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Application News

No. Q121

Powder Property Analysis

Characterization of Fiber Length and Dispersibility of Cellulose Nanofibers

Cellulose is a polysaccharide that is the main component of plant cell walls. The cellulose fibers called nanocellulose are produced by defibrating cellulose fibers. Nanocellulose fibers having a width of 4 nm to 100 nm, length of several μm or longer, and a high aspect ratio (100 or more) are generally called cellulose nanofibers (CNF). In addition to CNF produced from plant fibers, CNF also includes nano-fibrillated bacterial cellulose which is produced by cellulose synthesis bacteria (NFBC; also called fermented nanocellulose).

CNF is a high-performance material with a variety of desirable properties, including light weight, high strength, low thermal expansion, a high gas barrier, absorption, thickening performance, and transparency. Moreover, as a plant-derived material, CNF is also a sustainable, low environmental load resource. Future application in various fields is expected, beginning with automotive components and electronic materials.

However, the lack of adequate techniques for evaluating the physical properties of CNF is an issue. The physical properties of CNF are known to be related to the fiber length and diameter, which are currently measured mainly with a microscope. Although accurate microscopic measurement is possible because the fibers are measured individually, this is a time-consuming process. Furthermore, microscopic measurements are generally carried out after drying the specimen material, and the measurement results may be different from those in the water-dispersed state. Therefore, a quick, simple method for characterization of the fiber length and dispersibility of CNF in the water-dispersed state has been desired.

This article introduces an example of characterization of the fiber length and dispersibility of various CNF by using Shimadzu's high sensitivity model SALD™-7500nano nano particle size analyzer (special specification*, Fig. 1).

T. Sumoto

*Please inquire concerning details.

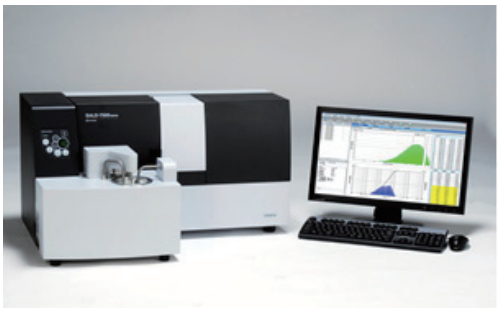

Fig. 1 High Sensitivity Model SALD™-7500nano Nano Particle Size Analyzer

Samples and Measurement Conditions

The measured samples were three types[*1] of a commercially-available water dispersion-type pulp-derived CNF with different fiber lengths (ultra-long, standard, ultra-short), carboxymethylcellulose (CMC),[*1] and nano-fibrillated bacterial cellulose (NFBC).[*2]

The device used in the measurements was a high sensitivity model SALD-7500nano nano particle size analyzer utilizing the laser diffraction method. Fig. 2 shows the device configuration.

Due to the extremely small fiber diameter and high transparency of the samples, it is difficult to obtain scattered light from the CNF. In order to adequate scattered light, it is necessary to increase the sample concentration in comparison with ordinary samples, but when measuring fibrous samples, the fibers tend to intertwine and aggregate under high concentration conditions. For this reason, the high sensitivity model SALD-7500nano was used in these measurements.

In the measurements, the mother liquors were prepared by diluting the samples with pure water, using an MS75 sampler, and the measurements were then carried out under the conditions in Table 1. To confirm the condition of CNF dispersion, the condition with only circulation and the condition with ultrasonic irradiation (sonication) in the circulating state were compared, and changes in the dispersion condition were also checked.

*1 Sugino Machine Limited, BiNFi-s™ water dispersion-type cellulose Product No.: IMa-10002 (ultra-long), WMa-10002 (standard), FMa-10002 (ultra-short), TMa-10002 (CMC)

*2 Kusano Sakko Inc., nano-fibrillated bacterial cellulose (NFBC; Fibnano)

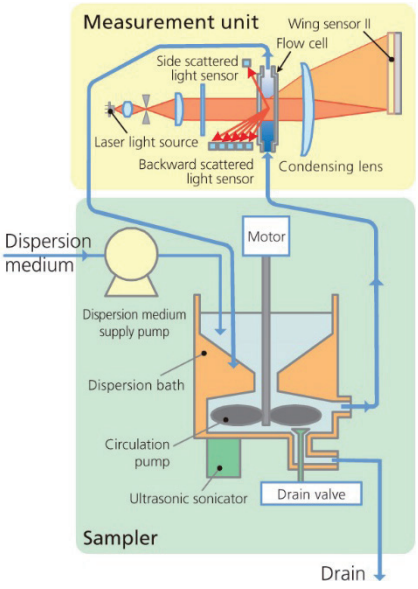

Fig. 2 Configuration of Device

Table 1 Measurement Conditions

Measurement unit	: SALD-MS75 Sampler
Dispersion medium	: Pure water
Dispersant	: None
Dispersion methods	: (1) Circulation only (2) Circulation with ultrasonic irradiation by internal sonicator
Average count	: 512
Water level	: Middle stage
Refractive index	: 1.60-1.00 i

