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Fibrillation tendency of cellulosic fibers—Part 4. Effects of alkali pretreatment of various cellulosic fibers

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Abstract

The effects of alkali type and the concentration in the alkali treatments on the weight loss in six cellulosic fibers and their influences on the fibrillation tendency were investigated. The fibril number of the cellulosic fibers pretreated with alkalis (FN_{pre}) increased with increasing the alkali concentrations as well as the weight loss of the fiber except in the lyocell fiber treated in NaOH and KOH solutions. The FN_{pre} in lyocell was reduced as the fibers were treated in 5 mol/l NaOH and KOH solutions. This result and the fact that the fibers were split in organic alkali such as tetramethylammonium hydroxide even at the low weight loss suggested that not only the loss of cellulose component but also reorganization of microfibril structure, inhomogeneous swelling of the fibers and other influences control the fibrillation tendency of cellulosic fibers.

Keywords: Alkali pretreatment; Fibrillation; Regenerated cellulosic fiber; Weight loss

1. Introduction

Aqueous alkaline solutions especially sodium hydroxide are widely used for cellulosic materials to improve their mechanical and chemical properties such as tensile strength, dimensional stability, dyeability and reactivity (Freytag & Donze, 1983). The numerous studies have been done to investigate the effects of these alkaline solutions on structure and morphology particularly of cotton (Colom & Carrillo, 2002; Tanczos, Borsa, Sajo, Laszlo, Juhasz, & Toth, 2000; Toth, Borsa, & Takacs, 2003; Toth, Borsa, Reicher, Sallay, Sajo, & Tanczos, 2003; Vickers, Briggs, Ibbett, Payne, & Smith, 2001). Other alkali solutions and solvent, e.g. aqueous tetraalkylammonium and liquid ammonium have also gained interest owing to their property not only as a swelling agent but also as a good solvent for cellulose. However, the effects of the alkaline solutions on the fiber properties of regenerated cellulosic fibers have been less investigated.

In the swollen state lyocell fibers, which is a regenerated cellulosic manufactured by means of *N*-methyl morpholine-*N*-oxide, has an extensive fibrillation tendency owing to linear high crystalline fibrillar morphology (Nemec, 1994; Rohrer, Retzl, & Firgo, 2001). The fibrillations induce a lot of troubles, e.g. rope marking defect in hank finishing, graying of dyed fabrics and a change of handle of clothes, that spoils garments features (Rohrer et al., 2001). Efforts to control the fibrillation tendency of lyocell fibers by dyeing with reactive dyestuffs or treating fabrics with crosslinking agents are undergone (Nechwatal, Nicolai, & Mieck, 1996; Nicolai, Nechwatal, & Mieck, 1998); however, few studies have investigated the mechanism of the fibrillation of the cellulosic fibers.

We have investigated fibrillation tendency of lyocell fiber after pretreatment in different type of aqueous alkalis at different concentrations (Zhang, Okubayashi, & Bechtold, submitted for publication-a–c). The fibrillation of lyocell fiber was minimized by the pretreatment in 5 mol/l of NaOH and KOH solutions. The parameter indicating fiber properties, e.g. alkaline retention value, water retention value and

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Nomenclature

CLY1	lyocell fiber
CLY2	lyocell fiber crosslinked with reagent 1
CLY2	lyocell fiber crosslinked with reagent 2
CV1	modal fiber with a linear density of 1.3 dtex
CV2	modal fiber with a linear density of 1.0 dtex
CV3	viscose fiber
TMAH	tetramethylammonium hydroxide
TBuAH	tetrabutylammonium hydroxide
FN_{pre}	fibril number of fiber treated in alkali solution counts
WL	weight loss%
CPF_{pre}	critical point of fibrillation after alkali pretreatment mol/l

weight loss of the fiber were constant regardless of the alkali concentration when the fibrillation was inhibited. These results and the scanning electron microscopic images suggested the reorganization of macrofibrils by the pretreatment in 5 mol/l of NaOH and KOH solutions.

