RECENT RESEARCH ACTIVITIES

Preparation of a high concentration cellulose nanofiber

(Laboratory of Active Bio-based Materials, RISH, Kyoto University)

Kentaro Abe and Hiroyuki Yano

The drying process in typical pulp production generates strong hydrogen bonding between cellulose microfibrils in refined cell walls and increases the difficulty in obtaining uniform cellulose nanofibers. In this study, we applied a ball-milling method in NaOH solutions to prepare a high-concentration cellulose nanofibers from dried pulp. NaOH treatments loosened the hydrogen bonding between cellulose microfibrils in dried pulps [1].

A dried bleached kraft pulp from softwood with an α-cellulose content of 90% was used. 8 wt% pulps immersed in 8% (w/w) NaOH aqueous solutions at room temperature. Nanofibrillation of pulps was performed using a ball mill (TSG-6U, IMEX Co. Ltd., Sooka, Japan) at 2000 rpm for 90 min with 5-mm zirconia balls at a filling rate of 33% (v/v). The samples were separated from the beads by filtration.

After fibrillation by ball milling in 8% NaOH solution for 90 min, smooth suspensions were obtained for all samples (Figure 1). FE-SEM observation revealed that fine nanofibers with a uniform diameter of approximately 12–20 nm were formed (Figure 2). These nanofibers are comparable to those prepared from never-dried refined wood samples using a grinder [2]. This result suggests that 8% NaOH treatment disrupted the hydrogen bonds between microfibrils in dried pulps and improved their nanofibrillation.

Interestingly, the nanofiber suspensions prepared in 8% NaOH were formed into hydrogels by neutralization because of surface entanglement and/or interdigitation between the nanofibers [3, 4]. Therefore, our method did not allow preparation of individual cellulose nanofibers in water from dried pulps. However, we are currently applying this method to the direct preparation of high-strength cast films and spun fibers. For general preparation of cellulose-based films and spun fibers, cellulosic raw materials must be dissolved using specific chemical agents such as carbon disulfide for rayon and cellophane. In contrast, our method can introduce formability to dried pulps without any dissolution process. In particular, nanofibrillation in 8 % NaOH solution can produce free-form cellulosic products while maintaining the original cellulose I crystal form, which has higher elastic modulus than cellulose II (cellulose I: 138 GPa, cellulose II: 88 GPa). Consequently, high-strength cellulosic products such as films and fibers can be produced with relative safety using only NaOH and coagulants.

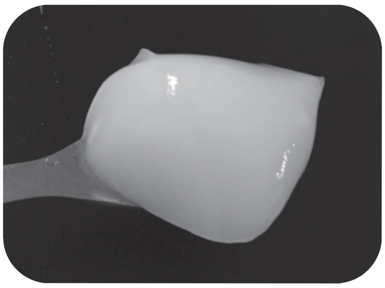

Figure 1. Appearance of 8 % cellulose nanofiber suspension in 8 % NaOH solution.

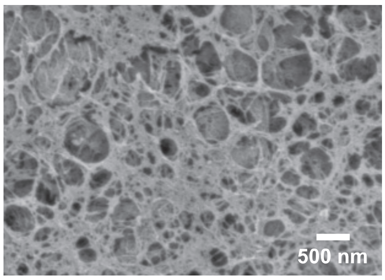

Figure 2.FE-SEM micrograph of freeze-dried cellulose nanofiber.

References

[1] Abe K, *Cellulose*, **23**, 1257-1261, 2016.

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

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

[4] Abe K, Yano H. *Cellulose*. **19**: 1907-1912, 2012.