Carbohydrate Polymers 82 (2010) 761–767

Contents lists available at ScienceDirect

Carbohydrate Polymers

journal homepage: www.elsevier.com/locate/carbpol

Swelling and dissolution mechanism of regenerated cellulosic fibers in aqueous alkaline solution containing ferric tartaric acid complex: Part I. Viscose fibers

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ARTICLE INFO

Article history:
Received 15 January 2010
Received in revised form 9 April 2010
Accepted 24 May 2010
Available online 1 June 2010

Keywords:
Cellulose
FeTNa
Fiber
Iron complex
Viscose
Swelling

ABSTRACT

Morphological studies of swollen viscose fibers in FeTNa (Fe–tartaric acid–NaOH complex solution) were investigated by means of microscopy. The molar ratio of Fe:tartaric acid:NaOH in FeTNa solutions was kept constant at 1:3.28:11.84 while Fe concentration ranged from 0.15 to 0.55 M and free NaOH concentration varied from 0.4 to 5 M. Fiber diameter measurements following 2 min of swelling and swelling rate of fibers up to 60 min were studied. Swelling, splitting and dissolution of viscose fibers were observed depending on concentration of Fe and free NaOH in FeTNa solutions and fiber swelling time. Splitting of a fiber into its fibrils was achieved by only swelling the fiber without a usage of any kind of force.

1. Introduction

Cellulose is the most abundant renewable organic polymer. It can be derivatized into ethers/esters and also can be converted to fibers, films, food casings, membranes, sponges, etc. (Klemm, Heublein, Fink, & Bohn, 2005). The commercial way to produce cellulosic fibers, films, non-woven fabrics is the viscose method (Cross et al., 1892) and also cuprammonium rayon method (Despeissis, 1890) for a limited amount. Modal fibers are produced via viscose method by the addition of various chemicals to viscose and coagulation bath (Cox, 1950). Due to technological complexity and generation of hazardous byproducts of these methods, an environmentally more friendly process, Lyocell, was developed (Franks & Varga, 1980). Cellulose is dissolved in *N*-methylmorpholine-*N*-oxide (NMMO) to produce lyocell fibers, which have better mechanical properties than that of viscose and cuprammonium fibers (Liu, Shao, & Hu, 2001; Woodings, 1995). But lyocell process suffers from high energy costs and high fibrillation tendency of lyocell fibers. An alternative derivatization method to viscose method, CarbaCell process, does not use sulfur-containing compounds and can use existing viscose spinning systems to produce carbamate fibers (Ekman et al., 1986; Klemm et al., 2005). A novel solvent system for cellulose, i.e. NaOH/urea aqueous solution pre-cooled to −12 °C, was developed to dissolve cellulose at ambient temperature (below 20 °C) to produce multifilament fibers (Cai et al., 2007; Qi, Cai, Zhang, Nishiyama, & Rattaz, 2008; Zhang, Cai, & Zhou, 2005). The carbamate and NaOH/urea processes to produce regenerated cellulosic fibers are still under extensive development and may play an important role in the future (Lewin, 2007).

Lyocell fibers have circular cross-section, while viscose and modal fibers have lobular cross-section (Krässig, Schurz, Steadmann, Schliefer, & Albrecht, 1986). The fibrillation tendency of fibers, which occurs due to the high orientation of fibrils, increases in this order: modal < viscose < lyocell (Sisson, 1960; Lenz, Schurz, & Wrentschur, 1993). Applying transmission electron microscopy (TEM) on ultra-thin cross-sections of lyocell fibers, nanopores in the bulk of the fiber with a slight gradient in pore density and a very porous skin layer was observed. In viscose and modal fibers, a very wide pore size distribution from nanometer to micrometer size was seen (Abu-Rous, Ingolic, & Schuster, 2006). Accessible pore volume of the fibers measured by inverse size exclusion chromatography increases in this order: modal < lyocell < viscose (Öztürk, 2008). The crystallinity (Schurz, 1994) and degree of polymerization (Kreze, Strnad, Stana-Kleinschek, & Ribitsch, 2001) increase in this order: viscose < modal < lyocell.

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doi:10.1016/j.carbpol.2010.05.048

Swelling investigations of cellulosic fibers have been studied by means of microscopy. The aim to study the swelling and dissolution of fibers is to find out the fiber morphology in different solutions. Schwertassek (1958) studied the swelling of cellulose fibers in the presence of Procion dyes to find out the dyeing mechanism. Rockstroh (1960) investigated the swelling mechanism of regenerated cellulosic fibers in copper-oxide-ammonia. Mares (1956) studied the swelling of the skin of viscose fibers in Marshall solvent. Lately, metal complex solvents such as Cadoxen and FeTNa have been used for swelling analyses of cellulosic fibers by means of microscopy. Compared to other aqueous metal complex solvents, FeTNa solution has more advantages to be used in the swelling-dissolution studies and viscosimetric analyses of cellulosic fibers owing to being more insensitive to oxidation by air and being odorless (Jayme & Bergmann, 1956).