The present work is a follow-up of our former studies. In order to study more details of the fibrillation mechanism of the cellulosic fibers, the effects of the pretreatment in organic base solutions such as tetramethylammonium hydroxide and tetrabutylammonium hydroxide and inorganic base solutions on the fibrillation tendency and the fiber properties of the lyocell, modal and viscose fibers were investigated.

2. Experimental

Six staple fibers of lyocell (CLY1, CLY2 and CLY3), modal (CV1 and CV2) and viscose (CV3) were supplied by Lenzing AG in Austria and used for experiments. The titer of the fibers was 1.3 dtex except of CV2 (1.0 dtex) and the length was 38 mm. The fibers of CLY2 and CLY3 were crosslinked with different chemical agents while CLY1 is untreated. Analytical grade of tetramethylammonium hydroxide (TMAH; aqueous $25\%_{w/w}$ solution), lithium hydroxide (LiOH; $>99\%$) sodium hydroxide (NaOH; $>98\%$), potassium hydroxide (KOH; $>99\%$), tetrabutylammonium hydroxide (TBuAH; aqueous $40\%_{w/w}$ solution) and other chemicals were purchased from Fluka.

The cellulosic fiber was treated in aqueous alkali solutions at various concentrations according to the previous work (Zhang et al., submitted for publication-a–c). Fibrillation was induced in water by a method using metal balls with tumbling (Zhang et al., submitted for publication-a–c). Degree of the fibrillation was assessed by the fibril numbers counted using an optical microscope. The weigh loss (WL) was calculated from the weights of the fiber before and after the treatment (Zhang et al., submitted for publication-a–c).

After the fibers were immersed into alkaline solution for 2 h, photo images of the fiber surfaces were obtained with a Canon digital camera (Power Shot S40) using an optical microscope.

3. Results and discussion

The fibril number of the cellulosic fibers in the length of 0.38 mm was counted after the pretreatment (FN_{pre}) in the aqueous TMAH solution and plotted against the concentration of TMAH in Fig. 1.

The FN_{pre} increases with increasing the concentration of TMAH for all the cellulosic fiber. The pretreatment in TMAH solution promotes the fibrillation of cellulosic fibers. The FN_{pre} of CLY1 is higher than that of CLY2 and CLY3. The crosslinking of fibers retards the fibrillation tendency not only before but also after the treatment in TMAH solution. The FN_{pre} of CLY2 is nearly the same as that of CLY3. There is no apparent effect of different crosslinking agents on the fibrillation tendency after the pretreatment in the same concentration of TMAH. The FN_{pre} of the cellulosic fibers are higher in the order of CLY1 > CLY2, CLY3 > CV1, CV2 > CV3. The fibrillation of the lyocell fibers are accelerated with the pretreatment in TMAH solution more significantly than the viscose fibers.

Fig. 2 shows the relation between the FN_{pre} after the alkali pretreatment in LiOH solution and the concentration of LiOH.

The FN_{pre} of the lyocell fibers and CV1, increase with increasing the concentration of LiOH. No fibrillation occurs in CV2 and CV3 at any concentration of LiOH solution used for the experiment. The FN_{pre} of CLY1 treated in 3 mol/l of LiOH solution is 29 counts and smaller than the FN_{pre} in

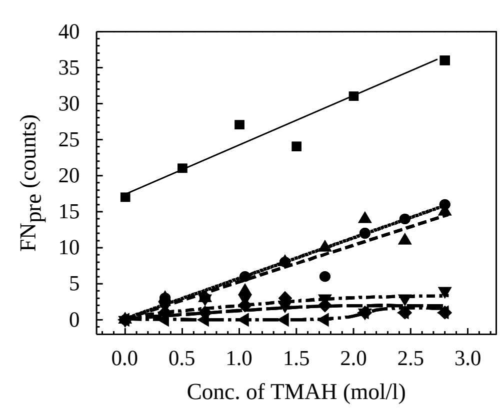

Fig. 1. Plots of fibril number of CLY1 (■), CLY2 (●), CLY3 (▲), CV1 (▼), CV2 (◀) and CV3 (♦) pretreated in TMAH solution against concentration of TMAH.

