abrasion tester. The right sides of the samples were abraded under a normal load of 250 g. Short-abrasion cycles (50, 200, and 500) and long-abrasion cycles (1000 and 5000) were applied with four repetitions for each cycle type. The samples were gently washed to remove wetting solvents and flat dried overnight.

Pilling visual rate

The samples were rated by visual estimation according to the International Standard ISO12945 Part 2: Modified Martindale Method. The samples were scrutinized in a viewing cabinet under day light illumination, rated from one to five, where one indicates the highest level of pill density. Two observers rated all samples once, where samples of each cycle type had four repetitions. Consequently, a mean value from eight rates was recorded for each type of cycles.

Microscope and SEM images observation

Pill appearance in the fabric surface

A Microscope (A. Krüss) attached with a digital camera Canon Power Shot S40 was used. The obtained images are classified based on pilling appearance encountered in the surface compared to the blank samples. One image per sample was recorded.

Pill shape and structure

A single pill was cut off from each sample and placed between a microscope slide and a cover glass. The pill images are classified based on the pill structure after different treatment conditions and/or the absence of fibrillation in the formed pill. A Zeiss Microscope AXIO Imager.D1 with lens Plan-Apochromat 10 × 0.45 PH1 and Body-Camera Canon EOS 20D was used.

SEM analysis of the fiber and pill

Phillips XL30 ESEM-FEG was used to scan the fabrics surfaces and fiber fibrillation inside the pills. The magnification powers were 100×, 350×, 1000×, 3500×, and 10,000× for observation, and images of 100× and 3500× were recorded.

RESULTS AND DISCUSSION

Assessment of conventional pilling inducement methods

The pill rates obtained at conventional pilling inducement methods were compared. Figure 1 shows the pilling rates of CLY and CV fabrics obtained after repetitive W–D, and Figure 2 shows pilling rates of dry-state Martindale test (ISO 12945). The number of ascending W–D cycles and abrasion cycles applied to the samples expresses the intensiveness of pill-inducing methods.

Pilling formation after W–D method showed the plateau cycles in the pill rating at 15 W–D cycles for CLY fabrics. However, the pilling of CV fabrics promptly reduced to the low rates at 10 W–D cycles.

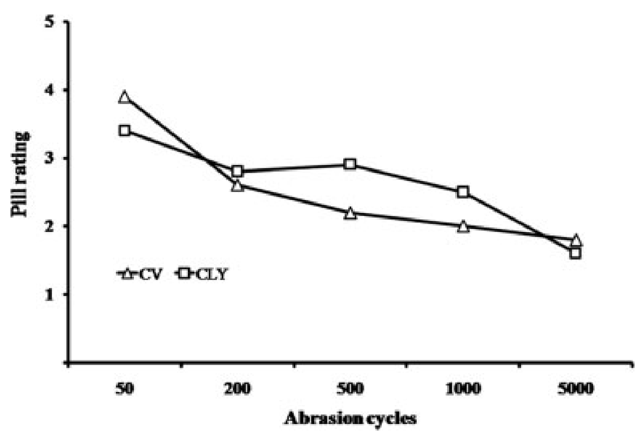


Figure 2 Effect of Martindale dry-abrasion cycles on pill formation in CLY and CV fabrics.

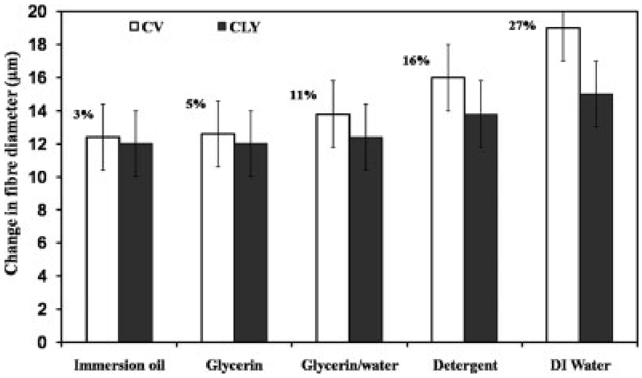


Figure 3 Fiber swelling expressed by changes in diameter in CLY and CV fabrics and the difference of swelling for CV and CLY fibers expressed in percentage.

