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Carbohydrate Polymers 58 (2004) 293–299

Carbohydrate Polymers

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A kinetic study of moisture sorption and desorption on lyocell fibers

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Received 22 March 2004; revised 28 June 2004; accepted 7 July 2004
Available online 7 August 2004

Abstract

Dynamic water vapor sorption on lyocell and cotton fibers was gravimetrically investigated at 20 °C. Lyocell fiber showed a higher equilibrium moisture regain of 9.23$\%_{w/w}$ at 60% relative humidity compared to 5.54$\%_{w/w}$ for the cotton. The hysteresis between the sorption and desorption isotherms for lyocell was 46.9% and higher than 24.7% for cotton. The hysteresis decreased with increasing relative humidity of the atmosphere. A good fit of the experimental data with the parallel exponential kinetics model suggests that moisture exchange on lyocell and on cotton is based on two different mechanisms. The kinetic parameters for the identified components of slow and fast sorption were estimated from the simulations and moreover the BET surface volume was calculated. The mechanism of water vapor sorption on cellulosic fibers are discussed considering the effects of the relative humidity on the kinetic parameters, water retention capacity, BET surface volume and the hysteresis effect.

Keywords: Lyocell; Moisture sorption; Hysteresis; Water retention; PEK model; BET model

1. Introduction

An important function of textiles is supplying a maximum of wearing comfort to human bodies. Clothing materials should thus have a high moisture retention capacity and high moisture transportation properties to maintain a constant temperature and humidity between skin and fabric. This is based on the fact that the moist fibers can act as a heat reservoir. Moreover, moisture also changes the fiber properties. The moisture uptake causes a swelling of the hygroscopic fibers, which is a dimensional change due to breaking of inter- and intramolecular hydrogen bonds between the cellulose molecules (Gruber, Schneider, & Schempp, 2001). Not only physical properties such as density, shape, stiffness and crystal structure of the fibers but also mechanical properties, e.g. fiber–fiber friction (Campos & Bechtold, 2003), tensile modulus and breaking stress (Ibbett & Hsieh, 2001) are altered by water sorption. This strongly affects also the general dyeing behavior of the fibers and the finishing processes of textiles, e.g. resin-coating and crosslinking treatments.

The lyocell fiber, which is a regenerated cellulosic fiber manufactured by means of *N*-methyl morpholine-N-oxide dissolution followed by coagulation, has a high crystallinity and fibrillar morphology and offer different characteristics compared to cotton fibers (Bartsch & Kling, 2001; Ibbett & Hsieh, 2001). The distinct fibrillar structure results in a high fibrillation of the fibers inducing, e.g. rope marking defect in hank finishing, graying of dyed fabrics and a change of handle of clothes (Rohrer, Retzl, & Firgo, 2001). The fibrillation occurs when water is adsorbed in the lyocell fibers which cause a change in the fiber structure. Hence, understanding the mechanism of water sorption on lyocell fibers may provide hints to improve not only the wearing comfort but also their textile processing.

A mechanism of moisture sorption especially in hydrophilic fiber materials, e.g. cellulosic fibers are comparatively complex because it involves a continuous change of the structure of the fiber owing to swelling. High internal temperature change caused by heat of sorption with large

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doi:10.1016/j.carbpol.2004.07.004

amount of moisture also complicates kinetics of the moisture sorption (Morton & Hearle, 1997). Various theories have been proposed and modified since 1893 to describe the sorption mechanisms of individual fiber material (Barrer, 1947; Hill, 1950; Taylor, 1954). Brunauer et al., derived a model for multi-layer adsorption (Brunauer, Emmett, & Teller, 1938) and Langmuir developed the classical model for adsorption isotherms which is applicable for gases adsorbed in a monolayer on material surfaces (Langmuir, 1918). Peirce (1929) introduced a model which is based on the assumption of direct and indirect sorption of water molecules on attractive groups of the materials and a theory, in which the interaction between water and the binding sites considers three types of water with different associating strength was proposed by Speakman (1944). Young and Nelson developed a complete sorption–desorption theory, starting from the assumption of a distinct behavior of bound and condensed water (Young & Nelson, 1967).

In the present study, we discuss the results of quantitative and kinetic investigations of moisture adsorption in a man-made cellulosic fiber lyocell by using a parallel exponential kinetics (PEK) model proposed from the Young-Nelson model by Kohler, Dueck, Ausperger, & Alex, (2003). A mechanism of water adsorption onto lyocell is applied considering BET surface volume, water retention capacity and hysteresis between the moisture regain isotherms and compared to those of cotton fibers.

