



Acceleration of dispersing calcium carbonate particle in aqueous media using jet milling method



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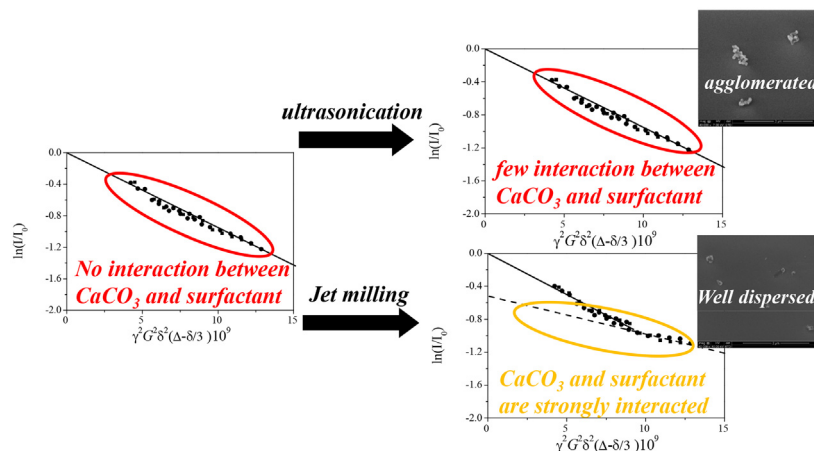
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HIGHLIGHTS

- Various characterizations make it clear the difference between jet milling and ultrasonication to disperse CaCO₃ particles.
- We found jet milling method is effective suspending method for CaCO₃ particles in aqueous suspension.
- PFG-NMR clarified jet milling accelerates interaction/adsorption between CaCO₃ particles and surfactant molecules.
- DLS and AF4 methods show the obvious difference of the size distribution depending on the dispersing methods.
- The difference of the surface potential caused by surfactant is also important for high dispersibility of CaCO₃ particles.

GRAPHICAL ABSTRACT

Jet milling accelerates interaction/adsorption between CaCO₃ particles and surfactant molecules compared to ultrasonication method.



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ABSTRACT

Suspending particles in a liquid phase is a key to producing various functional nano products. We prepared calcium carbonate (CaCO₃) particles in aqueous suspension using two methods, ultrasonication and jet milling, and characterized them using various techniques. Suspensions of CaCO₃ particles in aqueous media were prepared with different industrial surfactants: polyethylene glycol *p*-(1,1,3,3-tetramethylbutyl)-phenyl ether (Triton X-100), polyoxyethylene(23) lauryl ether (Brij35), polyoxyethylene alkylether (Softanol70), and sodium dodecyl sulfate (SDS). According to dynamic light scattering and asymmetric flow field-flow fractionation assessments, the dispersed CaCO₃ particles are clearly small when Triton X-100 or Softanol 70 is used as the surfactant regardless of whether ultrasonication or jet milling was used as the dispersion method, although the size distribution of the particles varies depending on the dispersion method. The observed zeta potentials of the CaCO₃ particles in various surfactant solutions support the finding that Triton X-100 and Softanol70 are good dispersants for CaCO₃ particles in aqueous media because of their larger electrostatic repulsion interaction. Pulsed field gradient nuclear magnetic resonance (PFG-NMR) indicated that jet milling accelerates interaction/adsorption between CaCO₃ particles and surfactant molecules, so it disperses the aggregated/agglomerated CaCO₃

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particles more effectively than ultrasonication. In addition, interestingly, we found that Softanol70 worked well as a dispersant for CaCO₃ particles in aqueous media for both ultrasonication and jet milling, as indicated by the PFG-NMR assessment.

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1. Introduction

Calcium carbonate (CaCO₃) is widely used in biological and industrial applications such as paint preparation, paper coating, polymer moulding, food processing, and preparation of clay materials owing to its high biocompatibility, ease of production, slow biodegradability, and antacid property [1–17]. In particular, preparation of smaller CaCO₃ nanoparticles in suspension is becoming an important key to producing superior functional materials in various fields because reducing the particle size leads to a large specific surface area and high surface energy [18,19]. Indeed, since such smaller CaCO₃ particles have been prepared and applied as aqueous dispersions, it is significant that CaCO₃ particles are found to be well dispersed as primary particles in the CaCO₃ slurry.