The same tendency is recognized for the pilling with Martindale-abrasion tests with the plateau point of 500 abrasion cycles. The higher abrasion intensiveness causes lower pill rating for the samples in dry-state Martindale. These tests showed similar tendency of results. However, the W–D drying test required 2–3 days testing to reach 25 cycles, and the Martindale test excludes the wet condition, which covers the changes of cellulosic fibers in swollen state.

Assessment of fiber changes in swollen state

The changes of fibers in swollen state are investigated here with three types of wetting solvents: glycerine/water mixture (GW), detergent solution (CD), and deionized water (DI water). The glycerine/water mixture was considered as non-drying liquid to lubricate the fibers for friction prevention, and the detergent solution (CD) was used to approach the W–D process. As a pill is mainly formed from fiber entanglement, the fiber swelling, fiber tenacity/elongation, and fiber wet-abrasion resistance in selected solvents were characterized. In general, the cellulosic fiber swelling in swollen state results in interlaced structures of both knitted and woven fabrics.[9] The swelling rate as well as liquid regain rate varying between different solutions could affect the swelling extent.[10]

Figure 3 shows the fiber expansion expressed in diameter. The immersion oil was used as standard nonswelling liquid. The glycerine/water mixture had negligible effect on fiber swelling, which kept the fibers near to the dry state. The CV fibers were swollen more than CLY fibers (3–27%) in the same wetting solutions. The swelling tendency indicates that CV fabrics can be easily wetted for wet-state abrasion test.

Table II describes the changes in fiber tenacity and elongation from dry to swollen state in water. The tensile stress of fiber was highly reduced in swollen state. However, CLY fibers showed higher tenacity than CV in the same conditions. On the contrary to tenacity, the elongation of fibers in swollen state is greater than in dry state, particularly with CV fibers. The fibers have greater deformation in the load direction in wet state. When loaded, the CLY fibers could undergo higher tensile stress but less deform compared to CV fibers, which correspond to the stress resistance. CV fibers could only be torn at 150 counts, whereas CLY fibers breakage occurred at 33 counts.

The physical characters of each type of cellulosic fiber have influence on the formation and the existence of pilling. In CV fabrics, the lower tenacity and higher swelling caused the faster pilling formation. The higher elongation and higher wet-abrasion resistance kept the pills to stay longer in the fabric surface.

Rapid pilling test

In rapid pilling test (RPT), the Martindale tester abraded the wetted samples with 250 g loading weight and increasing abrasion cycles. To keep the wet conditions during wet-state Martindale, the wet pick up and mass loss of samples were scrutinized.

The higher wet pick-up rates of CV fabrics (148% for GW, 94% for DI water, and 87% for CD) compared to CLY fabrics (125% for GW, 83% for DI water, and 80% for CD) can be explained by the physical properties of the single fiber, in which CV presents higher void volume, bigger internal surface, and bigger average pore size.[11] CV fibers showed a wide pore size (distribution 5 nm–1µm), whereas CLY fibers showed a more homogeneous distribution with a slight gradient in pore density of 20 nm.[12] In glycerin/water solvent, although the fibers were slightly swollen, both CV and CLY fibers showed very high wet pickup (over 100%) due to the high viscosity and molecular weight of glycerin.

