RECENT RESEARCH ACTIVITIES

Nanofibrillation of dried pulp in NaOH solutions and their regenerations

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Plant cell walls have a strong framework or scaffold consisting of nano-scale crystalline bundles of cellulose molecules, called microfibrils. Because of the stable structure provided mainly by inter/intramolecular hydrogen bonds, the crystal region displays strong mechanical properties longitudinally and low thermal expansion coefficient. In addition to these properties, because of their low weight and morphological features such as large specific surface areas and high aspect ratios, plant-based cellulose nanofibers have great potential for reinforcing polymer matrices.

A variety of fibrillation methods using mechanical treatment have been reported for the preparation of cellulose nanofibers, and currently nanofibers can be isolated from various plant sources. We have also reported an isolation method in which plant fibers are ground in an undried state, and have prepared cellulose nanofibers from various sources such as softwood, rice straw, potato tuber and bamboo[1-3].

However, when dried pups are used as a raw material, the drying process production generates strong hydrogen bonding between cellulose microfibrils after the removal of the matrix, which seems to make it difficult to obtain thin and uniform cellulose nanofibers. On the other hand, it is well known that alkali solutions swell cellulose fibers. It means that alkali-swelling may loose the hydrogen bond between microfibrils and facilitate the nanofibrillation. In the present study, we tried the fibrillation of pulps under alkaline condition in order to obtain fine nanofibers from dried pulps. The fibrillation of dried pulps was executed in 8% or 16% of NaOH using a bead-milling method.

As a result, the bead milling method in 8 wt% NaOH succeed to fibrillate the dried pulp into uniform nanofibers with a width of approximately 12 nm (Fig. 1a), which corresponds to the cellulose microfibril aggregates in wood cell wall. On the other hand, when fibrillated in 16 wt% NaOH, the sample neutralized showed a continuous and uniform network with a width of 12-30 nm (Fig. 1b). Moreover, it was found that both of nanofiber suspensions treated in 8 wt% or 16 wt% NaOH were formed into a stable hydrogel by neutralization. Such gelation behavior is most likely caused by the interdigitation of the neighboring nanofibers during NaOH treatment (mercerization)[4, 5]. This method can be applied to prepare wet-spun fibers based on cellulose nanofiber.

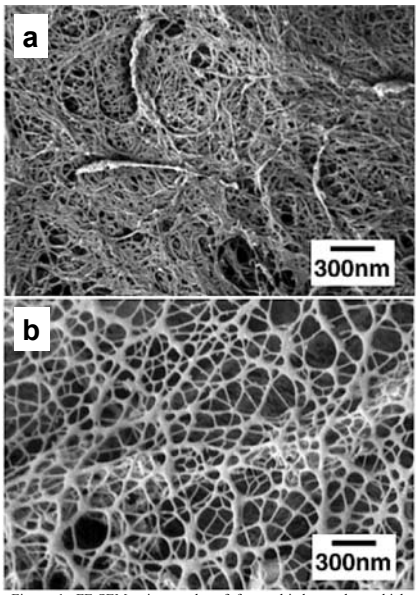

Figure 1. FE-SEM micrographs of freeze-dried samples, which were fibrillated using a beads-milling in 8 wt% (a) and NaOH (a–c) and 16 wt% NaOH (b) and then neutralized.

References

[1] Abe K, Iwamoto S, Yano H. *Biomacromolecules*. **8**: 3276-3278, 2007.

[2] Abe K, Yano H. *Cellulose* **16**: 1017-1023, 2009.

[3] Abe K, Yano H. *Cellulose*. **17**: 271-277, 2010.

[4] Abe K, Yano H. *Carbohydrate Polymers*. **85**: 733–737, 2011.

[5] Abe K, Yano H. *Cellulose*. **19**: 1907–1912, 2012.