The optimum combining molar ratio of Fe to tartrate in the FeTNa solution was found to be 1:4.5. The 1:3 molar ratio is found to be the most stable one and gives a green color which is used as solvent for wood pulp and other cellulosic materials (Hanby & Johnson, 1969). The ligand deficiency at 1:3 molar ratio of Fe:tartrate is filled by the two adjacent hydroxyl groups on carbon atoms two and three on the glucopyranoside repeating unit of cellulose to fulfill 1:4.5 molar ratio which is the optimum one. This leads to the dissolution of cellulose (Bayer, 1964). In the dissolution state of cellulose, the binding of Fe metal to cellulose has been analytically verified to have a molar ratio of bound iron to glucose units as 1:3. The location of Fe on cellulose is along the chains and has been observed without an indication of interchain crosslinks. The polychelate chain contains three successive glucose monomers with a replacement of their hydroxyl pairs with three FeTNa complexes of the original solvent. This implies that 2 out of 3 successive monomer–monomer bonds are immobilized which results in an increase in stiffness and a decrease in chain length (Valtasaari, 1971).

Mainly two methods, namely Jayme and Valtasaari methods, to prepare FeTNa solutions were used for the dissolution studies of cellulosic materials. Both Jayme and Valtasaari methods use 1:3:13 molar ratio for Fe:sodium tartrate dihydrate:NaOH, but the former one requires 0.5 M Fe in the presence of 2 M free NaOH, while the latter method requires 0.3 M Fe in the presence of 1.5 M free NaOH (Jayme & Bergmann, 1957; Valtasaari, 1957). FeTNa solution prepared according to Jayme method (Jayme & Bergmann, 1957) was used for the morphological structure analyses of viscose and modal fibers (Theidel, 1962), viscose cord fibers modified with various chemicals (Kling, Mahl, & Heumann, 1963). Valtasaari method (Valtasaari, 1957) was followed to prepare FeTNa solutions to study the morphological changes of modal fibers (Hoffrichter, 1963), viscose fibers (Wünsch & Hoffrichter, 1962).

The swelling studies of lyocell, viscose and modal fibers in the presence of various alkali solutions were investigated by means of splitting test and fiber diameter measurements. Split number of lyocell fibers was found to be the highest, while modal fibers has the least. It was due to the fibrillar structure of lyocell fibers enabling them to split into more fibrils than viscose and modal fibers can, which have lobular structure. The higher number of lobes of viscose fibers compared to that of modal fibers was found to be responsible for its higher split number (Öztürk & Bechtold, 2008). As a following study, the effect of NaOH concentration on *lyocell* fibers in the presence of FeTNa solution was studied (Vu-Manh, Öztürk, & Bechtold, 2010).

The key parameter for swelling and dissolution mechanism of cellulosic fibers was mentioned to be the morphology of the fiber. Hence Cuissinat et al. studied the swelling and dissolution of cotton and wood fibers in NMMO–water mixtures (Cuissinat & Navard, 2006a), NaOH–water (Cuissinat & Navard, 2006b) and ionic liquids (Cuissinat, Navard, & Heinze, 2008).

The aim of the current study is to show the structural changes of *viscose* fibers in various FeTNa solutions as well as to identify suitable FeTNa treatment conditions. The FeTNa system allows a high number of degrees of freedom in the concentration of environmentally safe chemicals, i.e. tartaric acid, Fe and free NaOH. Thus it can be a useful system for the cellulose fiber processing in the alkaline media. Fiber diameter measurement following swelling ca. for 2 min and swelling rate of fiber within 60 min were conducted to assess the potential of FeTNa system for textile processing.

2. Experimental procedure

2.1. Materials

Viscose staple fibers (1.3 dtex titer and 38 mm length) without spin finishing were provided from Lenzing AG. Analytical grade sodium hydroxide NaOH (>98%) from Fluka; iron (III) chloride-6 hydrate, $FeCl_3{\cdot}6H_2O$, (>99%) from Riedel-de Haen AG; tartaric acid, $C_4H_6O_6$, (>99.5%) from Merck and research grade sorbitol ($C_6H_{14}O_6$) from Serva-Feinbiochemica GmbH & Co were used.

2.2. Methods

2.2.1. Preparation of FeTNa solution

FeTNa solutions with a molar ratio of $FeCl_3{\cdot}6H_2O$:tartaric acid:NaOH as 1:3.28:11.84 were used. The concentration of Fe ion varied from 0.15 to 0.55 M. In addition, 0.4, 0.8, 1.25, 2.5 and 5 M free NaOH were used.

All FeTNa solutions were prepared by 'direct procedure' without isolation of an intermediate (Bayer, 1964; Valtasaari, 1957). A dark bottle which contains magnetic stirring bar was used to exclude light. A weighed amount of tartaric acid was dissolved in slightly more than the minimum needed amount of water. Ferric (III) chloride was quantitatively added to this solution with a few milliliters of water. After brief agitation, the dark bottle was introduced into a cooling bath to decrease the temperature of the mixture to about 0 °C. NaOH solution was introduced drop-wise to the mixture. The addition rate of NaOH was adjusted to prevent the temperature of the mixture from exceeding about 15 °C.

2.2.2. Fiber diameter measurements

The fibers were swollen in the solution for approximately 2 min. The diameter of swollen fiber was measured by Reichert projection microscope with a magnification of 500×. Besides, swelling rate of viscose fibers was found out by measuring fiber diameter after its swelling in varying times: 5, 10, 15, 20, 25, 30, 45 and 60 min. 10 fibers were counted and mean value was taken for measurement.