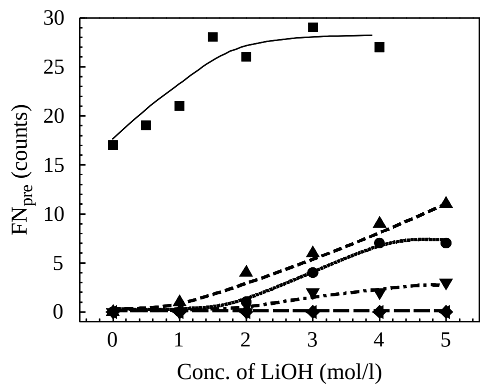

Fig. 2. Plots of fibril number of CLY1 (■), CLY2 (●), CLY3 (▲), CV1 (▼), CV2 (◀) and CV3 (♦) pretreated in LiOH solution against concentration of LiOH.

3 mol/l of TMAH solution (36 counts). The FN_{pre} of other cellulosic fibers treated in LiOH solution are also smaller than the FN_{pre} in TMAH solution at the same concentration. The pretreatment in LiOH solution accelerates the fibrillation of cellulosics less greatly than in TMAH solution. The CV1 with a linear density of 1.3 dtex is fibrillated in higher concentration of LiOH solution than 3 mol/l though the CV2 with a linear density of 1.0 dtex is not. The linear density may be a factor affecting the fibrillation tendency.

The effects of the concentration of aqueous NaOH solution used for the pretreatment on the FN_{pre} are given in Fig. 3.

The FN_{pre} of the fibers except CLY1 increases with increasing the concentration of NaOH. The FN_{pre} of CLY2 after the pretreatment in 4 mol/l of NaOH solution is 5 counts and smaller than the FN_{pre} in 4 mol/l of LiOH solution (6 counts). The pretreatment in NaOH solution enhances the fibrillation of CLY2, CLY3 and CV1 less remarkably than in LiOH and TMAH solutions. The relation between the FN_{pre} of CLY1 and the concentration of NaOH is entirely different from those of other cellulosics. After the FN_{pre} of the lyocell in NaOH solution increases up to 24 counts, it decreases down to 7 counts with increasing

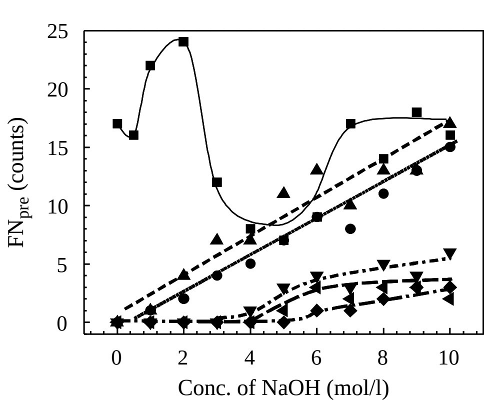

Fig. 3. Plots of fibril number of CLY1 (■), CLY2 (●), CLY3 (▲), CV1 (▼), CV2 (◀) and CV3 (♦) pretreated in NaOH solution against concentration of NaOH.