2. Experimental

2.1. Materials

Staple lyocell and cotton fibers supplied by Lenzing AG, Austria were used for the experiments. The titer of the lyocell fiber was 1.3 dtex and length was 38 mm.

2.2. Measurements

2.2.1. Dynamic water vapor sorption and desorption

Approximately 1.0 g of each fiber material in an aluminum sample dish (diameter: 50 mm) was placed in an automatic multisample moisture sorption analyzer SPS11 (Project-Messtechnik, D-Ulm). 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 11 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 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 ±0.5% (±1% at 80 and 90% RH).

2.2.2. Water retention capacity

0.5 g of fiber was soaked into distilled water for 24 h at ambient temperature. After centrifugation at 4000 *g* for 10 min, the weight of the fiber was measured (W_w) The sample was dried at 105 °C for 4 h and the sample was reweighed (W_d). The water retention value (WRV) was calculated from Eq. (1).

$$\mathrm{WRV}\ (\mathrm{g/g}) = (W_w - W_d)/W_d \tag{1}$$

3. Results and discussion

Moisture regain and loss on the fibers during sorption and desorption experiments were gravimetrically measured using the automatic moisture sorption analyzer. The time versus mass change (M_t) profile of a full sorption/desorption is shown in Fig. 1 (lyocell fiber). The moisture regain increased or decreased remarkably as the relative humidity was changed and then the increase or the decrease was gradually reduced until the sorption or desorption reached an equilibrium state over the whole humidity range. The fibers clearly reach an equilibrium state at each moisture interval. The values of the individual equilibrium states were plotted against regain. The equilibrium values of moisture content at each interval ($M_{\mathrm{inf(total)}}$) are plotted against the relative humidity to draw the equilibrium isotherm shown in Fig. 2. The equilibrium curves have a sigmoidal shape due to a stronger moisture uptake or release at very low or very high relative humidities which is consistent with the behavior of

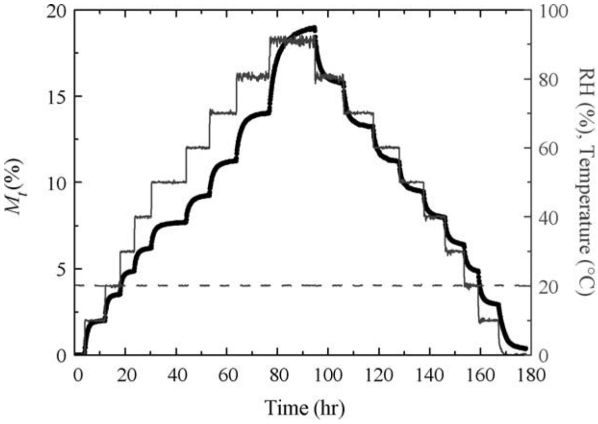


Fig. 1. Mass change over time profile of the full sorption/desorption cycle (interval method) for lyocell (●) at 20 °C (– – – –); the steplike curves shows the course of the relative humidity.

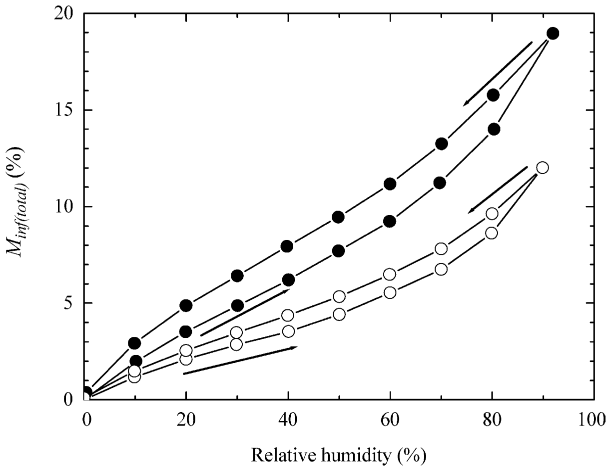


Fig. 2. Equilibrium moisture sorption and desorption isotherms of lyocell (●) and cotton (○) at 20 °C.

