Cellulose (2005) 12:403–410
DOI 10.1007/s10570-005-2179-y

Water accessibilities of man-made cellulosic fibers – effects of fiber characteristics

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Key words: BET, Cellulosic fiber, Fiber characteristic, Hysteresis, Kinetics, Lyocell, Moisture sorption, PEK model, Water retention capacity

Abstract

The dynamic vapor water sorption and desorption experiments were performed on cellulosic fibers with different characteristics. The hysteresis between moisture sorption and desorption cycle at 10% relative humidity (RH) was independent on the total moisture regain and approximately 45% for all materials except for viscose fibers. Brunauer–Emmett–Teller surface volume (V_m) for moisture sorption and retention capacity of liquid water (WRV) were also measured. The V_m and WRV increase in proportion to the total amount of moisture sorption ($M_{\mathrm{inf(total)}}$) in all specimen except in poplar fiber. The coefficients of parallel exponential kinetics (PEK) were estimated by the curve-fitting of experimental data of the moisture regain, and the influences of the fiber characteristics on the PEK coefficients, the moisture regain, the hysteresis, V_m and WRV are discussed. The total equilibrium moisture content in the viscose fibers was higher but the moisture uptake and release rate was slower than the lyocell and poplar fibers. The cationization and the modification of shape of cross section accelerated the total equilibrium moisture content in the viscose fiber. A drying process at low temperature enhanced both the equilibrium moisture content and the moisture uptake and release rate in lyocell fibers while a spin finish retarded them. The total equilibrium moisture content was heightened by the crosslinking of the fiber, however, no obvious effect of the crosslinking on the moisture uptake and release rate was found. Effects of the type of the specimen and linear density on the moisture accessibilities are also discussed.

Introduction

Comfortable clothing materials are obtained when they have a high moisture retention capacity and high moisture transportation properties. The moist fibers can act as a heat reservoir, thus temperature and humidity between skin and textiles are properly controlled. An affinity of moisture with fibers, which can be monitored by moisture sorption/desorption dynamics, is an important indicator to evaluate wearing comfort of textile materials.

Several researchers have investigated moisture sorption and desorption especially in hydrophilic fiber materials e.g. cotton, wool, viscose rayon, nylon and silk. Many models of the mechanism of water sorption on their sorption sites were suggested considering continuous structural and property change owing to the swelling (Peirce 1929; Speakman 1944; Barrer 1947; Hill 1950; Young et al. 1967). Kohler et al. have reported that water exchange on natural fibers such as flax and hemp was modeled by two parallel independent first order processes, which was defined parallel exponential kinetics (PEK) model (Bartsch et al. 2001). We also reported in a recent papers (Okubayashi et al. 2004) that moisture sorption and desorption kinetics on cellulosic fibers such as viscose and lyocell can be described by the PEK model, suggesting that a mechanism of moisture sorption in the lyocell fiber is particularly different from cotton considering the effects of humidities on the PEK coefficients, Brunauer–Emmett–Teller (BET) surface volume (Brunauer et al. 1938) and water retention capacity. In other study (Okubayashi et al. 2005) we have quantitatively investigated the moisture accessibilities of various cellulosics, wool, down, and polyester fiber using PEK model. The lyocell fibers showed higher equilibrium and kinetics of moisture sorption, i.e. integrally high moisture accessibility. The effects of temperature on the PEK parameters suggested thermodynamics of fast and slow moisture sorption on different fibers.

The present work is a follow-up of our former studies. We describe the dynamics of moisture sorption and desorption in regenerated cellulosic fibers with different characteristics quantitatively using the PEK model. The effects of the fiber characteristics on the PEK coefficients, the hysteresis, BET constant and the water retention value are also discussed.

Experimental

Materials

Nine different types of lyocell materials (L1 to L9), three viscose fibers (CV1, CV2 and CV3) and a poplar down fiber (Pp) were kindly supplied from Lenzing AG in Austria and used for experiments. The technical properties of the fibers are given in Table 1.

CV3 fibers were prepared by standard viscose spinning process following incorporation of cationic polymer in the viscose spinning (Sulek et al. 2002). The spun fibers were treated with silicon or non-silicon agent to decrease interfiber friction during the spinning according to general spin finishing process (Woodings 2001). The length of the fibers was 38–40 mm, yarn type was ring yarn with 930 T/m of twist, and the density of the knitted fabric was 163 g/m^2. The fibers were spun in a wet process and dried at 65% relative humidity (RH) and 20 °C in a climate chamber or dried at 100–105 °C in an oven.