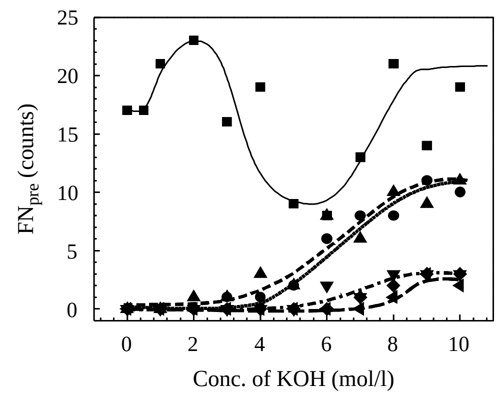

Fig. 4. Plots of fibril number of CLY1 (■), CLY2 (●), CLY3 (▲), CV1 (▼), CV2 (◀) and CV3 (♦) pretreated in KOH solution against concentration of KOH.

the alkaline concentration up to 5.0 mol/l. Further, the FN_{pre} increases again and reaches at constant value of 17 counts at 7.0 mol/l of NaOH solution. The fibrillation tendency of CLY1 pretreated in KOH solution is similar to that in NaOH solution (Fig. 4). The fibrillation tendency of CLY1 in NaOH and KOH solutions are related to the reorganization of its macrofibrils (Zhang et al., submitted for publication-a–c).

The critical point of fibrillation (CPF_{pre}) that is a concentration of the aqueous alkaline solution used for the pretreatment where the fibrillation begins is extrapolated. This CPF_{pre} corresponds to an intercept of *x*-axis in the graphs shown in Figs. 1–4. The results are given in Table 1. The CPF_{pre} is an indicator of the fibrillation stability (Zhang et al., submitted for publication-a–c).

The CLY1 is fibrillated without alkali pretreatment, that is, the CPF_{pre} of CLY1 is smaller than 0 mol/l of all alkaline solutions. The CLY1 fiber has the lowest fibrillation stability against the water and alkali pretreatment. The CPF_{pre} of CLY2 and CLY3 are between 0 and 2 mol/l of the alkaline solutions. The fibrillation stability of CLY2 and CLY3 are the same and higher than that of CLY1. There is no apparent effect of different crosslinking agents on the fibrillation tendency of the lyocell fiber at the same alkali concentration.

The CPF_{pre} of CV fibers is larger than that of lyocell fibers, indicating that the fibrillation stability of the CV

Table 1
Critical point of fibrillation of cellulosic fibers after alkali pretreatment

Material	CPF_{pre} (mol/l)			
	TMAH	LiOH	NaOH	KOH
CLY1	≫[a]	≫[a]	≫[a]	≫[a]
CLY2	0	1	0	2
CLY3	0	0	0	1
CV1	0	2	3	5
CV2	0	No fibrillation	5	3
CV3	No fibrillation	No fibrillation	4	7

[a] The CPF_{pre} of CLY1 is smaller than 0 mol/l of alkaline solution.

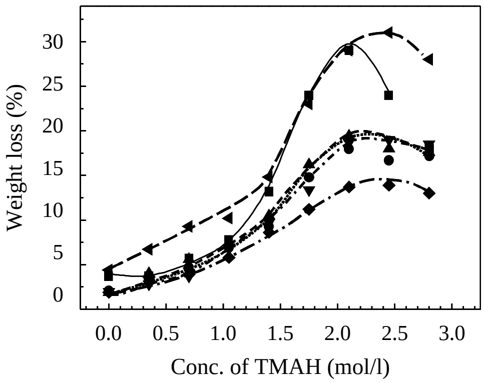

Fig. 5. Plots of weight loss of CLY1 (■), CLY2 (●), CLY3 (▲), CV1 (▼), CV2 (◀) and CV3 (♦) in TMAH solution against concentration of TMAH.

fibers is higher than that of the lyocell fibers. As compared with the CPF_{pre} of CV1 among four different alkalis, the value is smaller in the order of TMAH < LiOH < NaOH < KOH. The alkali pretreatment promotes the fibrillation of CV1 greatly in the order of TMAH > LiOH > NaOH > KOH. This order is consistent with the order of the fibril number of CLY1 obtained in alkaline solution at the same concentration (Zhang et al., submitted for publication-a–c). These results are attributed to the large size and non-polar part of TMAH being able to penetrate into the non-polar part of the lyocell fiber (Klemm, Philipp, Heinze, Heinze, & Wagenknecht, 1998) and the larger size of the hydrated cations in the order LiOH > NaOH > KOH (Kielland, 1937). On the other hands, the fibrillation of CV3 is accelerated with alkali pretreatment not in TMAH but in NaOH and KOH solutions. Further experiments are required to discuss in details.