Figure 4 displayed the mass loss, where the initial weight is the mass of samples immediately after padding. The mass loss of samples during wet-state Martindale was increased following the increasing

TABLE II
Physical Characters of Cellulosic Fibres in Dry and Wet States with Deionized Water

		CV	CLY
Tenacity in dry state	cN/tex	21.07	41.84
Tenacity in wet state	cN/tex	14.73	25.63
Elongation in dry state	%	11.25	11.35
Elongation in wet state	%	23.80	14.81
Wet abrasion resistance (NSF) ± 95% CI	Counts	150.1 ± 20.8	32.94 ± 8.43

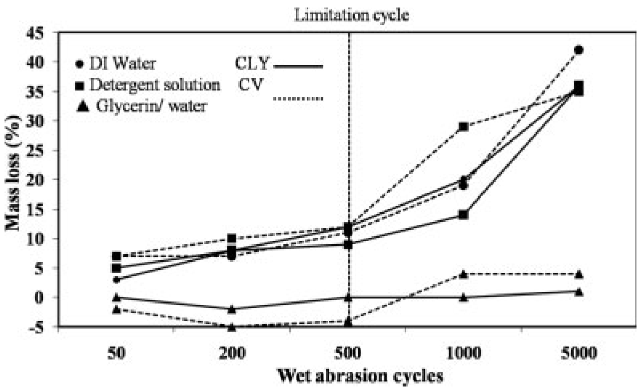


Figure 4 The mass loss of CV fabrics (dashed line) and CLY fabrics (solid line) during wet state Martindale with different solvents.

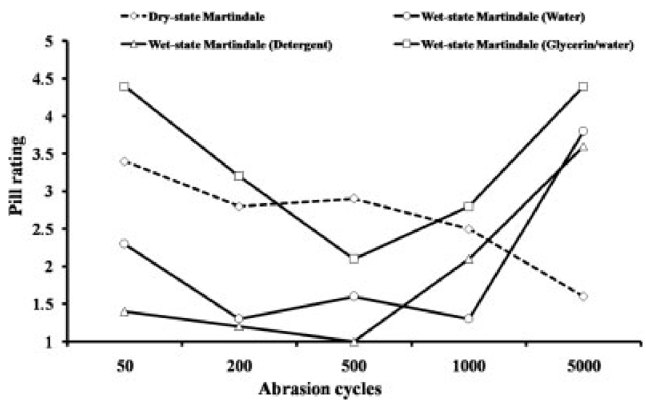


Figure 5 Effect of Martindale-abrasion cycles on pill formation in CLY fabrics.

number of abrasion cycles with remarkable increase at 500 wet-abrasion cycles. An exception is recorded for the fabrics treated with glycerin/water mixture. During wet abrasion, the liquid evaporation and fibers loss caused the mass loss. Those combinations of factors defined the differences in the mass loss for different materials and wetting solutions. At short-abrasion cycles, the fibers loss was insignificant and the mass loss mainly resulted from liquid evaporation. Figure 4 showed that the variation in mass loss of all samples was minor during Martindale test up to 500 cycles. In comparison with the initial wet weight of samples padded with all GW, DI water, and CD solution, the samples weight after 50, 200, and 500 abrasion was stable. The unchanged weight of abraded samples indicated that fabrics remained in wet state until 500 cycles.

It can be stated that the long-abrasion cycles could be inadequate for wet-state Martindale using swelling liquid. After 500 abrasion cycles of wet-state Martindale with swelling liquids, the weight loss reached 20% from the initial weight where samples are no longer considered to be in fully wetted state. The exceptional case is wet-state Martindale with glycerin/water mixture (considered as nonswelling liquid), where samples remained in wet condition until 5000 cycles.

To demonstrate the effectiveness of wet-state Martindale with short cycles, the pill rating results are plotted in Figure 5 for CLY fabrics and in Figure 6 for CV fabrics. The plotting includes pilling rates from the dry-state Martindale for comparison.

In this investigation, the outstanding difference between wet-state and dry-state Martindale is noticed. The results obtained in wet-state Martindale followed a change in trend in the pill rating from 500 abrasion cycles. The pill rating rose from 1.5 to 4.5 for CLY and from 1 to 4 for CV due to the fact that the pill density reduced after 500 abrasion cycles, although the pills kept forming continuously after 500 abrasion cycles. At this point, the pills were detached from the sample surfaces and 500 abrasion cycles is the bound where the pill removal starts. During abrasive treatment, the formation and existence of pills in cellulosic fabrics are noticeably influenced by abrasion force. The high–wet-abrasion force pulls fibers from yarn to form fuzz and the fuzzed fibers entangle to form pills. The consecutive high friction force in wet state tore the fibers and abraded the pills from fabrics surface causing the pill removal.