Measurements

Dynamic water vapor sorption and desorption was gravimetrically measured using an automatic multisample moisture sorption analyzer SPS11 (Project-Messtechnik, D-Ulm) at 20 °C (Okubayashi et al. 2004). Approximately 1.0 g of each fiber material in an aluminum sample dish (diameter: 50 mm) was placed in the moisture sorption analyzer. The system is equipped with an analytical balance with a resolution of 10 μg and a sample changer which allows the simultaneous gravimetric analysis of eleven samples. The atmosphere in the analyzer was conditioned at 20 °C and 0% relative humidity (RH) until equilibrium was achieved. Then the moisture sorption cycle was started rising the relative humidity in 10% RH steps (interval method). The mass change of the

Table 1. Properties of various cellulosic materials and treatment conditions.

Symbol	Material	Type	Title (dtex)	Crosslinking	Drying temperature (°C)	Finishing agent	Cross section
L1	Lyocell	Fibre	1.3	–	ca. 100	Non-silicon	Round
L2	Lyocell	Fibre	0.9	–	ca. 100	Non-silicon	Round
L3	Lyocell	Fibre	0.9	–	ca. 100	Silicon	Round
L4	Lyocell	Fibre	1.3	–	105	–	Round
L5	Lyocell	Fibre	1.3	–	20	–	Round
L6	Lyocell	Knit	1.3	–	ca. 100	Non-silicon	Round
L7	Lyocell	Yarn	1.3	–	ca. 100	Non-silicon	Round
L8	Lyocell	Yarn	1.3	Agent A	ca. 100	Non-silicon	Round
L9	Lyocell	Yarn	1.3	Agent B	ca. 100	Non-silicon	Round
CV1	Viscose	Fibre	1.3	–	ca. 100	Non-silicon	Popcorn
CV2	Viscose	Fibre	1.3	–	ca. 100	Non-silicon	Trilobal
CV3	Viscose	Fibre	1.3	–	ca. 100	Cationic polymer	Popcorn
Pp	Poplar	Fibre	–	–	–	–	–

material was recorded every 8 min and the equilibrium condition was set to $<0.02\%$ total mass change within 40 min. Every time this condition was fulfilled for all samples the relative humidity was automatically increased by 10% RH up to 90% RH and then stepwise decreased down to 0% RH. The full sorption/desorption cycle took 4 days and the mass change at each equilibrium condition was used to draw the moisture sorption isotherms. The temperature was 20 ± 0.1 °C and the variation of the humidity values were less than $\pm 0.5\%$ ($\pm 1\%$ at 80 and 90% RH).

The water retention capacity (WRV) was evaluated by a centrifugal method according to the previous work (Okubayashi et al. 2004).

Results and Discussion

Moisture regain and loss of the fibers at stepwise increasing and decreasing relative humidities were gravimetrically measured with the automatic moisture sorption analyzer. The time versus mass change (M_t) profile of a full sorption/desorption cycle is shown in Figure 1 (L5). The fibers clearly reach an equilibrium state at each moisture interval. The values of the individual equilibrium states ($M_{\mathrm{inf(total)}}$) were plotted against the relative humidity (RH) to draw the equilibrium isotherm shown in Figure 2. The isotherm curves have almost similar sigmoidal shapes among the sample materials due to a stronger moisture uptake or release at very low or very high relative humidities. The equilibrium moisture isotherms show a distinct hysteresis between sorption and desorption cycle, indicating structural changes of the fiber caused by the interaction with water (Hermans 1949).

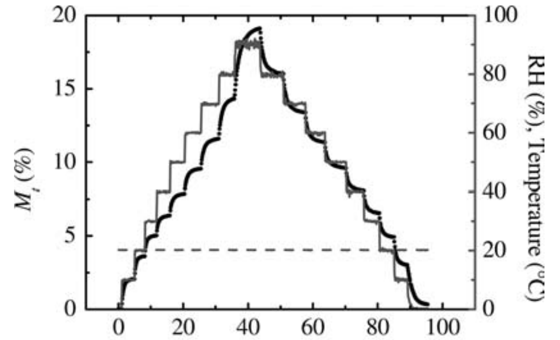

Figure 1. Mass change over time profile of the full sorption/desorption cycle (interval method) for L5 (●) at 20 °C (- - - -); the steplike curves shows the course of the relative humidity.