In order to understand the mechanism of fibrillation of the cellulosic fibers, the effect of weight loss of the fibers in alkaline solutions on the fibrillation tendency was investigated. The weight loss is estimated and plotted against the concentration of the alkaline solutions in Figs. 5–8.

The curves of the weight loss show peaks around 2.0–2.5 mol/l of TMAH solution regardless of the type of cellulosics (Fig. 5). In other alkaline solutions the weight loss increases with increasing the alkali concentration. The weight loss of CLY1 in 2.0 mol/l of TMAH, LiOH, NaOH and KOH solution are 29, 11, 5 and 4%. Larger amount of the cellulose component dissolves into alkaline solution in the order of TMAH > LiOH > NaOH > KOH. This order agrees with the order of the FN_{pre} in the alkaline solutions, which indicates that the fibrillation tendency is associated with the weight loss of the fibers. The weight loss of the viscose fiber (CV3) is lower than those of any other fibers in the alkaline solution at the same concentration in spite of its low crystallinity, i.e. large amorphous area (Kreze & Malej, 2003). The weight loss is not assuredly related to the crystallinity. The weight loss of CV2 which is thinner modal fiber is larger than those of other cellulosics in alkaline solution except in LiOH solution. This result could be referred to the larger surface area of the thinner fibers.

For further discussion the FN_{pre} are plotted against the weight loss of the fiber in the alkaline solutions in Figs. 9–12.

The FN_{pre} of CLY1 increases with increasing the weight loss in TMAH and LiOH solutions as shown in Figs. 9 and 10. The fibrillation of CLY1 is remarkably higher than those

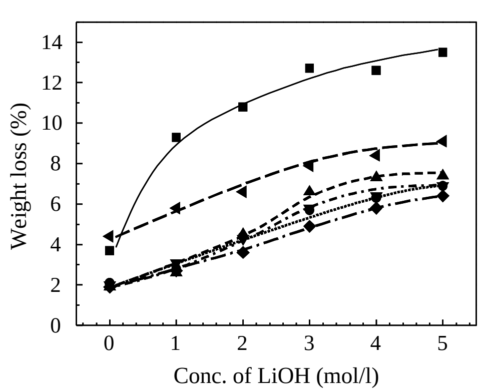

Fig. 6. Plots of weight loss of CLY1 (■), CLY2 (●), CLY3 (▲), CV1 (▼), CV2 (◀) and CV3 (♦) in LiOH solution against concentration of LiOH.

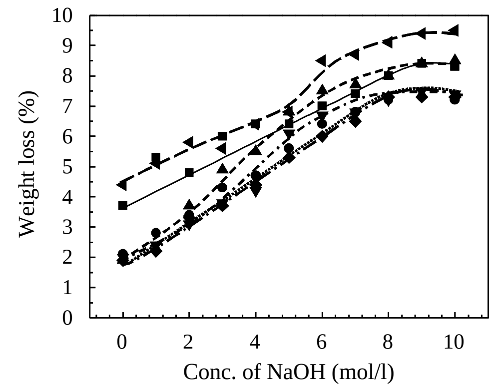

Fig. 7. Plots of weight loss of CLY1 (■), CLY2 (●), CLY3 (▲), CV1 (▼), CV2 (◀) and CV3 (♦) in NaOH solution against concentration of NaOH.