The relation between pilling propensity and fiber characters in swollen state is defined by the influence of fiber swelling. Resulting from higher fiber swelling, the pill in CV and CLY fabrics is formed faster in DI water and detergent solvent (swelling liquids) than in glycerine/water mixture (nonswelling liquid) during wet-state Martindale. The tenacity of cellulosic fibers is weaker in swollen state resulting in rapid pill formation and rapid pill removal. The wet-abrasion resistance (Table II) may be responsible for the faster pill formation in CV than in CLY fabrics. The obvious pilling rates obtained by wet-state Martindale with water and detergent

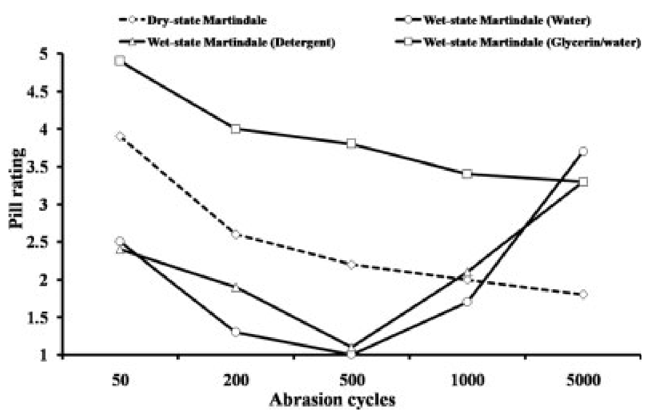
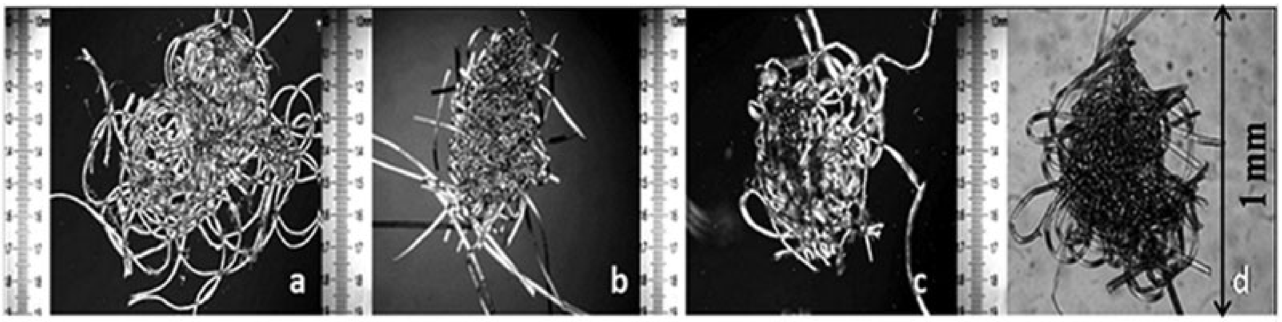


Figure 6 Effect of Martindale-abrasion cycles on pill formation in CV fabrics.



15 W-D cycles
Fibrillation

5000 abrasion cycles
Dry-state Martindale
No Fibrillation

500 abrasion cycles
Wet-state Martindale - Water
Fibrillation

5000 abrasion cycles
Wet-state Martindale
Glycerin/water
No Fibrillation

Figure 7 Microscope images of pill shape and fiber fibrillation in CV fabrics after different pill formation methods.

solvents lead to the possibility of applying the method in substitution of W–D processes. The wet-state Martindale tests with glycerine/water mixture solvent showed incomparable pilling rates. The use of detergent solvent demands extra preparations, whereas the use of water had pinpointed the same rates implying its usage for the RPT.