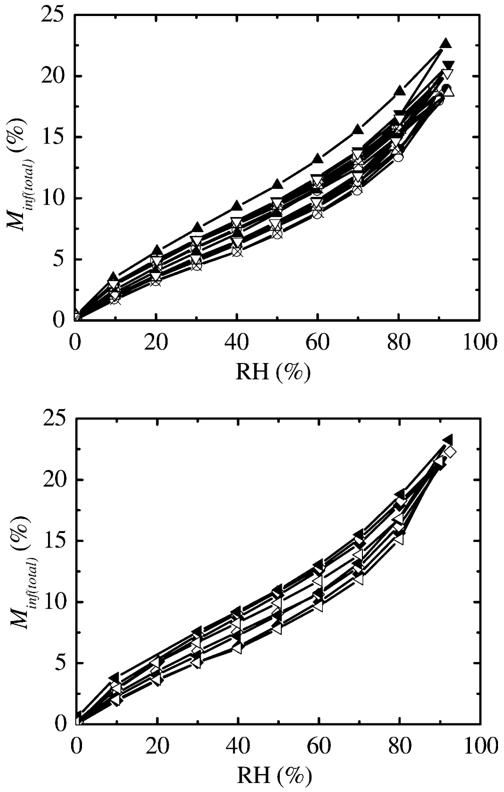

Figure 2. Equilibrium moisture sorption and desorption isotherms of L1 (●), L2 (○), L3 (x), L4 (■), L5 (□), L6 (▲), L7 (△), L8 (▼), L9 (▽), CV1 (◆), CV2 (◇), CV3 (◀), and PP (◁) at 20 °C.

The extent of the hysteresis at 20 °C was calculated according to the previous study (Okubayashi et al. 2004) and plotted against the relative humidities in Figure 3. The extent of the hysteresis clearly decreases with increasing relative humidity regardless the character of the samples. This suggests that the fiber structure strongly changes when moisture is adsorbed by the dry fiber or all moisture desorbs from the fiber. Obviously, this structural change becomes smaller after some moisture has been absorbed by the fiber. No significant difference of the curves is observed among the lyocell materials. The effect of the relative humidities on the hysteresis in CV3 is significant. It could be due to the fact that the cationized viscose CV3 has the larger amount of attractive sites for moisture sorption (Sulek et al. 2002).

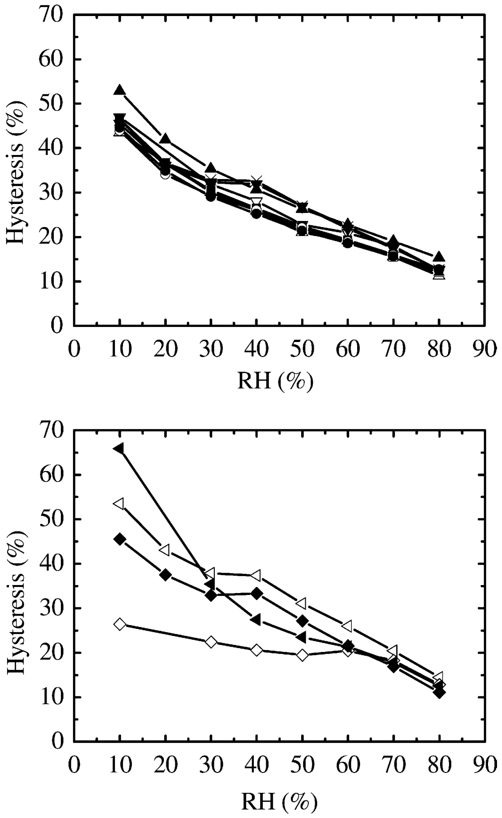

Figure 3. Plot of the relative hysteresis between the moisture sorption and desorption cycle versus relative humidity for L1 (●), L2 (○), L3 (x), L4 (■), L5 (□), L6 (▲), L7 (△), L8 (▼), L9 (▽), CV1 (◆), CV2 (◇), CV3 (◀), and PP (◁) at 20 °C

Contrarily, the effect of the relative humidities on the hysteresis in CV2 was less remarkable. The three dimensions of structure could reversibly change during the moisture sorption and desorption owing to the trilobal cross section of CV2 (Schmidtbauer 2002).