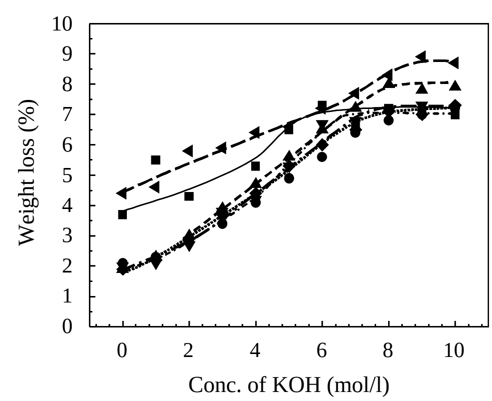

Fig. 8. Plots of weight loss of CLY1 (■), CLY2 (●), CLY3 (▲), CV1 (▼), CV2 (◀) and CV3 (♦) in KOH solution against concentration of KOH.

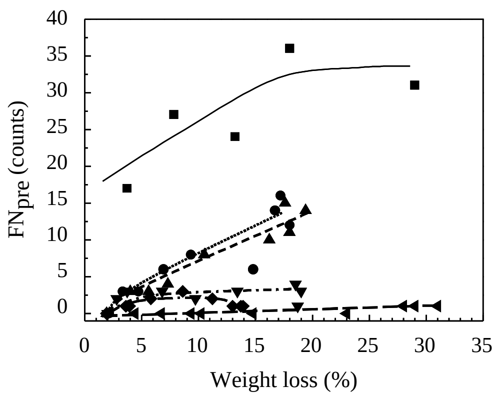

Fig. 9. Plots of fibril number of CLY1 (■), CLY2 (●), CLY3 (▲), CV1 (▼), CV2 (◀) and CV3 (♦) pretreated in TMAH solution against weight loss.

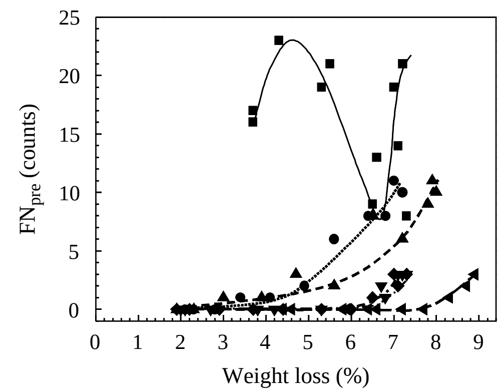

Fig. 11. Plots of fibril number of CLY1 (■), CLY2 (●), CLY3 (▲), CV1 (▼), CV2 (◀) and CV3 (♦) pretreated in NaOH solution against weight loss.

of other fibers although when the weigh loss is low. Not only the weight loss of cellulose component but also other effects should be considerable to promote the fibrillation of CLY1. On the other hands, the FN_{pre} of CLY1 in both NaOH and KOH solutions increases with increase in the weight loss up to 23% and decreases down to 8% and then increases again. This result is due to the reorganization of fibrils in CLY1 in ca. 5 mol/l of NaOH and KOH solutions (Zhang et al., submitted for publication-a–c). The treatment of the lyocell fiber in TMAH and LiOH solutions causes different fibrillation tendency from that in NaOH and KOH solutions.

The FN_{pre} of CLY2 and CLY3 exponentially increase with increasing the weight loss in LiOH, NaOH and KOH solutions. The FN_{pre} of CLY2 and CLY3 enhances more significantly around the weight loss of 6% in LiOH, NaOH, and KOH solutions. The value of 6% is a maximal weight loss to obtain low fibrillation of crosslinked lyocell fibers when inorganic alkalis such as LiOH, NaOH and KOH are used for the treatment of the fibers. Considering that the fibrillation of CLY1 retards at 6.5% of the weight loss in NaOH and KOH, the critical reorganization of fibrils could occur with accompanying 6–6.5% of the weight loss of cellulose component.