Commercial CaCO₃ particles are commonly supplied as a dry powder and must be suspended in aqueous solution using various surfactants. In the preparation stage, selection of an appropriate surfactant is important. The surfactant molecules usually interact with the CaCO₃ particles; some surfactants may be strongly adsorbed on CaCO₃ particle surfaces, and some may be exchanged between the surfaces and the aqueous solution because of their weak binding interactions with the surfaces of the CaCO₃ particles, resulting in well-suspended particles. According to the Derjaguin, Landau, Verwey, and Overbeek (DLVO) theory [20,21], interaction between CaCO₃ particles in an aqueous surfactant solution is characterized by two types of interactions, electrostatic repulsive interactions and van der Waals attractive interactions. The electrostatic repulsive energy results from the overlap between diffuse double layers of particles, and it decays exponentially with the distance between the particles. Thus, interactions between the particles continue even over long ranges. On the other hand, the van der Waals attractive energy decays according to a power law. Smaller CaCO₃ nanoparticles should be more easily aggregated/agglomerated with each other by the van der Waals attractive energy than larger CaCO₃ particles. Particle aggregation can therefore be prevented by adsorption of a surfactant on the particle surface according to the DLVO theory; i.e., the electrostatic repulsive interaction can be controlled by adding a surfactant [22–28]. Furthermore, individual particles in the aqueous surfactant solution remain suspended by steric interaction of the adsorbed surfactant for a long period in a non-DLVO system [19,29].

The method used to suspend of CaCO₃ particles is also important for preparation of well-dispersed particle suspensions. There are many methods for reducing the size of larger particles, i.e., vortex mixing, use of an ultrasonic bath, use of an ultrasonic homogenizer, and high-pressure homogenization (jet milling). Ultrasonication methods are especially useful for preparing particle suspensions and thus are commonly used. Wet-type jet milling is also used to prepare suspensions of nanomaterials and drug particles [30–32]. In this method, particles suspended in a liquid medium are passed through a narrow channel at high pressure; then, suspension of particles is enhanced by the complex shear force arising from turbulent flow in the channel and cavitation from the abrupt pressure change. Although jet milling is a newer method, the advantage of this dispersion technique is that it yields contamination-free suspensions, unlike other methods. In addition, during preparation, the

particle suspension obtained using jet milling has the same shear force throughout, in contrast to those in ultrasonication methods, where the sonication power depends partly on the position of the ultrasonic transducer.

This study aims to compare the effectiveness of jet milling and ultrasonication for preparing suspensions of CaCO₃ particles within a shorter time using a general industrial surfactant. The dispersibility of the prepared CaCO₃ particle suspensions was investigated through dynamic light scattering (DLS), asymmetric flow field–flow fractionation (AF4), and electrophoretic mobility measurements. The interaction of diffused surfactant molecules on the surface of CaCO₃ particles in aqueous media was directly observed by pulsed field gradient nuclear magnetic resonance (PFG-NMR) measurements. Because the PFG-NMR method can mathematically and physically predict the diffusion behaviour of much smaller targets in solution than the DLS method can, it has been used to study the diffusion behaviour of various surfactant molecules in aqueous suspensions of CaCO₃ particles [29,33–35]; therefore, we employed this method in this study. Additionally, using the DLVO theory and zeta potential measurement, the experimental results were analyzed by simulating the interactions between CaCO₃ particles in various aqueous surfactant solutions. Using these various characterizations, we clarified the effectiveness of jet milling for preparing suspensions of CaCO₃ particles in aqueous media.

2. Experimental section

2.1. Materials

High-purity CaCO₃ particles (Hakuenka-CC-R) were obtained from Shiraishi Kogyo Kaisha, Ltd (Osaka, Japan). The Brunauer–Emmett–Teller specific surface area of CaCO₃ particles was 17.5 m²g⁻¹. We used non-pore type of CaCO₃ particles in this study. Polyethylene glycol *p*-(1,1,3,3,-tetramethylbutyl)-phenyl ether (Triton X-100; MW = 646.85) was purchased from Thermo Fisher Scientific (Geel, Belgium). Polyoxyethylene(23) lauryl ether (Brij35; MW = 1199.54) was purchased from Wako Pure Chemical Industries, Ltd (Osaka, Japan). Sodium dodecyl sulfate (SDS; MW = 288.38) was purchased from Wako Pure Chemical Industries, Ltd (Osaka, Japan). Polyoxyethylene alkylether (Softanol70) was obtained from Nippon Shokubai Co., Ltd (Osaka, Japan). The aqueous surfactant solutions were prepared with ultrapure water obtained by using an ultrapure water preparation system (Organo, Tokyo, Japan). The electrical resistivity of the ultrapure water was 18.2 MΩ cm, and the total organic carbon content of the water was less than 1 ppb.

2.2. Preparation of aqueous suspensions of CaCO₃ particles

Homogeneous aqueous suspensions of CaCO₃ particles (0.1 mg mL⁻¹) were suspended in 0.1 mg mL⁻¹ aqueous solutions of Triton X-100, Brij35, SDS, or Softanol70 using an ultrasonic bath (140 W, 25 kHz) at 15 min intervals for 6 h. Although the dispersing time seemed longer, we checked the change of the size of CaCO₃ particles in all suspension at time by time, then; stopped the dispersing process when we found no-change of the size of CaCO₃ particles by ultrasonication. Suspensions with the

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