Figure 4 shows the relation between the hysteresis at 10% RH and $M_{\mathrm{inf(total)}}$ at 60% RH of the specimen tested in the experiment. The $M_{\mathrm{inf(total)}}$ of L2 with thinner linear density is lower than that of L1. The spin finishing hinders the moisture sorption. The drying at lower temperature enhances it as the results of L2, L3, L4 and L5 are compared. The $M_{\mathrm{inf(total)}}$ of lyocell knitted fabric (L6) is significantly high compared to the other lyocell materials. Additional experiments are required to explain the high moisture sorption on the lyocell knit. The $M_{\mathrm{inf(total)}}$ of the lyocell fabrics is accelerated by the crosslinking treatment. This result is owing to the change of fiber structure after the treatment with crosslinking agent under alkaline condition (Rohrer et al. 2001). The moisture regain of the viscose fibers is higher than that of the lyocells because of its lower crystallinity (Schurz 1994) and larger pore volume (Brederick et al. 1996).

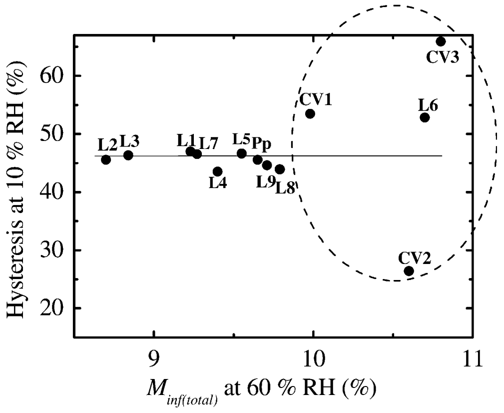

Figure 4. Plots of the relative hysteresis at 10% RH and 20 °C against total moisture regain at 60% RH and 20 °C.

Regarding the hysteresis, all the lyocell materials except in L6 demonstrated similar values of 45% regardless of the fiber characters. As the $M_{\mathrm{inf(total)}}$ is higher than 10%, the hysteresis increases up to 67% in CV3 and decreases down to 27% in CV2. The effect of the fiber modification on the hysteresis is more remarkable in the viscose than in the lyocell materials.

A quantity of moisture adsorbed in a monomolecular layer on the surface of the material (V_{m}) was determined according to the Brunauer–Emmett–Teller (BET) model (Okubayashi et al. 2004) and plotted against the moisture regain at 60% RH in Figure 5. The V_{m} increases with increase in linear density of the lyocell fiber though there is no significant effect of spin finishing, drying temperature and type of the specimen on V_{m}. The V_{m} of the viscose fibers is larger than that of the lyocells, which is consistent with the fact that the $M_{\mathrm{inf(total)}}$ at 60% RH of the viscose is larger than that of the lyocells. The values of V_{m} increases in proportion to $M_{inf(total)}$ at 60% RH in all materials used for the experiments regardless of the fiber characters.

The WRV was measured by a centrifugal method. Figure 6 gives the plots of the WRV against $M_{\mathrm{inf(total)}}$ at 60% RH. The WRV of L5

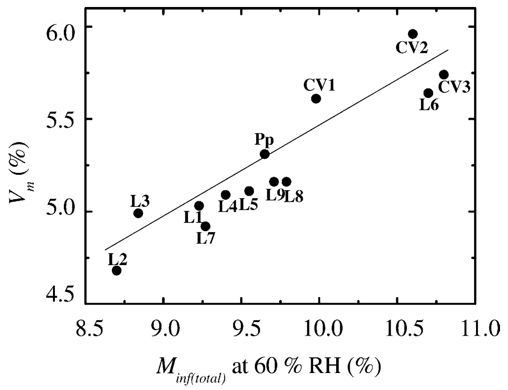

Figure 5. Plots of V_m at 20 °C against total moisture regain at 60% RH and 20 °C.