other polymer materials (Urquhart & Eckersall, 1930). 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). Generally, the hysteresis is calculated as the difference between $M_{\text{inf(total in desorp.)}}$ and $M_{\text{inf(total in sorp.)}}$ that are the absolute equilibrium mass values of adsorbed water in the material of the desorption and sorption process respectively at the same relative humidity. However, this value could include the effect of moisture contents in the fibers. The degree of the hysteresis was calculated according to Eq. (2) to evaluate the extent of specific structural change.

$$\begin{aligned}&\text{Hysteresis (\%)}\\&\quad = 100 \times (M_{\text{inf(total in desorp.)}}\\&\qquad - M_{\text{inf(total in sorp.)}})/M_{\text{inf(total in sorp.)}} \qquad (2)\end{aligned}$$

The calculated data are plotted against relative humidity in Fig. 3. The extent of the hysteresis clearly decreases with increasing relative humidity, which suggests that the fiber structure strongly changes when moisture adsorbs on the dry fiber or reversely when all moisture desorbs from the fiber. Obviously, this structural change becomes smaller after some moisture have been absorbed on the fiber. The lyocell fiber shows a more distinct hysteresis than the cotton fiber especially at low humidities. This result indicates that the fiber structure of lyocell is less stable than that of cotton and water can more easily invade the structure which can be explained by the lower crystallinity (Lenz, Schurz, & Eichinger, 1994), larger pore volume and inner surface area of lyocell (Brederick, Gruber, Otterbach, & Schulz, 1996).

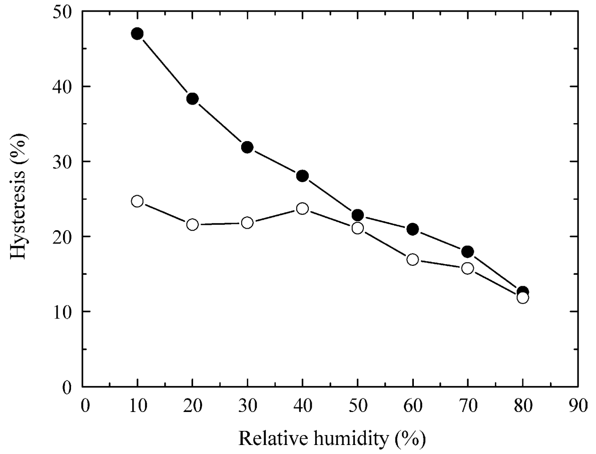


Fig. 3. Effects of relative humidity on hysteresis between sorption and desorption isotherms for lyocell (●) and cotton (○) at 20 °C.

Kohler et al. have reported that water exchange on natural fibers such as flax and hemp can be modeled by two parallel independent first order processes, which was defined PEK model (Kohler et al., 2003). The model offered two different mechanisms for the exchange of water vapor relating to different sorption sites. According to their method, experimental data of the moisture content is simulated as a function of time using Eq. (3), where M_t and M_{inf} are the mass change at any time t and at equilibrium state. τ is a characteristic time to obtain approximately 63% of M_{inf}.

$$M_t = M_{\text{inf1}} \cdot (1 - e^{-t/\tau 1}) + M_{\text{inf2}} \cdot (1 - e^{-t/\tau 2}) \qquad (3)$$

$$M_{\tau 1(\tau 2)} = M_{\text{inf1(inf2)}} \cdot \left(1 - e^{-1}\right) \approx 0.6321 \cdot M_{\text{inf1(inf2)}} \qquad (4)$$

Subscripts 1 and 2 indicate the two kinetic processes defined as fast and slow, corresponding to slow and fast sorption sites. The fast and slow sorption sites can be related to different types of amorphous regions, external/internal fiber surfaces and direct/indirect sorption (Morton & Hearle, 1997) as shown in Fig. 4. Water molecules adsorb directly on hydroxyl groups of external surface, amorphous regions, inner surface of voids and crystallites. Additional water molecules can adsorb on the water molecules binding the fiber directly. The direct sorption of the water molecules onto the external surface and amorphous regions could be fast while the indirect sorption onto the inner surface and crystallites could be relatively slow.

A result of the simulation for lyocell is shown in Fig. 5. The simulation curve shows a good fit with experimental data, which suggests that PEK is a proper model to describe water sorption of the lyocell fiber. Generally in any sorption kinetics the structure and size of sample has to be taken in account. Particularly during sorption processes physical sample structure (fiber, yarn and fabric) could influence the final result, thus in this study the same mass of fibrous materials with similar physical structure (diameter and length) were selected.