The FN_{pre} of CLY2 is higher than that of CLY3 at the same weight loss in NaOH and KOH solutions especially at higher weight loss. There was no apparent difference of fibrillation tendency between different crosslinking agents at the same alkali concentration. However, CLY2 is fibrillated more highly than CLY3 when the weight loss is the same.

The fibrillation tendency of cellulosic fibers in an organic alkali TMAH is different from that in inorganic alkaline solutions. The fibers are easily fibrillated at lower weight loss as compared with inorganic alkalis. This could be owing to the property of TMAH as a solvent for cellulose.

The viscose fiber (CV3) shows the highest resistance to the fibrillation after the alkaline pretreatment as compared with other cellulosic fibers. Few or no fibrillation is observed in TMAH and LiOH solutions in which other fibers are greatly fibrillated. On the other hands, the fibrillation of CV3 occurs when the weight loss is 8 and 7% in NaOH and in KOH solutions. The fibrillation mechanism of viscose could be different from that of lyocell and modal fibers because of their different morphology. Additional experiments will be performed to clarify the difference of the mechanism.

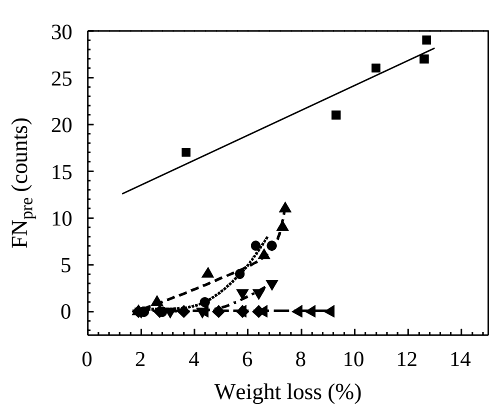

Fig. 10. Plots of fibril number of CLY1 (■), CLY2 (●), CLY3 (▲), CV1 (▼), CV2 (◀) and CV3 (♦) pretreated in LiOH solution against weight loss.

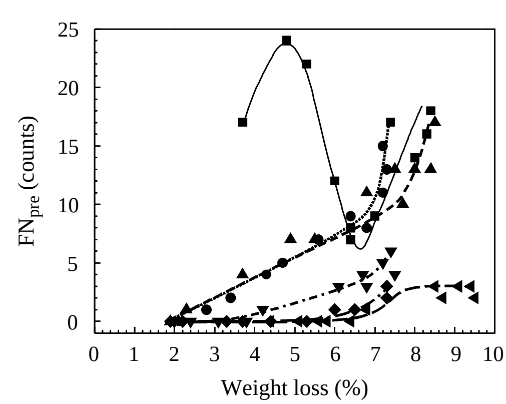

Fig. 12. Plots of fibril number of CLY1 (■), CLY2 (●), CLY3 (▲), CV1 (▼), CV2 (◀) and CV3 (♦) pretreated in KOH solution against weight loss.