dried at lower temperature is larger than L1, L2, L3, L4 and L7. The slow evaporation of water from never-dried fibers interrupts collapse of voids inside the fiber (Gruber et al. 2001), and thus, L5 remains higher water retention capacity. A comparison of Figure 6 with Figure 5 indicates that the fiber crosslinking heighten the WRV in the same manner as it enhances V_m. The viscose fibers demonstrated higher WRV than the lyocell materials as well as the higher moisture regain and V_m. The WRV increases with increase in $M_{inf(total)}$ of the materials except in Pp. This tendency is similar to the fact that V_m increases with increase in $M_{inf(total)}$. The WRV is clearly related to V_m. The WRV of Pp is remarkably high though its moisture regain is middle value among the specimen used for the measurements. This is owing to the high amorphous fraction of Pp fiber (Simionescu et al. 1971).

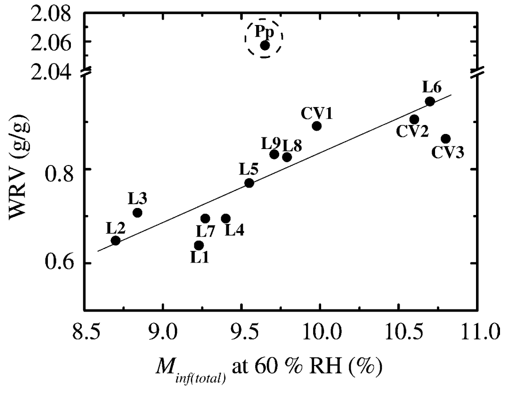

Figure 6. Plots of water retention value against total moisture regain at 60% RH and at 20 °C.

The total equilibrium moisture content of the materials was discussed in Figure 1 to Figure 6. In order to investigate the dynamics of moisture sorption/desorption in two distinct fast and slow processes the parallel exponential kinetic (PEK) model was applied. Quantity of partial equilibrium moisture sorption M_{inf} and characteristic time τ at 60% RH and 20 °C were estimated from curve-fitting of experimental data (Okubayashi et al. 2004). Figures 7 and 8 summarize the results of M_{inf} and τ. Subscripts 1 and 2 correspond to the two distinct kinetic processes defined as fast and slow, corresponding to slow and fast sorption sites.

The M_{inf1} value of thinner lyocell fiber (L2 and L3) is larger than M_{inf2} value though M_{inf1} value of the other lyocell fibers is smaller than M_{inf2} value. This indicates that quantity of the site for fast sorption is larger than that for slow sorption

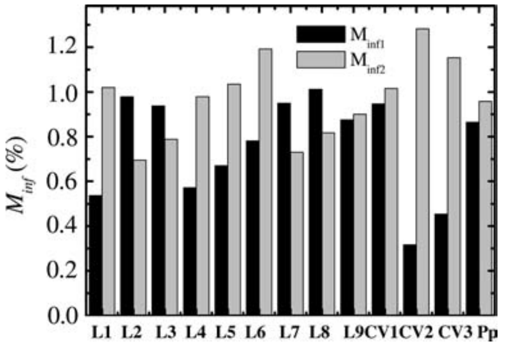

Figure 7. Partial equilibrium amount of moisture sorption in cellulosic materials at 60% RH and at 20 °C.

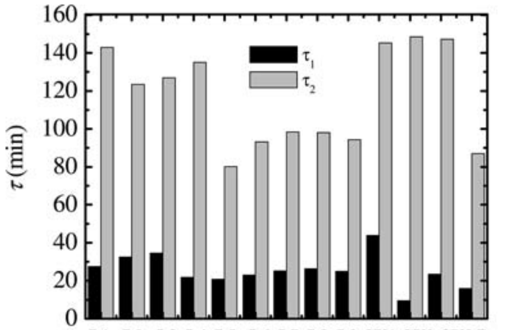

Figure 8. Characteristic time of moisture sorption in cellulosic materials at 60% RH and at 20 °C.

in thinner lyocell fibers. The large amount of the site for fast sorption is due to the larger surface area of thinner fibers per certain mass of the specimen. The viscose fibers especially CV2 and CV3 show the lowest M_{inf1} and the highest M_{inf2} among all the sample materials examined in the experiments. The effect of the fiber modification using additional polymer or change of the shape in the cross section on kinetics of moisture sorption is prominent.