'Sp' refers to 'splitting of a fiber into its fibrils', and the sign 'X' refers to the dissolution of the fiber for which the fiber diameter could not be measured.

3. Results and discussion

3.1. Morphological changes of viscose fibers during FeTNa treatment

During FeTNa treatment of viscose fibers, four different structural changes were found out depending on Fe and free NaOH concentration in FeTNa solution and also swelling time of fiber. The structural changes of viscose fiber are as following:

(a) limited swelling, i.e. uniform swelling at a low degree comparable to that of in water (Fig. 1: S_1),
(b) splitting of a fiber into its fibrils (Fig. 1: Sp_{1-5}),
(c) swelling (Fig. 1: S_2),
(d) dissolution.

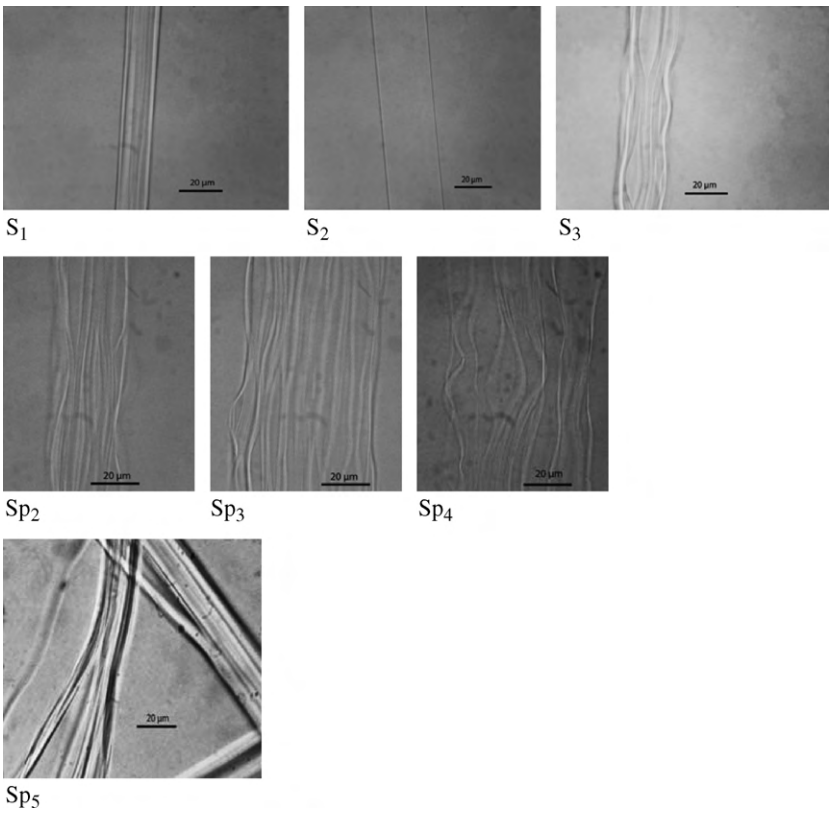

Fig. 1. Morphological changes of viscose fiber during FeTNa treatment: S_1 symbolizes limited swelling (uniform swelling at a low degree comparable to that of in pure water); S_2 shows an example for 'swelling'; Sp_{1-5} denote splitting of one fiber into its fibrils (S_1, S_2 and Sp_{1-4} show one viscose fiber swollen for ca. 5, 7, 10, 15, 30 and 45 min, respectively in FeTNa solution having 0.15 M Fe and 5 M free NaOH; Sp_5 shows viscose fibers swollen for ca. 20 min in FeTNa solution having 0.25 M Fe and 0.8 M free NaOH).

Viscose fibers have skin-core structure with a rugged surface and a folded cross-section. The core has a lower degree of orientation and crystallinity but smaller crystallite size compared to that of the skin (Krässig et al., 1986; Sisson, 1960).

In the literature, splitting of viscose fiber in FeTNa solution (1:3:6 molar ratio for Fe(III)hydroxide:tartaric acid:NaOH) into its fibrils was attributed to the high swelling of the core and lower swelling of the skin at the same time. Splitting of fibrils occurred with the increase in swelling stress (Theidel, 1962).

In the literature, splitting of regenerated cellulosic fibers (lyocell, viscose, modal) was achieved when a *swollen* fiber was placed to a crockmeter in order to achieve the required *shear force* on the fiber. Both swelling of the fiber and shear force attained by crockmeter were needed to split lyocell, viscose and modal fibers in LiOH, NaOH, KOH and TMAH (tetramethyl ammonium hydroxide) (Öztürk & Bechtold, 2008; Öztürk, Okubayashi, & Bechtold, 2006a; Öztürk, Okubayashi, & Bechtold, 2006b). Moreover, the current study shows the achievement of splitting of viscose fibers in FeTNa solution without using any force but only swelling of the fiber for a time.

