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Ultracentrifugal sample classification and particle diameter measurement of two-component mixed standard particles

CS-GXII series micro ultracentrifuge and S52ST swing rotor

In line with the rapid development of the modern nanotechnology industry, nano-order particle diameters are now being controlled in materials in various fields. Particle diameter control is a critical factor in the pretreatment process of separation and refinement, in order to achieve a targeted particle preparation and eliminate the effects of contamination.
This paper describes the use of the isopycnic centrifugation, one of the centrifugal separation methods, to separate mixed particles based on density differences and confirm the particle diameter of each band of particles by means of a particle size analyzer.

Description

1. Separated samples
   ① 29nm latex particles (made of polystyrene, density: 1.115 g/cm^3, selling by JSR Corporation) 10,000 ppm
   ② 100nm latex particles (made of polystyrene, density: 1.050 g/cm^3, selling by Thermo Fisher Scientific Inc.) 200 ppm

2. Centrifugation and measurement conditions

Centrifuge:	CS150GXII micro ultracentrifuge
Rotor:	S52ST swing rotor (four tubes)
Tube:	5PET tube
Speed:	52,000 rpm
Maximum RCF:	276,000 x g
Centrifugation time:	20 hours
Density-gradient fluid:	10-30 wt% sucrose solution 4.5 mL
Particle size analyzer:	nano Partica SZ-100 (manufactured by HORIBA, Ltd.)
Measuring temperature:	25°C
Particle diameter distribution standard:	Scattered light intensity standard

3. Results
   Figure 1 shows a picture of a sample under test before centrifugal separation. Figure 2 shows the plotting of measurement results regarding the particle diameter distribution of the mixed sample before separation. Figure 2 shows that particle diameter distributions of 29 nm and 150 nm were obtained.

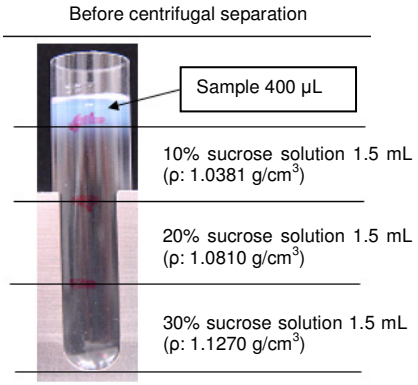

Fig. 1 Sample before centrifugal separation

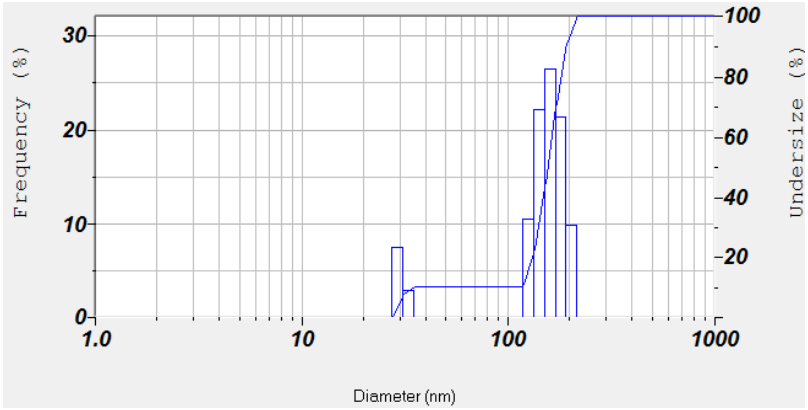

Fig. 2 Measurement results of particle diameter distribution before centrifugal separation (provided by HORIBA, Ltd.)

Figure 3 shows a picture of mixed particles after centrifugal separation. The centrifugal separation process resulted in the formation of two bands, each of which was collected to measure its particle diameter on a particle diameter measuring instrument manufactured by HORIBA, Ltd. The band closer to the bottom was found to have an arithmetic mean diameter of 29 nm, as compared with 150 nm for the band closer to the top (see Fig. 4). This suggests that particle separation has been conducted based on differences in density instead of particle size, thereby confirming the possibility of centrifugal separation by particle diameter using the isopycnic centrifugation.

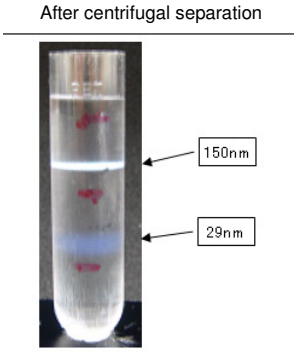

Fig. 3 Sample after centrifugal separation

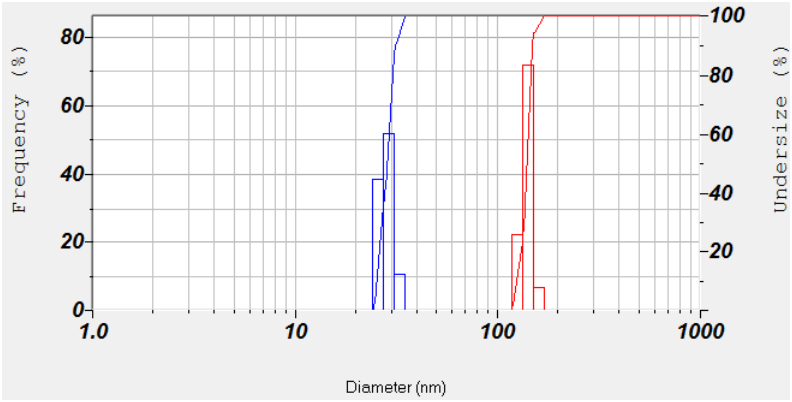

Fig. 4 Measurement results of particle diameter distribution after centrifugal separation (provided by HORIBA, Ltd.)

4. Discussion

The measurement of particle diameter distribution confirms that a higher-density particle 29 nm in diameter (density: 1.115 g/cm^3) appeared as a bottom band as compared with a lower-density particle 150 nm in diameter (density: 1.050 g/cm^3) appearing as a top band after centrifugal separation using the isopycnic centrifugation.

The separation of mixed nanoparticles using the isopycnic centorifugation, one of the centrifugal separation methods, enables the fractionation of nano-size particles. Moreover, the measurement of fractionated particle diameter also confirms that the targeted particles have been achieved for added reliability.

The present method offers a useful tool for refining carbon nanotubes or fractionating fine pigment particles contained in an ink or toner formulation.