The τ_2 of L5 that is the lyocell fiber dried at room temperature, is 80 min and smaller than the τ_2 of the other lyocell fibers. The expeditious moisture sorption in slow process of L5 is strongly related to the high $M_{\mathrm{inf(total)}}$, high V_{m} and high WRV. A comparison of τ_2 of the lyocell fiber with that of the lyocell yarn suggests that slow moisture sorption is slower in the lyocell fiber than in the lyocell yarn. No further obvious effect of title and finishing agent on τ is found. The rate of moisture sorption is affected by the drying rate and the type of lyocell specimen more greatly than by the other factors. The τ_1 and τ_2 of Pp are 16 and 87 min, which are comparatively smaller than the other materials. This means that the rate of the moisture sorption in Pp is remarkably high, whereas the apparent equilibrium moisture content is moderate but the accessibility of liquid water is high as shown in Figures 5 and 6. The large values of τ_1 and τ_2 of CV1 indicate that CV1 gradually adsorbs water molecules in any sites. The τ_1 of CV2 and CV3 is small and the τ_2 is large, which indicates that fast sorption is more rapid and slow sorption is slower in CV2 and CV3.

The effects of the characteristics of the materials on the kinetics of the moisture sorption are summarized in Table 2. The viscose fiber showed the high equilibrium moisture content but the low moisture uptake rate as compared to the lyocell and the poplar fibers. The results of τ indicate that the moisture sorption in poplar is remarkably rapid. This could be because of the high amorphous fraction, low orientation of crystallite and semi-amorphous region of the poplar down fiber (Simionescu et al. 1971).

The τ_1 and τ_2 of the lyocell knitted fabric and yarn are smaller than those of the lyocell fiber, indicating that the moisture uptake rate of the knit and yarn lyocell are higher than the fiber. The knit and yarn lyocell also show a higher equilibrium moisture content than the fiber. The bulk density of the sample mass is different among various material types, which maybe affect the kinetics of moisture sorption. Additionally, some capillary condensation might occur due to close contacts of the fibers in the yarn and knit. Further experiments should be conducted to clarify these assumptions.

Concerning the effect of the linear density, the total equilibrium moisture content of the lyocell fiber with a linear density of 1.3 dtex is higher than that with 0.9 dtex. However, the partial amount of moisture sorption in fast sorption is larger in the thinner lyocell fiber while the rate of moisture sorption in fast sorption is lower in the thinner lyocell. Contrarily, the amount of moisture sorption in slow sorption is larger in the thicker lyocell fiber while the rate of moisture sorption in slow sorption is lower in the thicker lyocell fiber. The different water accessibility between the fibers bearing different linear density may be caused by the different crystallinity, orientation of the amorphous region as well as other physical parameters.

Table 2. Effects of fiber characteristics on PEK coefficients at 60% RH and at 20 °C.

Factor	Comparison	PEK coefficient				
		Minf(total)	Minf1	Minf2	τ_1	τ_2
Material	L1, CV1, PP	CV > Pp > L	CV = Pp > L	CV = L = Pp	CV > L > Pp	CV = L > > Pp
Type	L1, L6, L7	K > Y = F	Y > K > F	K > F > Y	F > K = Y	F > K = Y
Title (dtex)	L1, L2	1.3 > 0.9	0.9 > 1.3	1.3 > 0.9	0.9 > 1.3	1.3 > 0.9
Crosslinking	L7, L8, L9	A = B > No	A > No > B	B > A > No	A = B = No	A = B = No
Drying temperature (°C)	L4, L5	20 > 100	20 > 100	20 > 100	20 = 100	100 > 20
Spin finishing	L1, L4	No > Finish	Finish > No	No = Finish	Finish > No	Finish > No
Modification	CV1, CV2, CV3	Cat > Tri > No	No > Cat > Tri	Tri > Cat > No	No > Cat > Tri	Cat = Tri = No

K, Y, F, No, Cat and Tri indicate knitted fabric, yarn, fiber, no treatment, cationized and trilobal fiber.