3.2. Fiber diameter measurements

The effect of FeTNa solution on the fiber diameter of viscose fibers after 2 min of swelling is given in Fig. 2, while Fe and free NaOH concentration of FeTNa solution ranged 0.15–0.55 and 0.4–5 M, respectively. Independent of Fe concentration, fiber diameter in FeTNa solution having either 0.4 or 0.8 M free NaOH was found to be around 20 μm (limited swelling) which is comparable to that found in pure water. FeTNa solution having 1.25 M free NaOH in the presence of 0.15–0.25 M Fe resulted in the splitting of viscose fiber, whereas at Fe concentrations of above 0.30 M caused swelling. FeTNa solutions having a free NaOH concentration of 2.5 or 5 M also swelled the viscose fiber.

3.3. Effects of Fe concentration in FeTNa solutions on the swelling rate of viscose fibers

Fig. 3 shows the swelling rate of viscose fiber in FeTNa solution having varying Fe concentration in the presence of 0.4 M free NaOH. The swelling rate reached equilibrium after 5 min of

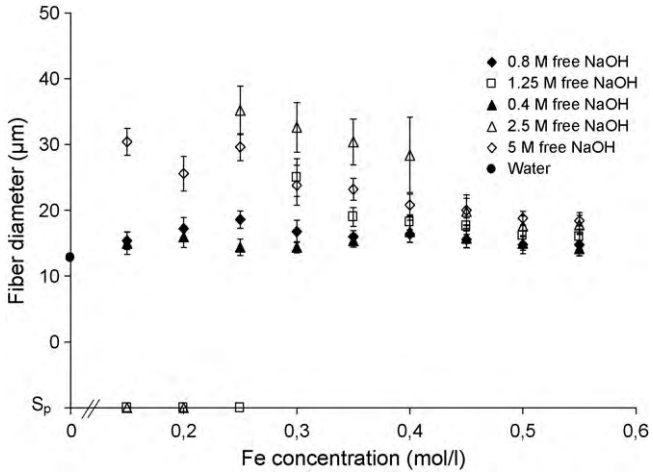

Fig. 2. Fiber diameter of viscose in FeTNa solutions containing varying Fe and free NaOH concentration (Sp = splitting).

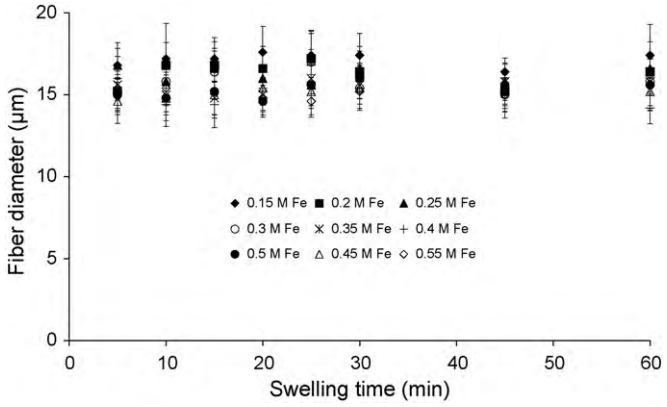

Fig. 3. Swelling rate of viscose fiber in FeTNa solutions containing varying Fe concentration in the presence of 0.4 M free NaOH.

swelling. Fiber diameter of viscose fiber was found to be comparable to the swelling degree obtained in pure water when fiber was swollen up to 60 min (limited swelling). In conclusion, this solution did not affect the fiber diameter of viscose fibers more than pure water can do. Only it took longer time (ca. 5 min) to reach equilibrium swelling compared to that of pure water (ca. 1 min).

Fig. 4 shows the swelling rate of viscose fiber in FeTNa solution having varying Fe concentration in the presence of 0.8 M free NaOH. Swelling rate of viscose fibers reached equilibrium after 5 min of swelling in FeTNa solutions having a Fe concentration of above 0.40 M. It was limited swelling which is comparable to the swelling in pure water. FeTNa solutions having a Fe concentration lower than 0.35 M resulted in a higher fiber diameter, but with increase in swelling time splitting of fibers were observed.

Fig. 5 shows the swelling rate of viscose fiber in FeTNa solution having 1.25 M free NaOH in the presence of varying Fe concentration. Swelling rate of viscose fibers reached equilibrium after 5 min of swelling in FeTNa solutions having a Fe concentration of above 0.50 M. It was limited swelling which is comparable to the swelling in pure water. FeTNa solutions having a Fe concentration lower than 0.45 M resulted also in limited swelling, but with increase in swelling time splitting of fibers were observed.

Low concentrations of Fe are required in FeTNa solutions when free NaOH concentration is 0.8 and 1.25 M in order to affect the fiber diameter, i.e. in order to have interaction between solution and the fiber. For example, Fe concentrations lower than 0.35 M is required in FeTNa solutions in the presence of 0.8 M free NaOH. Fe concentrations lower than 0.45 M is required when the free NaOH

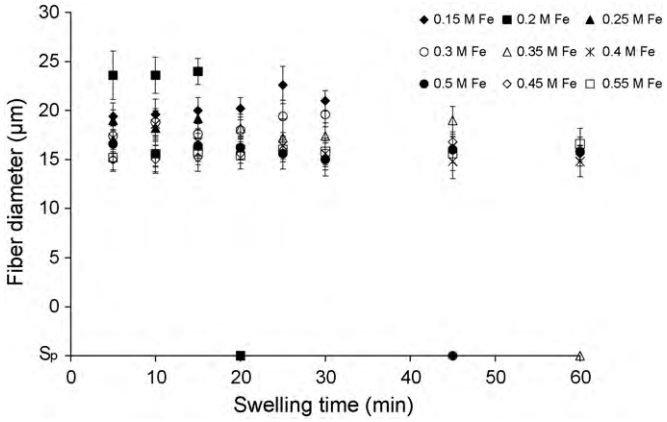

Fig. 4. Swelling rate of viscose fiber in FeTNa solutions containing varying Fe concentration in the presence of 0.8 M free NaOH (Sp = splitting).

