Particle Size Analysis of TEMPO-Oxidized Cellulose Nanocrystal

Outline

Nanocellulose is a fibrous material derived from wood and is used as composite materials, etc. It is attracting attention as a cutting-edge material. Cellulose nanocrystals (CNC) are needle-like materials with a width of about 5-20 nm and a length of about 100-250 nm defibrated using chemical treatment. CNC is cheaper and more attractive than other high-performance nanomaterials as an alternative to some other petrochemical products. It is expected to be useable for oil spill cleaning, engineering, pharmaceuticals, container substitutes, biodegradable food packaging, etc. It is important to understand the fiber size and dispersion state. It is necessary to have an analysis method that can quickly evaluate a wide ranges of particle size distribution for separated particles to agglomerates.
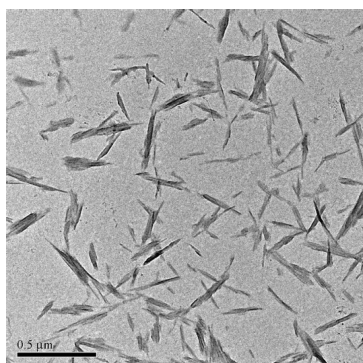

Fig. 1 TEM image of CNC
Source: TEM image is used with permission from Cellulose Lab Inc.

Method

Apparatus: HORIBA Partica CENTRIFUGE
Measurement mode: Homogeneous
Samples: CNC dispersion (Cellulose Lab, CNC-TEMPO, width: 5-20 nm, length: 140-200 nm)
Particle: TEMPO (anionic type) CNC (Solid conc. 0.5-2%, RI: 1.840, Density: 1,500 kg/m³)
Medium: De-ionized water (Refractive index: 1.333, Viscosity: 0.797 mPa·s, Density: 996 kg/m³)
Particle size distribution (PSD) base: Volume based
Calculation setting: Custom mode
Extinction coefficient correction: ON
Smoothing: 4

Results

The purple line in Fig. 2 shows the volume-based PSD measured by Partica CENRIFUGE, calculated without extinction coefficient correction. It has a median diameter of 1.57 µm and a mean diameter of 2.21 µm, with 2 peaks of agglomerated particles. The blue line shows another volume-based PSD calculated with extinction coefficient correction. It has a median diameter of 19.1 nm and a mean diameter is 19.8 nm, resulted from separate, dispersed particles.
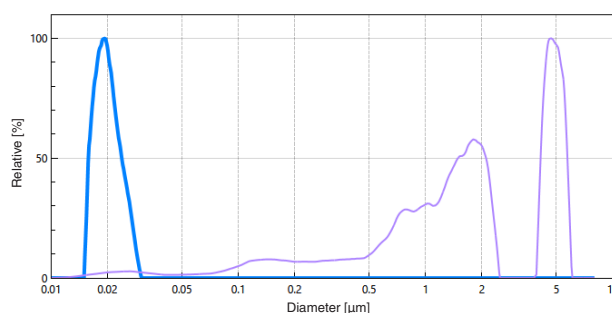

Fig. 2 Volume-based PSD

Conclusion

We believe that the CNC dispersion state is affected by fiber width, not fiber length. In addition, since the measurement result of un-agglomerated CNC is less than the lower detection limit specified by ISO, care must be taken when handling the particle size result. In other words, it is believed that such small particles cannot be calculated using the classical Stokes equation because they are significantly affected by Brown diffusion. However, based on this measurement result, it is possible to measure dispersed ones together with the liquid dispersant at the same time. It is possible to improve quantitation by extinction coefficient correction. Thus, we think that this method can be used for evaluation of the dispersion state of CNC.