The effect of crosslinking the lyocell fiber on kinetics of moisture sorption is not significant. However, the crosslinking treatment enhances the total equilibrium moisture content. The effect of the drying temperature on the water accessibilities is striking. The drying process at lower temperature e.g. at 20 °C accelerates both the equilibrium moisture content and the moisture uptake rate of the lyocell fiber. The collapse of the voids in the fiber is escaped and the large quantity of the sites for moisture sorption is retained owing to the slow evaporation of water from the never-dried fiber (Gruber et al. 2001). The spin finishing slightly retards both the equilibrium moisture content and the moisture uptake rate. It could be due to the fact that the spinning oil covers the fiber surface and interrupts the moisture sorption into the fiber.

The water accessibilities of the cellulosic fibers are also affected by the cationization and the alternation of the shape in cross section. The total equilibrium moisture content of the cationized viscose and the viscose with the trilobal cross section is higher than the untreated viscose because of their larger quantity of sorption sites due to the incorporation of a cationic polymer and the higher surface area due to the trilobal cross section. However, the amount of the site for fast sorption is reduced by those fiber modifications although the rate of the fast sorption is elevated.

Conclusions

In the present study, the water accessibilities of the thirteen different cellulosic materials with various characteristics were quantitatively investigated by gravimetrical method. The relative hysteresis between moisture sorption and desorption cycle, BET surface volume for moisture sorption, the retention capacity of liquid water, and the PEK coefficients were estimated and their relations were discussed. The total amount of moisture sorption ($M_{\mathrm{inf(total)}}$) at 60% RH and 20 °C increases in proportion to V_{m} and WRV for all materials used in the experiments except in Pp having high amorphous traction. The relative hysteresis is independent on $M_{\mathrm{inf(total)}}$ and the value is 45% for all lyocell fibers except for CV fibers.

The effects of the various properties of the specimen on the water accessibilities were also investigated in details. The total equilibrium moisture content of CV fiber is higher but the moisture uptake rate is slower than those of the lyocell and the poplar fibers. The knit and yarn lyocell show higher equilibrium moisture content and lower moisture uptake rate in comparison with the lyocell fiber. There is no significant effect of crosslinking treatment on the moisture uptake rate though it enhances the equilibrium moisture content. The effect of the temperature is remarkable. The drying the fiber at 65% RH and 20 °C accelerates both the equilibrium moisture content and the moisture uptake rate. The spin finishing retards both of the moisture accessibilities. The cationization and modification of the cross section also promote the equilibrium moisture content. However, the partial amount of fast sorption is reduced although the rate of the fast sorption is elevated by those fiber modifications. The thicker lyocell fiber shows higher total equilibrium moisture content than the thinner one. The partial amount of fast moisture sorption is larger in the thinner lyocell fiber while that of slow sorption is larger in the thicker one. Contrarily, the rate of fast moisture sorption is higher in the thicker lyocell while that of slow sorption is higher in the thinner lyocell fiber.

Considering the morphology of the materials, the kinetic results of the moisture sorption/desorption suggest that: (1) The increase in amorphous fraction, pore volume, fiber surface area, additional sorption sites and the decrease in orientation of crystalline/amorphous regions mostly heighten the total extent of moisture sorption while the coverage of fiber with spin finishing agents reduces it. (2) The increase in pore volume, fiber surface area, additional sorption site and the decrease in orientation of crystalline/amorphous regions raise the moisture sorption rate while the increase in amorphous fraction and the coverage of fiber with spin finishing agents diminish it. (3) Regarding the fast and slow sorption reactions, no effect of the fiber surface area and additional sorption sites on the slow sorption rate is observed while the increase in those factors decreases the equilibrium amount of fast sorption. Further experiments especially concerning the morphological characteristics of the fiber materials could lead to more details of the moisture sorption mechanism in the cellulosic fibers.

Acknowledgements

We are grateful to the CD-Research Society/ Vienna and Lenzing AG for financial and material support.

Appendix

Symbols		
RH	Relative humidity	%
BET	Brunauer, Emmett and Teller	
PEK	Parallel exponential kinetics	
t	Time	min
M_t	Mass change at t	%
$M_{\mathrm{inf(total)}}$	Total moisture regain at equilibrium state	%
M_{inf1}	Mass change at equilibrium state in fast sorption-desorption	%
M_{inf2}	Mass change at equilibrium state in slow sorption-desorption	%
τ_1	Characteristic time to obtain 63% M_{inf1}	min
τ_2	Characteristic time to obtain 63% M_{inf2}	min
WRV	Water retention value	g/g
V_m	BET surface volume	%

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