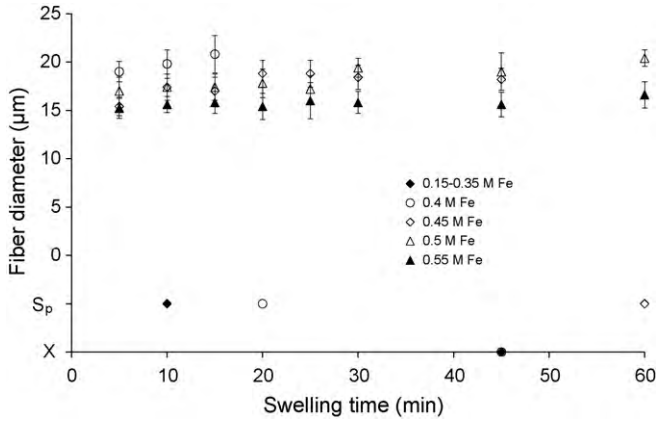

Fig. 5. Swelling rate of viscose fiber in FeTNa solutions containing varying Fe concentration in the presence of 1.25 M free NaOH (Sp = splitting, X = dissolution).

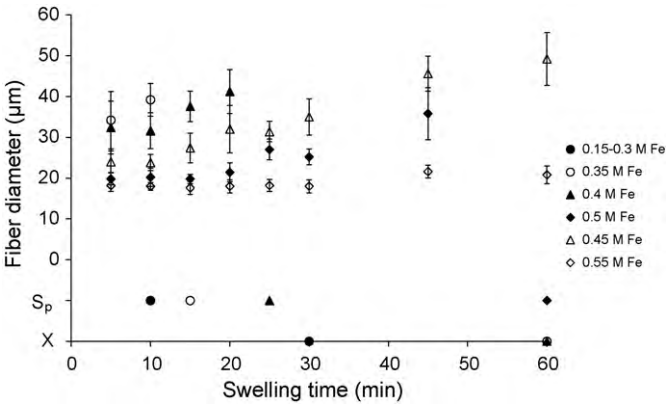

Fig. 6. Swelling rate of viscose fiber in FeTNa solutions containing varying Fe concentration in the presence of 2.5 M free NaOH (Sp = splitting, X = dissolution).

concentration is 1.25 M in FeTNa solutions. These findings are in agreement with literature which mentioned that the increase in Fe concentration of FeTNa solution in the presence of high concentrations of free NaOH (5 M) caused a decrease in conductivity, i.e. interaction between ions and cellulose decreases because of association of ions (Hamann & Vielstich, 1975; Vu-Manh et al., 2010).

Figs. 6 and 7 show the swelling rates of viscose fiber in FeTNa solutions containing varying Fe concentration in the presence of 2.5 and 5 M free NaOH, respectively. Swelling rate of viscose fibers reached equilibrium after 5 min of swelling in FeTNa solutions. Swelling proceeded into splitting and then into dissolution with

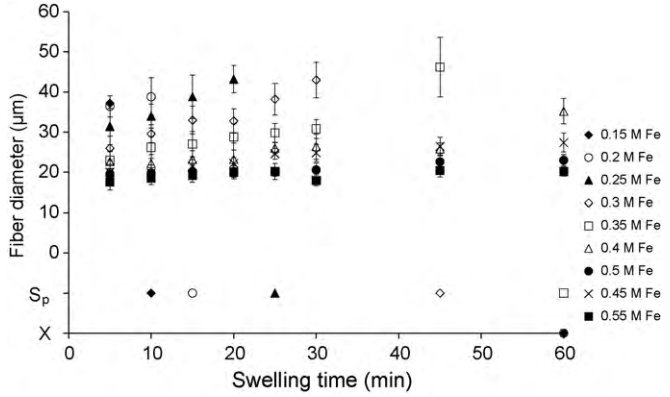

Fig. 7. Swelling rate of viscose fiber in FeTNa solutions containing varying Fe concentration in the presence of 5 M free NaOH (Sp = splitting, X = dissolution).

