

Rheological behaviors of nanosilica suspensions with different dispersion levels prepared by the bead milling technique



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ABSTRACT

The different dispersion levels of silica nanoparticles in organic suspensions were deliberately fabricated using the bead milling technique by controlling the milling time. The dispersion level was confirmed by the dynamic light scattering technique, the transmission electron microscopy, the stability of the suspensions as well as the optical properties of the coatings made from the suspensions. The rheological behaviors of the suspensions were investigated by the steady and dynamic shear tests. It was found that larger agglomerations had a stronger impedance for suspension to flow, leading to a higher viscosity and stiffness. Some important rheological parameters were correlated with the dispersion levels of the suspensions and the reasonable explanation was discussed.

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1. Introduction

In recent years, organic–inorganic nanocomposites have received great interest in both fundamental and applied research owing to their superior properties in comparison with those of the microcomposites [1–3]. The homogeneous distribution of nanoparticles in polymers is a prerequisite for the superior properties of nanocomposites [4,5]. However, most of the nanoparticles, such as the fumed nano-silica, nano-alumina, carbon nanotubes, are known to agglomerate usually in dispersions or polymer matrices because of the strong attraction between nanoparticles [6,7]. The larger nanoparticle agglomerations (often corresponding to the poor dispersion level in suspensions or polymers) can induce stress concentration in polymers and thus leading to premature failure of the nanocomposites, giving rise to negative effects to the mechanical properties of the nanocomposites. For the transparent coatings, the larger agglomerations can interfere with light propagation and finally reduce the optical transmittance and increase haze values of the coatings [4,8].

Also, the addition of nanoparticles has a significant effect on the rheological behaviors as well as the processability of the dispersions or polymers that the nanoparticles fill [9–11]. Nanoparticles usually bring about stronger thickening effects in dispersions or

polymer matrices than the microparticles [12,13]. With the loading increasing, nanoparticle networks (clusters) may form through the Van der Waals force, hydrogen bonding or other molecular interactions [14,15], leading to an elastic rheological response, which is characterized by the fact that the storage modulus G' of the polymer nanocomposites is higher than the loss modulus G'' [16] in the dynamic frequency rheological tests. Surface modification of nanoparticles is a common way to reduce their interactions, enhance the chemical compatibility with polymer matrices and improve dispersion levels of nanoparticles in the polymers [17,18]. After surface modification, the rheological behaviors of the blends are found to undergo a transition from elastic to viscous response [19,20].

Although the effects of nanoparticle size, loading and surface properties on the rheological behaviors of the dispersions or polymers have been thoroughly investigated, the works related to the dispersion level of nanoparticles are still lack to the best of our knowledge. It can be found from many works that the suspensions or polymers having poor dispersion level of nanoparticles show relatively larger viscosities, in comparison with those having good dispersion level of nanoparticles [13,21]. However, the dispersion levels are normally evaluated by transmission electron microscopy (TEM) or scanning electron microscopy (SEM), the results obtained are not quantitative but qualitative in those works, the systematic works reporting the correlation between the rheological behaviors and dispersion level of nanoparticles are still needed.

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In this work, the fumed silica nanoparticles were dispersed in a mixture of trimethylolpropane triacrylate (TMPTA) and butyl acetate (BA) by the bead mill technique. By adjusting the milling time, different dispersion levels of nanoparticles in the suspensions can be obtained and evaluated by the dynamic light scattering technique (DLS) and TEM. The rheological behaviors of the suspensions were analyzed by the steady shear test, dynamic strain amplitude test and dynamic frequency test. Some important rheological parameters were correlated with the dispersion levels; and the possible mechanisms behind the correlation was discussed.

2. Experimental

2.1. Materials

Fumed silica nanoparticles, surface-modified with methyl groups, were supplied by Evonik Industries AG (R9200; purity = 99.8%; BET = 150–190 m²/g; tapped density = 200 g/L; diameter of primary particle = 13 nm). A hexa-functional aliphatic urethane-acrylate (UA) and three-functional trimethylolpropane triacrylate (TMPTA) were supplied by Cytec Industries Inc; they were used as oligomer and reactive diluent, respectively. Butyl acetate (BA, purity ≥ 99%), used as solvent, was purchased from Sinopharm Chemical Reagent Beijing Co., Ltd. A radical photoinitiator (1-hydroxycyclohexyl phenyl ketone, Irgacure 184) was given by Ciba Specialty Chemicals. All the raw materials were used as received without further purification.