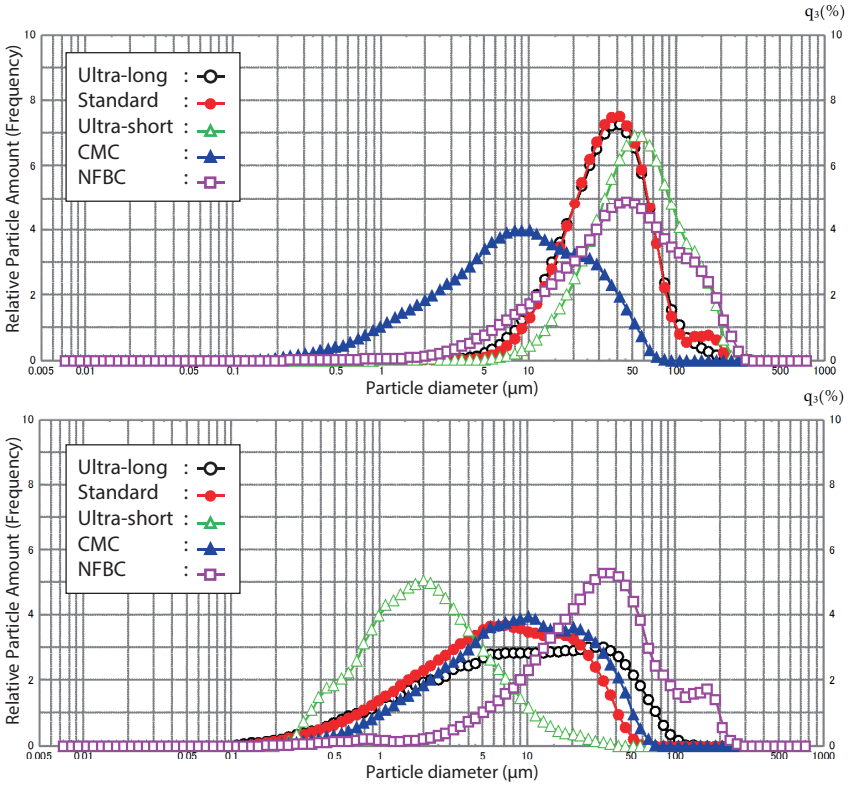

Fig. 3 Results of Particle Size Distribution Measurements
Top: Before Sonication Bottom: After Sonication

Measurement Results and Discussion

Fig. 3 shows the measurement results. The upper part of Fig. 3 shows the measurement results when the sample was simply circulated by the pump without ultrasonic irradiation by the internal sonicator of the sampler.

In the pulp-derived cellulose, the results for the samples with the different fiber lengths, i.e., ultra-long, standard, and ultra-short, showed approximately the same particle diameters for the ultra-long and standard fibers, while the diameter of the ultra-short fibers was large in comparison with the extra-long and standard fibers.

Therefore, dispersion treatment was performed by sonication with the internal sonicator of the sampler. The graph in the lower part of Fig. 3 shows the results after dispersion treatment was performed until changes in the particle diameter were eliminated by sonication.

With the pulp-derived sample, in comparison with the results before sonication, it was found that the dispersion state changed and a correlation between the fiber length and particle diameter could be obtained. This shows that accurate characterization of the fiber length is possible by performing sonication to obtain a dispersed state. The particle size of the NFBC also changed as a result of sonication, but the amount of that change was minimal. Similarly, no clear change in the particle size could be observed in the CMC before and after sonication. This is attributed to the fact that the surface of CMC molecules became negatively charged due to the carboxymethyl group introduced by chemical treatment, and this resulted in a molecular dispersion state (complete dissolution) in water.

Conclusion

Although it is possible to obtain highly accurate measurement results with current microscopic measurement techniques, measurements of the total distribution of samples require excessive time, and when samples are dried as a pretreatment process, change in the dispersion state is an issue. On the other hand, measurement in the water-dispersed state is possible by using the high sensitivity model SALD-7500nano.

From the results described here, it was found that the high sensitivity model SALD-7500nano enables simple characterization of the fiber length and dispersibility of CNF when dispersed in a liquid.

In the future, use in diverse situations, including research and development, inspections, and quality control is expected by applying the SALD-7500nano in combination with direct microscopic measurement.

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