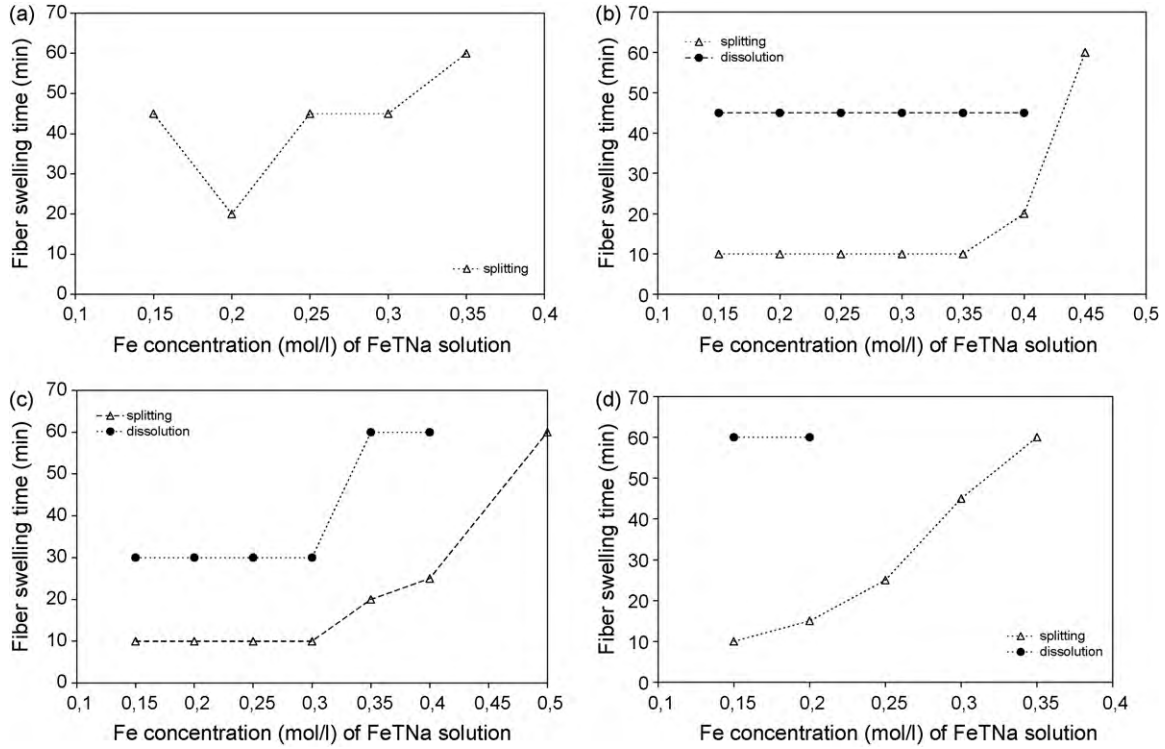

Fig. 8. Changes in the morphology (splitting, dissolution) of viscose fibers in relation to fiber swelling time (min) and Fe concentration (mol/l) of FeTNa solution while free NaOH concentration of FeTNa solution was (a) 0.8 M, (b) 1.25 M, (c) 2.5 M and (d) 5 M.

the increase in Fe concentration in FeTNa solution together with swelling time of fiber.

Fig. 8 shows the fiber swelling time to split or dissolve viscose fibers depending on Fe concentration of FeTNa solution in the presence of 0.8 M (Fig. 4), 1.25 M (Fig. 5), 2.5 M (Fig. 6) and 5 M (Fig. 7) free NaOH. As the Fe concentration increases in FeTNa solution, the time to split or dissolve viscose fiber also increased. An explanation can be the higher interaction between ions of the complex occurring owing to increase in the concentration of ions in the complex. This results in a less interaction between complex and cellulose (Vu-Manh et al., 2010). Another explanation can be the skin of the viscose fiber preventing FeTNa solutions entering inside the fiber when solutions contain high concentrations of metal complexes.

3.4. Morphological changes of viscose fibers depending on Fe and free NaOH concentration for different periods of swelling

Fig. 9 shows swelling-dissolution diagram of viscose fibers depending on swelling time in FeTNa solutions, while FeTNa solutions had varying Fe and free NaOH concentration. FeTNa solutions having either 0.4 or 0.8 M free NaOH caused limited swelling of viscose fibers. FeTNa solutions having free NaOH concentration above 1.25 M in the presence of Fe concentration lower than 0.35 M caused splitting or swelling of viscose fibers depending on swelling time. When Fe concentration in FeTNa solutions was more than 0.35 M in the presence of free NaOH concentration above 1.25 M, either limited swelling or higher degree of swelling of viscose fibers occurred. This shows that free NaOH concentration above 1.25 M in the presence of Fe concentration lower than 0.35 M in FeTNa solutions is of importance to get high degree of swelling and even splitting of viscose fibers.

Fig. 9a and b shows that the borderline between different morphology of viscose fibers *shifted* to higher Fe concentrations of FeTNa solution when swelling time increased from 10 to 20 min. For example, *splitting* region *grew* both into the right (to higher Fe concentrations) and down (to higher free NaOH concentrations) side, while *swelling* region *shifted* to right side. Fig. 9b–d shows that swelling of viscose fibers from 20 min to 30 min and then to 60 min *shifted* the borderline for *splitting* to higher Fe concentrations of FeTNa solution. Dissolution occurred following splitting of fibers.