Influence of fibrillation on pilling formation

Resulting from a series of microscope and SEM pictures, the appearance of fiber fibrillation in pilling mechanism in cellulosic fabrics turned into an important point to be discussed here. It is due to the fact that the fibrillation tendency of cellulosic fibers occupies an important role within the fibers properties. It is well known that the viscose-type fibers showed medium degree of fibrillation compared to other types of cellulosic fibers.[9] To understand how the pill is formed during different pilling-inducement methods, a series of images were accounted here. The microscope and SEM observation showed that fiber fibrillation occurred in the wet state, and the degree of fibrillation intensified following an increasing in W–D cycles and wet-state Martindale-abrasion cycles. Furthermore, the image series indicated that the fibers strongly fibrillated after W–D and wet-state Martindale with water and detergent solvents, whereas no fibrillation was found after wet-state Martindale with glycerin/water mixture and in the dry-state Martindale.

Figure 7 shows the microscopy image series illustrating pill shape and fibrillation on CV fabrics as representatives of images series. To investigate the influence of fibrillation on pilling formation, the pills obtained in four different tests were selected and recorded. Figure 7(a) illustrates the pill formed at the plateau cycles of pill formation of 15 W–D. Figure 7(b) presents the pill at highest abrasion cycles (5000 cycles) in dry-state Martindale. Figure 7(c) is the pill in wet-state Martindale with water at 500 abrasion cycles where the pill removal started. Figure 7(d) is the pill at 5000 abrasion cycles in wet-state Martindale with glycerin/water mixture solvent that is considered as lubrication for preventing high abrasion. Regarding to the role of fibrillation in pill structure, the fibrillation degree could lead to the differences of pill shape based on the way that fibers entangled. The pills formed at W–D are loose and spread in the surface of the fabrics, where the fibers entangled and bended with the rough surface [Fig. 7(a)]. At dry-state Martindale, the pills are round, compact, and the fibers entangled mainly by fiber bending [Fig. 7(b)], whereas at the wet-state Martindale, the pills are rather elongated and fuzzy with fibers entanglement formed by the rough surface and by joining with other protruded fibers [Fig. 7(c)]. The wet-state Martindale with glycerin/water mixture is a particular case where the fibers were slightly swollen and the lubricant nature of mixtures could prevent fibrillation, hence the pill showed a similar shape to the one formed in dry-state Martindale [Fig. 7(d)].

The appearance of fibrillation in pilling formed by different methods is sorted in Table III. The role of fibrillation in pilling mechanism is illustrated at the series of SEM images focusing the differences of fiber in the single pill and fibers in the yarn/fabric surfaces. Figure 8 showed representatives of a series of images. The wet-state abrasion damaged first the fiber on the surface leading to the formation of pilling. Therefore, fibrillation could be found in the fibers inside the pill formed at wet-state Martindale with CD and DI water, whereas the fibers in yarn/fabrics surfaces had no fibrillation as shown in Figure 8(a–c). For each single pill, the fibrillation was found both in the fibers inside the pill structure and in the surface of the pill. It can be assumed that

TABLE III
The Conditions of Pilling with and Without Fibrillation

Pill-inducement methods	Fiber conditions	Pilling
Washing–drying	High wet friction	With fibrillation
Wet-state Martindale with water		
Wet-state Martindale with detergent solutions	High fiber swelling	
Wet state Martindale with glycerine/water mixture	Low wet friction Low fiber swelling	Without fibrillation
Dry-state Martindale	No wet friction No fiber swelling	

cellulosic fiber fibrillation is one factor in pill mechanism at wet-state Martindale and accelerates the formation and removal of pill. During wet-state Martindale test with DI water and CD, the wet fibers were abraded, and the fibrillated fibers were crumpled into the pills. Fibrillation might cause the increase in friction between pills and fabrics surface, resulting in the acceleration of faster pill removal and higher pill rating accordingly.