PEK parameters M_{inf1} and M_{inf2} were calculated for at each moisture interval. The value of M_{inf} was summed from 0% RH to a given relative humidity ($M_{\text{inf(total)}}$) and plotted against the relative humidity in Fig. 6 for lyocell and cotton

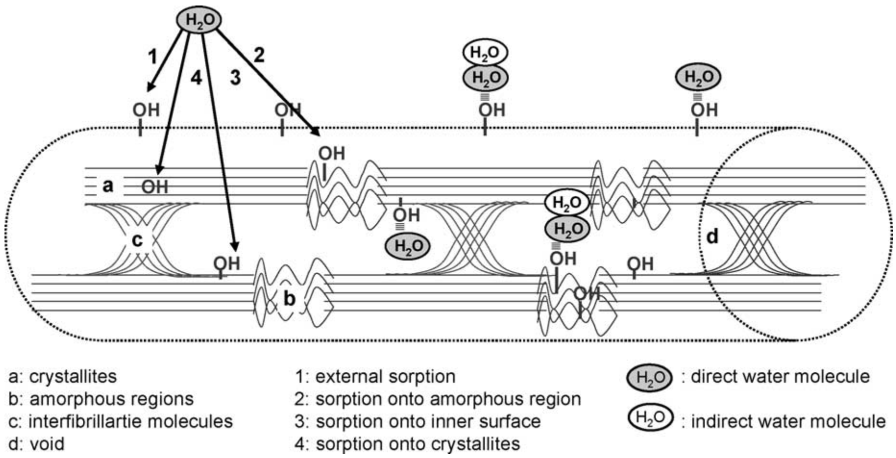


Fig. 4. A schematic diagram of direct and indirect moisture sorption onto external surface (1), amorphous regions (2), inner surface of voids (3), and crystallites (4).

fibers, respectively. The individual fast and slow sorption isotherms, displayed as triangles and inverted triangles respectively, were obtained as components of the total isotherms by the PEK model. The fast and slow processes occur simultaneously and the fast component clearly dominates over the slow one in the whole relative humidity range.

In order to analyze the effect of the humidity on the moisture sorption behavior in more detail, the PEK parameters obtained at each sorption steps are plotted against relative humidity in Fig. 7. The τ_1 and τ_2 values decrease with increasing relative humidity up to 30–40% RH, which implies that water slowly adsorbs onto dry fibers regardless of the type of the process. However, once the position of water molecules advancing front is determined, further water molecule are easily and promptly adsorbed in the moist fiber until the moisture uptake reaches a certain level (Hermans & Vermaas, 1946). Once this level has been achieved, the rate of water sorption was gradually retarded with increasing relative humidity up to 90% RH. Considering that M_{inf1} and M_{inf2} equilibrium moisture sorption of the slow and fast processes increase when the humidity exceeds 40% RH (Fig. 7), the retardation of the sorption rate might be explained by the large heat evolution which is

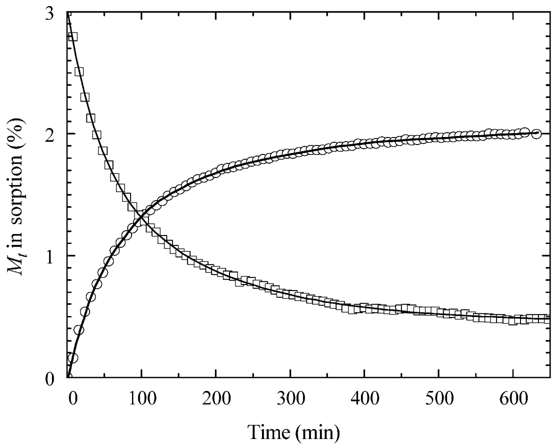


Fig. 5. PEK simulations of experimental data of moisture regain (○) and loss (□) for lyocell at 70% RH and 20 °C. Results of simulation are shown in solid lines.