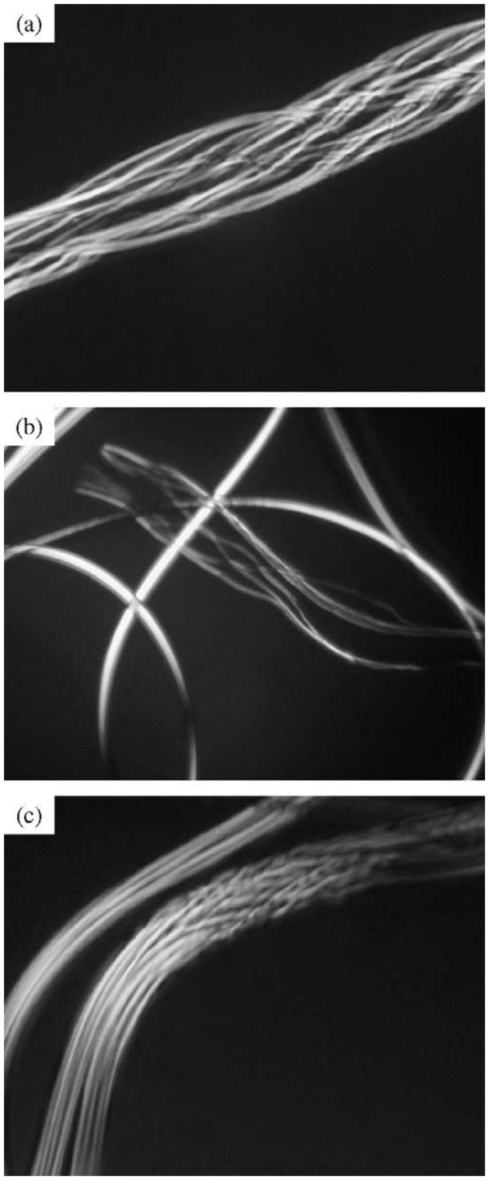

Fig. 13. Photo images of fiber surface of CLY1 in 1.75 mol/l of TMAH solution (a), CV1 in 1.75 mol/l of TMAH solution (b) and CV2 in 2.8 mol/l of TMAH solution.

The cellulosic fibers were immersed into alkaline solution for 2 h and the fiber surface was observed with an optical microscope. The results are shown in Fig. 13.

The CLY1, CV1 and CV2 fiber are split in 1.75, 1.75 and 2.8 mol/l of TMAH solution. The results in other alkaline solutions are summarized in Table 2.

There is no split of the cellulosic fibers except of CLY1 in high concentration of inorganic alkaline solutions. Contrarily, TMAH and TBuAH solutions promote the fiber splits at the low concentrations. Taking into account that the weigh loss of cellulosic fibers in 1.05 mol/l of TMAH solution is lower than those in 5 mol/l of LiOH solution except in CLY1, in 8 mol/l of NaOH solution and in 8 mol/l of KOH solution, the organic alkalis such as TMAH and TBuAH could not only dissolve the cellulose more remarkably but also penetrate between macrofibrils of the fiber more easily and decrease the interfibril force than the inorganic alkalis.

4. Conclusions

The extent of fibrillation was enhanced with increasing the alkali concentration and the size of hydrated alkali resulting in the increment of weight loss. However, lyocell fiber (CLY1) was fibrillated even at low weight loss besides the fibrillation tendency in CLY was lowered due to the reorganization of macrofibril structure when the fibers were treated in 5 mol/l of NaOH and KOH solutions. These results indicate that not only the weight loss but also other effects control the fibrillation of cellulosic fibers. The fiber split observed in TMAH and TBuAH solutions at lower weight loss also suggests the other influences, e.g. inhomogeneous swelling in the solutions leading to high degree of fibrillation. The fibrillation tendency of the cellulosic fibers was markedly promoted in all the alkali solutions when the weight loss in the fibers was larger than 6–6.5%, suggesting that the critical reorientation occurs or interfibril force is reduced at the weight loss of 6–6.5%.

Table 2
Effect of alkali concentration on split of fibers

Material	Concentration of alkali (mol/1)												
	TMAH									TBuAH	LiOH	NaOH	KOH
	0.00	0.35	0.70	1.05	1.40	1.75	2.10	2.45	2.80	0.75	5.00	8.00	8.00
CLY1	n	n	n	s	s	s	s	s	s	s	s	n	n
CLY2	–	–	–	–	–	–	–	–	–	–	–	–	–
CLY3	–	–	–	–	–	–	–	–	–	–	–	–	–
CV1	n	n	s	s	s	s	s	s	s	s	n	n	n
CV2	n	n	s	s	s	s	s	s	s	s	n	n	n
CV3	n	n	n	n	s	s	s	s	s	s	n	n	n

The symbols n and s indicate that no split (n) and some splits (s) of fiber were observed in the alkaline solution.

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