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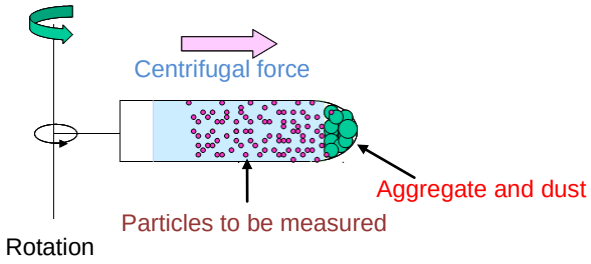
Pretreatment by ultracentrifugation and particle size distribution measurement

CP-WX series preparative ultracentrifuge and P40ST swinging bucket rotor

Given the considerable development of the nanotechnological industry in recent years, particle sizes of the nanometer order are now being controlled in materials used in various fields. Pretreatment is important in measuring the particle size distribution of nanometer-order samples with high reproducibility and accuracy. Among the methods of such pretreatment are separation and refinery that entail eliminating the effects of contamination and aggregate by using an ultracentrifuge, and the concentration of samples that are low in concentration or scattering light intensity.

This paper introduces a method of separating and removing the contamination and aggregate from samples, and examining the target particles (to be measured) by using a laser diffraction/scattering-type particle characterization device.



Description

1. Samples
   - ① 200-nm PSL (NIST Standard 3200A, density: 1.050 g/cm^3, manufactured by Thermo Fisher Scientific)
   - ② 700-nm PSL (NIST Standard 3700A, density: 1.050 g/cm^3, manufactured by Thermo Fisher Scientific)
   - ① and ② mixed together at a ratio of 10 to 1
2. Centrifugal and measuring conditions

   Centrifuge: CP100WX ultracentrifuge
   Rotor: P40ST swinging bucket rotor (6 tubes)
   Centrifuging tube: 13PA tube
   Rotating speed: 15,000 rpm
   Maximum RCF: 40,000 Xg
   Centrifugal time: 5 minutes
   Particle characterization device: LA-950V2 laser diffraction (manufactured by Horiba, Ltd.)
   Measuring temperature: 25°C
   Particle size distribution standard: Volume
   Sample refraction index: 1.59-0.00i
   Dispersion medium refraction index: 1.33-0.00i
   Measuring technique: Batch-type cell (for very small trace quantities: approx. 10 ml)

3. Results

A mixture of 200-nm PSL and 700-nm PSL was prepared at a ratio of 10 to 1, and then measured for its particle size distribution, thereby revealing a distribution near 200 nm and another near 700 nm (Figure 1).

These samples were centrifugally treated for five minutes, followed by their supernatant and precipitate being measured for grain size distribution. Figure 2 (supernatant) and Figure 3 (precipitate) show the results obtained. Figure 2 indicates that the 700-nm PSL has been removed from the supernatant.

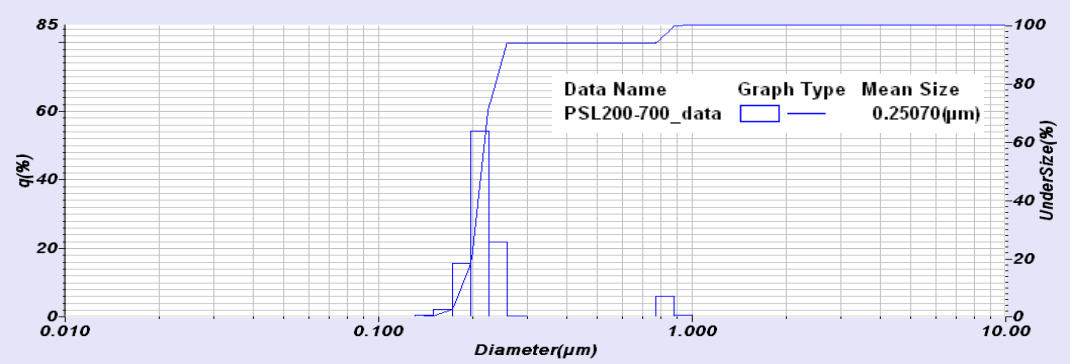


Fig. 1 Particle size distribution measurements of 200-nm and 700-nm mixture solutions (at a ratio of 10 to 1)

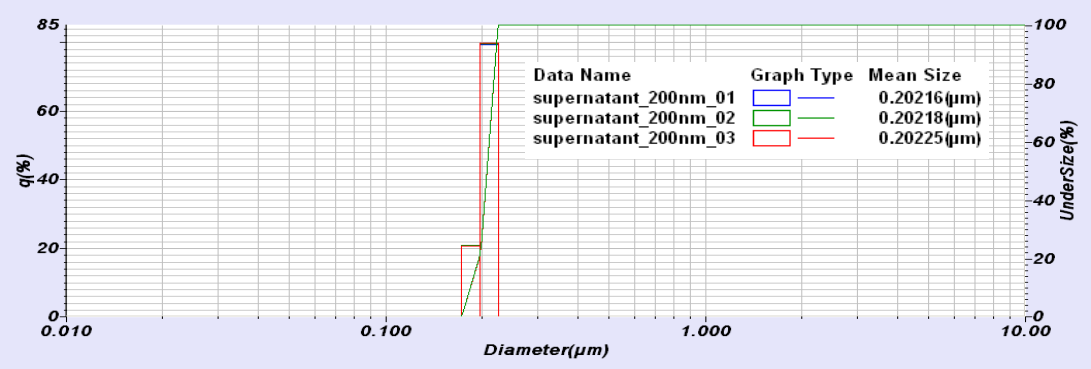


Fig. 2 Particle size distribution measurements of supernatant after centrifugal treatment

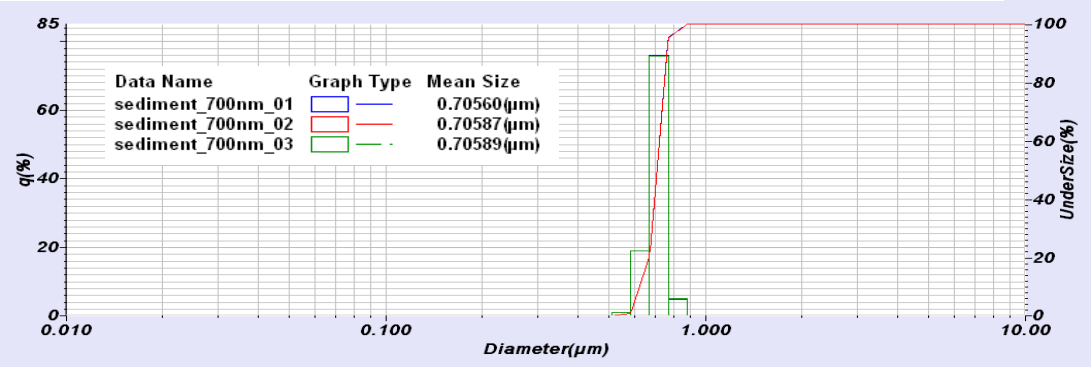


Fig. 3 Particle size distribution measurements of precipitate after centrifugal treatment

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