Contrary to wet-state Martindale, the fiber fibrillation could be found in the fibers in both inner and outer of the single pill formed during W–D test. The fibers fibrillated most at 15 cycles of W–D and 500 cycles of abrasion in wet-state Martindale. It could be attributed to the influence of wet treatment that deeply penetrated into the fibers. In wet-state Martindale, the pill and fiber fibrillation have fast-growing formation rates, even in short cycles of abrasion where the quick pilling inducement can be applied.

CONCLUSIONS

For the application of effective pilling test for cellulosic fabrics, the RPT using Martindale tester with short-abrasion cycles and wet samples is feasible. Because the changes of fiber characters in swollen state are the key point to study pilling propensity in hydrophilic fabrics, we attempted to provide wet state in pilling test to man-made cellulosic fabrics. Moreover, these changes could accelerate the fuzz and pill formation and the obtained pilling rates could be comparable to the rates obtained in W–D. In general, in swollen state, the tenacity of regenerated cellulosic fibers weakens, and the internal friction force of fibers in yarn increases. In addition, the samples are clenched in the Martindale tester modeling the circumstances of W–D cycles. The result is the rapid developments of pills in cellulosic fabrics in shorten time. The test performed with 500 abrasion cycles consumed 10 min in lieu of 2 h consumed by dry-state Martindale and 1–3 days consumed by W–D method. The RPT showed to be effective for the cellulosic fabrics, easy handling, and time saving. Furthermore, performing the RPT in wet-state Martindale justifies the statement that the wet-state reflects the characters of cellulosic fibers including swelling and fibrillation. Although the fiber–fiber friction plays an important role in the pill formation during both wet and dry states, the fiber fibrillation is only visible with fibers during wet state. The pill rates obtained with glycerin/water mixture were similar to the ones obtained with dry-state Martindale. The pill rates obtained with swelling liquids (water and detergent solvents) are highly related to the ones obtained with W–D cycles. Because both swelling liquids showed similar rates, water is sufficient for performing the RPT. However, detergent solutions can be applied to simulate the W–D process depending on the specific purpose. Furthermore, the changes of fiber characters in the wet state could also be the key for the further investigation of pilling in cellullosic fabrics.

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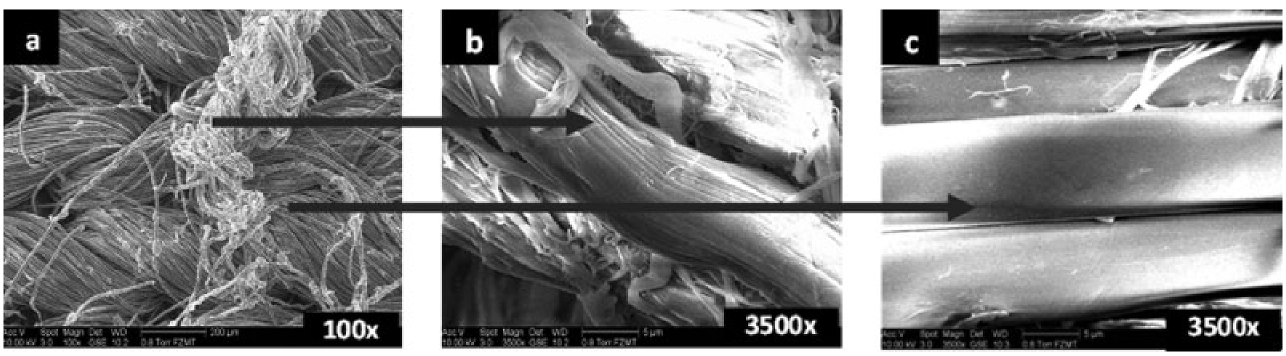

Figure 8 SEM images of a pill (a), fibers from the inner pill (b), and fibers from the outer pill in (c) in CLY fabrics after 50 abrasion cycles at wet-state Martindale (DI water).

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