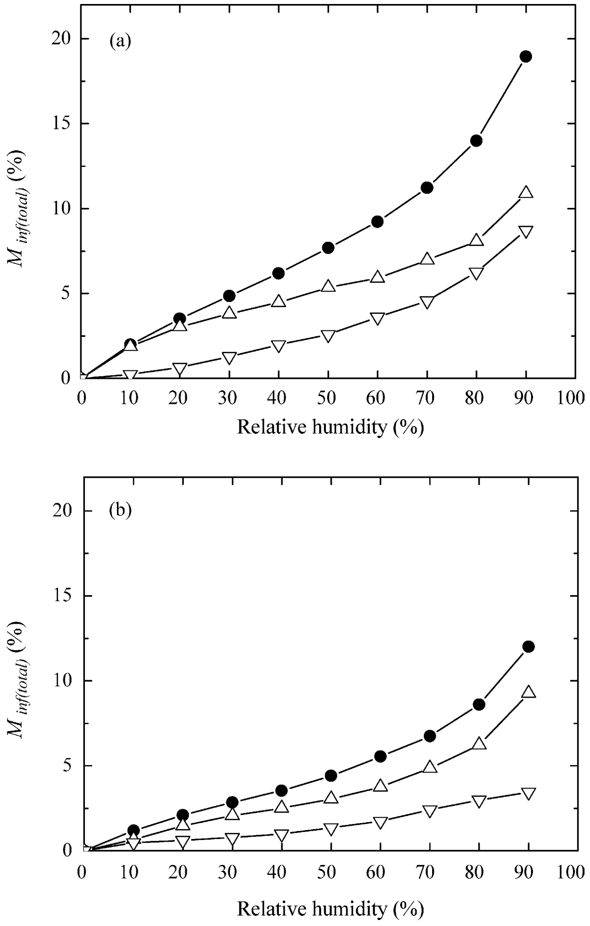
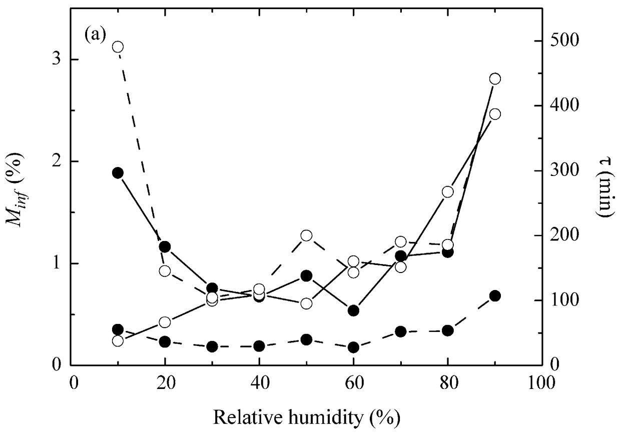


Fig. 6. Isotherm components in moisture sorption for lyocell (a) and cotton (b) at 20 °C. Total moisture regain $M_{\mathrm{inf(total)}}$ (●), moisture regain in fast reaction $M_{\mathrm{inf1(total)}}$ (△) and in slow reaction $M_{\mathrm{inf2(total)}}$ (▽) were obtained by PEK simulations.



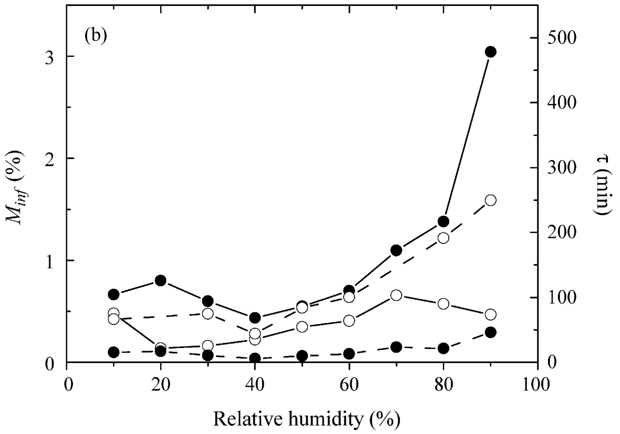


Fig. 7. Effects of relative humidity on PEK parameters for lyocell (a) and cotton (b) at 20 °C. The PEK parameters M_{inf1} (●), M_{inf2} (○), τ_1 (●) and τ_2 (○) were obtained by PEK simulations.

generated when water molecules adsorb on the fibers (Watt & McMahon, 1966). Further experiments are required to clarify the effect of heat of sorption on the kinetics. The decrease of the equilibrium amount of sorbed moisture of the fast process below 40% of RH indicates that a large amount of moisture is adsorbed on the dry fiber because the sites for water sorption are completely free. However, after some water molecules have occupied the most reactive sites of the cellulose, the attraction of additional water molecules are hardly adsorbed in the fiber. The adsorbed moisture induces structural changes in the fiber such as swelling and some internal rearrangement of the cellulose molecules. Thus with increasing water uptake the fiber structure gets looser and at higher water vapor pressures water molecules can easily invade the structure.