2.2. Milling process

BA, TMPTA and silica nanoparticles were mechanically mixed at a mass ratio of 5:4:1. The silica nanoparticle suspension was intensively stirred by a high-speed dissolver (DISPERMAR AE) at a rotation speed of up to 3000 rpm for 1 h. Then, the suspension was poured to a mixing tank of a bead mill (NETZSCH Machinery and Instruments Co., Ltd.) for further process. The configuration of the bead mill is schematically illustrated in Fig. 1. It is mainly composed of a 160 mL vessel, which contains zirconia beads (0.2 mm in diameter), a peristaltic pump and a mixing tank. The volume of the

zirconia beads equals about 80% of the vessel. During milling process, the suspension was injected by the peristaltic pump from the mixing tank into the vessel. The beads were intensively agitated by a rotor shaft at a speed of 1500 rpm, so as to break up nanoparticle agglomerations in the suspension. On the right side of the vessel, the suspension was filtered by a wire-cloth mesh with gap width of 0.1 mm to separate the beads and then it came back to the mixing tank. This cyclic milling process was repeated many times. In this work, the mass flow of the suspension was set as 10 kg/h; the milling time varied from 5 to 100 min, depending on the nature of the suspensions. To prevent generation of excess heat, the vessel was enclosed by a cooling jacket and completely sealed from the outside environment.

2.3. Coating preparation

After milling process, UA and photoinitiator were added to the suspension; the mixture was stirred at room temperature until a clear dispersion was obtained. A proper amount of dispersion was dropped onto a polycarbonate (PC) plate and spin-coated at a rotation speed of 4000 rpm. The coated PC plate was put in an oven of 60 °C for 8–10 min to remove solvent completely, and subsequently cured using a UV curing machine (Fusion UV Systems Co., Ltd.) for 60 s at light intensity of 560 mJ/cm². The thickness of the coating samples can be controlled in the range of 20–30 μm. The resultant samples contained 10 wt.% of silica nanoparticles.

2.4. Characterization

After milling process, the suspension having a fixed volume was poured to a centrifuge tube and was stored for 24 h at room temperature. After that time, some sediments often settled down on the bottom of the tube, leaving a clear boundary between sedimentation and supernatant, as illustrated in Fig. 3. Usually, a suspension with more sediments (i.e. unstable suspension) showed a clearer supernatant. Therefore, the height of supernatant may be used to evaluate the stability of the prepared suspensions roughly. The height of supernatant was measured by a ruler.

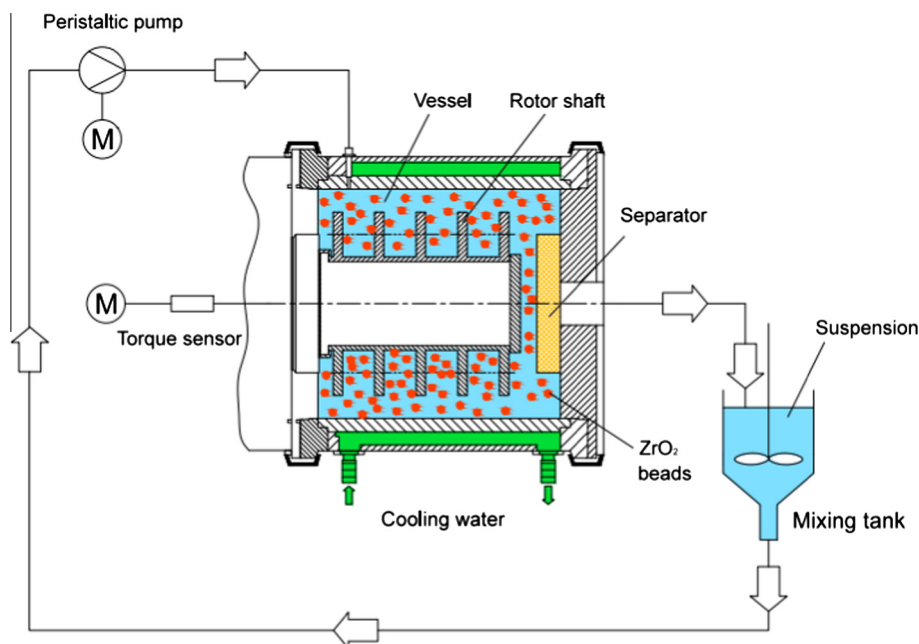


Fig. 1. Schematic of the bead milling system. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

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