Swelling of cellulosic fibers in water causes the filling of the pores and amorphous regions between the oriented crystalline regions with water, i.e. water accesses non-crystalline regions (Lenz, Schurz, & Wrentschur, 1982). FeTNa solutions containing 0.8–1.25 M free NaOH (Figs. 4 and 5) swelled viscose fiber limitedly (comparable to the swelling in water, i.e. ca. 20 μm fiber diameter), which shows the accessibility of amorphous regions and pores of the fiber. FeTNa solutions containing 2.5–5 M free NaOH (Figs. 6 and 7) swelled viscose fiber to ca. 30–40 μm fiber diameter. Following either limited swelling (FeTNa solutions containing 0.8–1.25 M free NaOH) or swelling (FeTNa solutions containing 2.5–5 M free NaOH), splitting and later dissolution were observed depending on Fe and free NaOH concentration of FeTNa solution. Fig. 1 shows the follow-up morphological changes of the fiber [swelling (S_{1-2}), splitting (Sp_{1-3}), begin of the dissolution on splitted fibrils (Sp_4)]. Because of the *initial* FeTNa–cellulose interaction in the amorphous regions between the oriented crystalline regions, viscose fiber splits into crystalline fibrillar domains (fibrils) (Sp_1) and the distance between the splitted fibrils increases with time (Sp_{2-3}). When FeTNa interacts with the splitted fibrils, dissolution starts (Sp_4). As a comparison to viscose fibers, the morphological changes of *lyocell* fibers are swelling, dramatic swelling, disintegration and dissolution (Vu-Manh et al., 2010). This shows that differences in morphological architecture of lyocell and viscose fibers cause *dramatic swelling*, *disintegration* for lyocell fibers which have homogeneous fibrillar structure, while *splitting* for viscose fibers which have skin-core structure.

The effect of molecular weight, supramolecular order (crystalline-amorphous regions) and accessible pore volume (APV) of lyocell and viscose fibers on their fiber morphology in

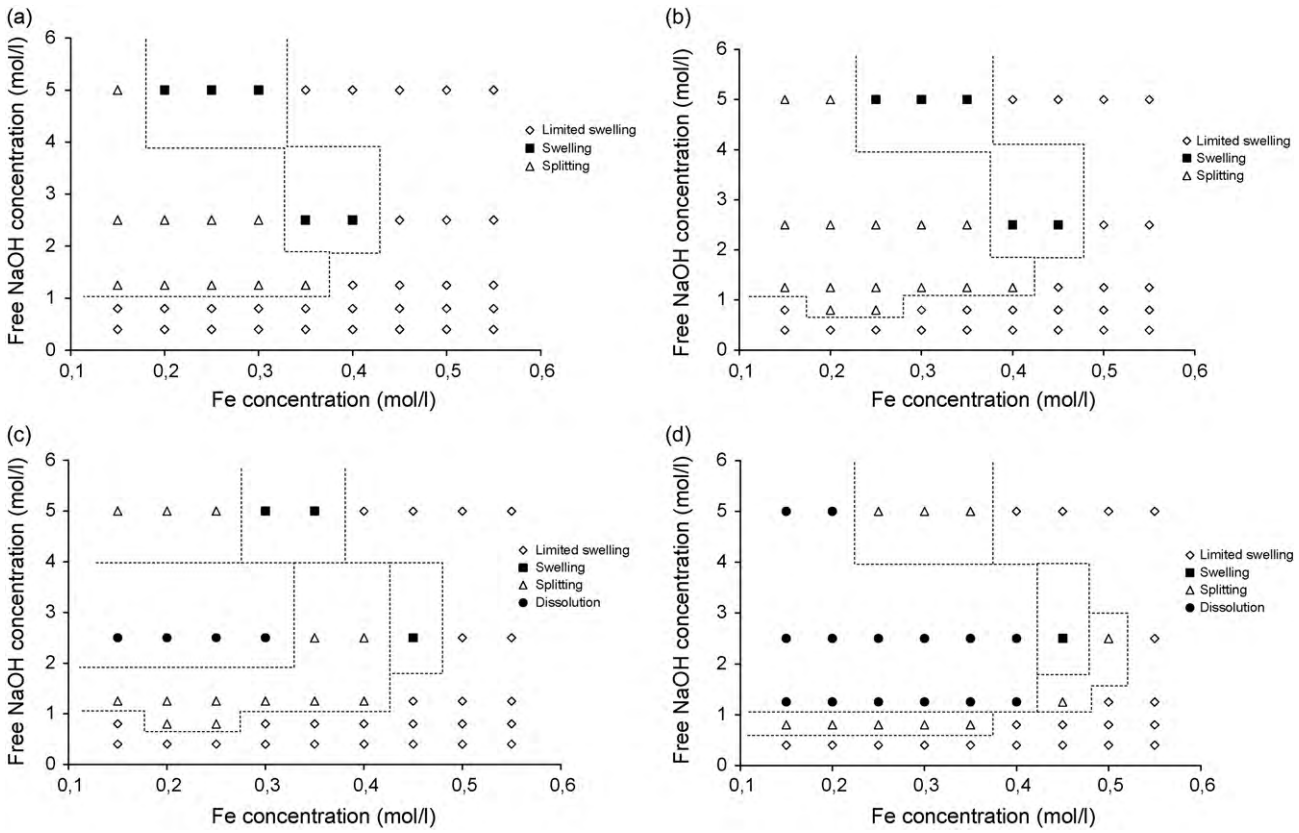

Fig. 9. Swelling-dissolution diagram of viscose fibers swollen for (a) 10 min, (b) 20 min, (c) 30 min and (d) 60 min in FeTNa solutions containing varying Fe and free NaOH concentration.

FeTNa solutions are not visible. Viscose fibers have lower molecular weight, less crystallinity and higher APV compared to lyocell fibers (Öztürk, 2008), but viscose fibers did not loose the fiber structure (dramatic swelling, disintegration) as lyocell fibers did.