In comparison of M_{inf1} with M_{inf2}, and M_{inf1} the component of fast sorption dominated over that of slow sorption process especially at lower relative humidities. This suggests that water molecules predominantly adsorb directly on external surfaces and on the low-ordered amorphous regions. Less moisture penetrates into internal surfaces and in high-ordered amorphous regions at low relative humidities. The cotton fibers show a similar tendency as the lyocell fibers concerning τ_1, τ_2, M_{inf1} and M_{inf2} except at low relative humidities. As the hysteresis at low relative humidity (Fig. 3) is smaller in cotton than in lyocell, the effects of relative humidity on the PEK parameters and the moisture sorption is less distinct. The rates of the sorption process are more or less constant at low relative humidities because of smaller structural changes of cotton.

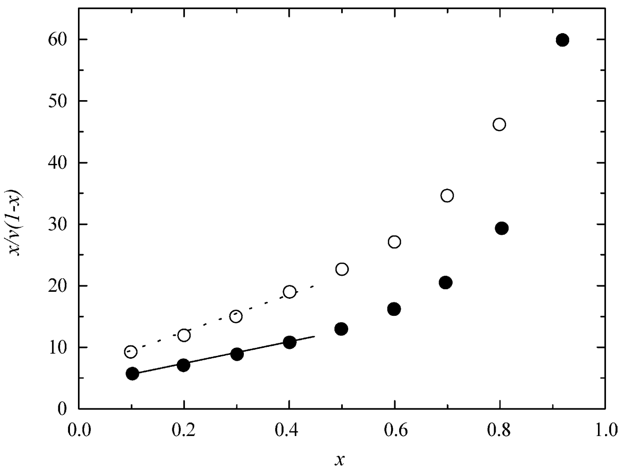


Fig. 8. BET plot of moisture regain for lyocell (●) and cotton (○) fibers at 20 °C. Symbols x and V are partial pressure of atmosphere surrounding fiber and moisture regain at x.

The quantity of moisture adsorbed as a monomolecular layer on the surface of the material (V_m) was determined by the Brunauer–Emmett–Teller (BET) Eq. (5),

$$(x/100)/V(1 - x/100) = 1/V_m c + (c - 1)(x/100)/V_m c \qquad (5)$$

where x, is the partial vapor pressure of water, V the moisture regain at x and where c is a constant (Brunauer et al., 1938). The BET plot for lyocell fiber is given in Fig. 8. The curve between 0.1 and 0.5 of x is almost linear, which suggests that the BET model fits to the moisture sorption data of the lyocell fiber. The V_m calculated from the slope and the intercept of the y-axis is given in Table 1 along with other parameters indicating water accessibilities.

At 30–40% RH a V_m of 5.0 and 2.9 was obtained for lyocell and cotton respectively, which corresponds to the inflection points in the curves of the PEK model. That means that the mechanism of water sorption changes after the fiber surface is occupied by a monolayer of water molecules. The fact that the difference of the hysteresis extent between lyocell and cotton remarkably decreases at 40% RH, also proves a change of the mechanism at the point of V_m.

The total moisture regains at 10, 60 and 90% RH, V_m and WRV are higher for lyocell than for cotton, which indicates that lyocell has quantitatively higher water

Table 1
Characteristic parameters of water accessibility, PEK model parameters and BET surface volume for lyocell and cotton fibers at 20 °C

RH (%)	$M_{\mathrm{inf(total\ in\ sorp.)}}$ (%)		Hysteresis (%)		M_{inf1} (%)		τ_1 (min)		M_{inf2} (%)		τ_2 (min)		V_{m} (%)		WRV (g/g)	
	Lyocell	Cotton	Lyocell	Cotton	Lyocell	Cotton	Lyocell	Cotton	Lyocell	Cotton	Lyocell	Cotton	Lyocell	Cotton	Lyocell	Cotton
10	1.99	1.19	46.9	24.7	1.89	0.67	55	15	0.24	0.48	491	67				
60	9.23	5.54	21.0	21.1	0.54	0.70	25	13	1.02	0.41	143	100	5.03	2.86	0.638	0.555
90	18.9	12.0	12.6	11.8	2.81	3.04	107	46	2.46	0.47	442	–				