Shortly cut viscose fiber swollen in FeTNa solution (1:3:6 molar ratio for Fe(III)hydroxide:tartaric acid:NaOH) showed firstly swelling and later splitting of bundles of fibrils from the skin of the fiber. The swelling rate of *shortly cut* viscose fibers showed that splitting of fibrils starts from the ends of the fibers and moved to the middle of the fiber while forming rolled splitted fibrils from a straight fiber (Theidel, 1962). On the other hand current study shows the splitting of staple viscose fibers with a length ca. 38 mm. In the literature, splitting of viscose fibers with a length of 38 mm did not start from the ends of the fibers but splitting occurred due to swelling stress in alkali solutions (NaOH, LiOH, KOH) where applied shear force on the fiber (Öztürk & Bechtold, 2008).

A mechanically damaged viscose fiber swollen in FeTNa solution (1:3:6 molar ratio for Fe(III)hydroxide:tartaric acid:NaOH) showed that fibrillation (separation of fibrils from the surface of the fiber) started from the damaged point together with a formation of an arch in the same point due to the high inner swelling stress (Theidel, 1962). The skin of the viscose fiber was dissolved after 15 min of swelling in FeTNa solution having a Fe: sodium tartrate: NaOH molar ratio of 1:3:13 while Fe concentration was 0.3 M. 100% stretched viscose fiber splitted into 5 μm of fibrils in 15 min of swelling in FeTNa. FeTNa dissolved the core of the CHA (cyclohexylamine) modified viscose fibers so that hollow fibers were achieved (Baudisch & Philipp, 1965).

At the beginning of the dissolution process, FeTNa attacks the surface of the cotton linters and sulfite pulp intensively. Therefore a swelling layer is formed. Then, the solvent penetrates into the fiber and loosens up the structure very irregularly. At a later stage, formation of fragments and isolation of fibrils occur. (Pionteck, Berger, Morgenstern, & Fengel, 1996).

4. Conclusions

Swelling-dissolution studies of viscose fibers in FeTNa solution show that swelling equilibrium was attained after ca. 5 min of swelling. Depending on Fe and free NaOH concentration of FeTNa solution, treatment in FeTNa solutions caused limited swelling, swelling and splitting of viscose fibers. Above 30 min of treatment time in *selected* FeTNa solutions caused the dissolution of the fibers.

Morphology of viscose fibers in FeTNa solutions were found to differ when free NaOH concentration in FeTNa solution was 0.4, 0.8 and 1.25–5 M. FeTNa solutions having 0.4 M free NaOH led to limited swelling, whereas 0.8 M free NaOH containing FeTNa solutions resulted in swelling and splitting. Free NaOH concentrations at 1.25–5 M caused follow-up morphological changes which are swelling, splitting and dissolution when Fe concentration increased together with the swelling time.

Splitting of viscose fibers occurred when Fe concentration of FeTNa solution was

- 0.15–0.20 M in the presence of 1.25–2.5 M free NaOH and
- 0.25 M in the presence of 1.25 M free NaOH after ca. 2 min of swelling,
- 0.20–0.25, 0.15 and 0.35 M, and 0.35 M in the presence of 0.8 M free NaOH after ca. 20, 45 and 60 min of swelling, respectively,
- 0.15–0.35, 0.4 and 0.45 M in the presence of 1.25 M free NaOH after ca. 10, 20 and 60 min of swelling, respectively,
- 0.15–0.30, 0.35, 0.40 and 0.5 M in the presence of 2.5 M free NaOH after ca. 10, 15, 25 and 60 min of swelling, respectively,

- 0.15, 0.20, 0.25, 0.30 and 0.35 M in the presence of 5 M free NaOH after ca. 10, 15, 25, 45 and 60 min of swelling, respectively,

Dissolution of viscose fibers occurred when Fe concentration of FeTNa solution was

- 0.15–0.40 M in the presence of 1.25 M free NaOH after 45 min of swelling,
- 0.15–0.30 M in the presence of 2.5 M free NaOH after 30 min of swelling,
- 0.35–0.40 M in the presence of 2.5 M free NaOH after 60 min of swelling,
- 0.15–0.20 M in the presence of 5 M free NaOH after 60 min of swelling.

Swelling is an indicator for the interaction between the fiber and complex ions. Limited swelling is due to the less interaction between the complex ions and cellulose. Splitting of viscose fibers into its fibrils was achieved in FeTNa solutions by *only* swelling the fiber.

Current study suggests the possible usage of FeTNa solutions as a both solvent and treatment solution for viscose fibers. In order to get various fiber morphology types such as swelling, splitting, dissolution, etc. Fe and free NaOH concentration in FeTNa solutions, and the fiber swelling time should be varied.

Acknowledgements

The Authors gratefully acknowledge to Lenzing AG in Austria for material support, to Versuchsanstalt and HTL Dornbirn for equipment, to ÖAD (Austrian Exchange Service) for the scholarship grant to MSc. Hai Vu-Manh, to Vorarlberger Landesregierung for EFRE (Europäischer Fonds zur Regionalen Entwicklung) grant to Dr.rer.nat Hale Bahar Öztürk.

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