$M_{\mathrm{inf(total)}}$, M_{inf} and τ were obtained at 60% RH, and hysteresis was obtained at 10% RH.

accessibility than cotton does. However, as the slow and the fast kinetic processes are considered, M_{inf1} of lyocell at 10% RH (1.89%) is higher than that of cotton (0.67%) though M_{inf1} of lyocell at 60 and 90% RH (0.54, 2.81%) are lower than that of cotton (0.70, 3.04%). Contrarily, M_{inf2} of lyocell at 10% RH (0.24%) is lower than that of cotton (0.48%) though M_{inf2} of lyocell at 60 and 90% RH (1.02, 2.46%) are higher than that of cotton (0.41, 0.47%). These results indicate that the cotton fibers adsorb water molecules evenly on both sites relating to fast and slow sorption at low humidities while the lyocell fibers adsorb the water molecules predominantly on the site relating to fast sorption.

The cotton fiber showed small τ_1 and τ_2 at any humidity, meaning that the water sorption in both fast and slow processes is fast though it has small surface area compared to the lyocell. This suggests that the cotton has dynamically high water accessibility. Further experiments are required to clarify the reason for the different kinetics between lyocell and cotton fibers.

4. Conclusion

The present study deals with the kinetics of dynamic water vapor sorption and desorption of lyocell and cotton using a PEK model. The simulation curves showed good fits with the experimental data of moisture regain, suggesting that the water exchange of lyocell and cotton is based on two different mechanisms occurring simultaneously in slow and fast processes. The kinetic parameters showed a strong dependence on the relative humidity of atmosphere but this dependence changed at the relative humidity where a monolayer of adsorbed water molecules (V_{m}) is achieved. This indicates that the mechanism of water sorption in lyocell fibers altered when the surface is occupied by a monolayer of water molecules. At moisture contents below V_{m}, the water vapor adsorption and/or penetration at low relative humidities (dry fiber) is slow in both, fast and slow processes of the PEK model. Once the fiber is moistened, the sorption and/or the penetration rate increases but decreases again when the adsorbed moisture exceeds V_{m}. The larger value of M_{inf1} (1.89%) for lyocell at 10% RH suggests larger quantity of the sorption site relating to the fast moisture sorption compared to M_{inf1} (0.67%) for cotton. This agreed with the large V_{m} (5.03%) of lyocell as compared to the value of 2.86% of the cotton fiber. Contrarily, M_{inf2} of cotton at 10% RH was larger than that of lyocell, which implies that the quantity of the sites relating to slow sorption for the cotton fiber is larger than for lyocell fiber. The larger values of V_{m} and $M_{\mathrm{inf(total\ in\ sorp.)}}$ of the lyocell than those of the cotton intimates that the lyocell fiber has quantitatively higher water accessibility. The smaller τ_1 and τ_2 of the cotton than those of the lyocell fiber indicates that cotton fiber has dynamically higher water accessibility than the lyocell.

Acknowledgements

We are grateful to the CD-Research Society/Vienna and Lenzing AG for financial and material support, and to Dr Robert Kohler, FH-Reutlingen, University of Applied Sciences for his insightful comments.

Appendix A

PEK	parallel exponential kinetics
BET	Brunauer, Emmett and Teller
RH	relative humidity (%)
W_{w}	weight of fiber after centrifuge (g)
W_{d}	weigh of fiber after drying (g)
WRV	water retention value (g/g)
M_{t}	mass change at t (%)
$M_{\mathrm{inf(total\ in\ desorp.)}}$	equilibrium moisture regain in desorption (%)
$M_{\mathrm{inf(total\ in\ sorp.)}}$	equilibrium moisture regain in sorption (%)
$M_{\mathrm{inf(total)}}$	total moisture regain at equilibrium state (%)
$M_{\mathrm{inf1(total)}}$	total moisture regain at equilibrium state in fast sorption (%)
$M_{\mathrm{inf2(total)}}$	total moisture regain at equilibrium state in slow sorption (%)
M_{inf1}	mass change at equilibrium state in fast sorption–desorption (%)
M_{inf2}	mass change at equilibrium state in slow sorption–desorption (%)
t	time (min)
τ_1	characteristic time to obtain 63% M_{inf1} (min)
τ_2	characteristic time to obtain 63% M_{inf2} (min)
M_{t}	mass change at t (%)
V_{m}	BET surface volume (%)
V	equilibrium moisture regain at x (g/g)
x	partial pressure
c	BET constant

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