



Study of the particles' structure dependent rheological behavior for polymer nanospheres based shear thickening fluid



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ABSTRACT

A novel kind of shear thickening fluid (STF) was developed via dispersing poly(styrene–acrylic acid) (PS–AA) nanospheres into ethylene glycol (EG). By varying the structure characteristics of the PS–AA particles, STFs with different rheological properties can be obtained. Firstly, the influence of the styrene/acrylic acid ratio on the PS–AA nanospheres was investigated. It was found that the higher ratio often led to the better shear thickening (ST) effects and under the optimum condition the maximum viscosity of the STF could reach to 152 Pa s, while the ST effects decreased under further increasing the monomer ratio. Then, the divinyl benzene (DVB) was introduced to increase the cross-link density of the PS–AA. In comparison with the non-cross-link PS–AA nanospheres, the poly(styrene–acrylic acid–divinyl benzene) (PS–AA–DVB) based STFs exhibited much higher ST effects and the maximum viscosity was up to 385 Pa s when the DVB was only increased to 0.3%. In combination of the rheological properties and the structure characterization, a possible mechanism for the ST behavior was proposed and the influence of the particles' characteristics on the mechanical performance of the PS–AA based STF was carefully analyzed.

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1. Introduction

When subjected to a strong impact, a liquid suspension with flowable behavior can become a solid like state with rigid behavior. This phenomenon was defined as shear thickening (ST) behavior, characterized by a pronounced increase in suspension viscosity when the shear rate reaches up to a critical value [1–3]. The liquid–solid phase transition is reversible, which means that the viscosity decreases immediately as soon as the shear rate decreases [3]. ST behavior initially attracted considerable interest due to its damage to process equipment and dramatic changes in suspensions microstructures, resulting in poor fluid and coating properties, which was believed to be a severe problem in industrial situations. The early investigations in ST behavior were to prevent this phenomenon for a given range of processing conditions or for specific applications. The STFs also have been utilized in many commercial applications including the ski boot cushioning, liquid coupling, shock absorber, rotary speed limiters, damping device, control devices, body armor [3–6], etc.

Based on their microstructure dependent macroscopic properties, two mechanisms order-to-disorder transition (ODT) and hydrocluster have been developed to analyze the ST behavior.

Hoffman [1,2,7] firstly proposed the ODT mechanism and the ST was ascribed to a transition from an order two-dimensional structure (where the particles were ordered into layers) to a more random three-dimensional structure at a critical shear rate. This phenomenon had been experimentally demonstrated by small angle light scattering [1,8] and the small angle neutron scattering measurement [9]. However, several researchers have questioned whether an order–disorder transition was necessary for ST, and some papers [10,11] stated that the order structure was not always observed prior to the onset of ST. Therefore, order–disorder transition may accompany ST, but is not prerequisite for ST. The hydrocluster mechanism proposed that ST was a consequence of a shear-induced non-equilibrium and self-organization of the particles into stress-bearing particle clusters. This self-organized microstructure was owing to the dominance of short-range hydrodynamic lubrication forces [12], which has been proven by many experimental results including the rheo-optical experiment [13], stress-jump rheological measurements [14], neutron scattering [15,16]. Very recently, Cheng et al. [17] identified and visualized the hydroclusters as the origin of ST in colloidal suspensions by combining fast confocal microscopy with simultaneous force measurements. The Stokesian dynamics simulations also supported this mechanism [18,19], therefore the hydrocluster mechanism was accepted as the standard model to explain the ST phenomenon.

Rheological properties of STFs were highly dependent on the suspended phase, the solvent [20,21], and the additives [22–25],

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in which the suspended phase was the most important factor. Barnes [3] pointed that all suspensions of solid particles would exhibit the ST phenomenon given the right circumstances. To our knowledge, the particles for the STF can be divided into three categories. Firstly, the ST phenomenon was originated from many important industrial process including minerals processing and tailings disposal, the forming of ceramic components, pumping of coal–water mixtures, construction with concrete, and pharmaceutical production [24], thus the particles early studied by researchers were clay, quartz powder, red iron oxide pigment, shale, limestone [3] and some inorganic particles such as silica [26,27], titania [28,29], calcium carbonate [30,31], alumina [32] and barium sulfate precipitated [33]. The second kind is the deformable particles such as blood cells [3], starch particles [34], wheat starch particles [35] and cornstarch particles [36]. The last kind is the man-made particles such as polyvinylchloride (PVC), poly(styrene–acrylonitrile) (PS–AN), polystyrene (PSt) [3], polymethylmethacrylate (PMMA) [20], poly(styrene–ethyl acrylate) (PS–EA) [37], carbon nanofibers [38], and the most popular silica particles for their easy preparation.

Due to the versatile molecular structures and surface properties, the polymer particles were very favorable in the STFs. Since the tunable hardness, charges, and elastic property, many polymer particles based STF exhibited strong ST behavior. The suspensions of 58 vol.% of PS–EA in EG exhibited a strong ST effect with a small initial viscosity under 10 Pa s at a static state but the maximum viscosity up to 10^4 Pa s at the transition in a steady shear test with a gap size of 800 μm [37]. However, the ST effect of the PMMA–STF was tender and more practical in the energy absorption under low intensity due to the low maximum viscosity in the ST region [20]. It was reported that the particle hardness exhibited great influence on the rheological properties of the STF which further affected the correlative STF–fabric performance [39,40]. Unfortunately, the detailed mechanism for this phenomenon was still expectative and the relative experimental evidences were needed. In the past decades, the influences of parameters of the particles such as the particle concentration [3,20], particle size [3,41], size distribution [28], sharp [42,30] and particle surface on the rheological properties of STFs (the onset of ST, the severity of ST, and the formation of hydrocluster) have been carefully studied. However, there has been comparatively little research into the influence of the inner structure of the suspended particles. Therefore, how about the particles' characteristics affected the ST behavior was emergent for understanding the essence of the ST behavior.

In this work, poly(styrene–acrylic acid) (PS–AA) nanospheres with different molecular structures were synthesized and dispersed into the EG solvent for the preparation of STF. The characteristics of the PS–AA nanospheres can be controlled by varying both the monomer molar ratio of styrene/acrylic acid and cross-linking reagent DVB. The rheological properties of these novel kind STFs were tested by using a rheometer and the particles' structure dependent ST behaviors was systematically investigated. Based on experimental results, a possible mechanism for the structural dependent ST behavior was proposed and the relative parameters were analyzed. This work was essential for developing new kind of STF system with better ST behavior, which further supplied novel understandings for the STF.

2. Experimental section

2.1. Materials

Acrylic acid (AA, Chemical Pure), Ethylene glycol (EG, analytical reagent), Potassium peroxydisulfate ($\text{K}_2\text{S}_2\text{O}_8$, analytical reagent), Sodium hydroxide (NaOH, analytical reagent), were used as re-

ceived. Styrene (St, Chemical Pure), Divinyl benzene (DVB, Chemical Pure), were distilled under vacuum before being used. All the reagents were purchased from Sinopharm Chemical Reagent Co., Ltd. Twice-distilled water was used in the present study.

2.2. The preparation of PS–AA particles

All polymerizations were conducted in a 500-ml three-neck flask, which was fitted with a reflux condenser, a mechanical stirrer and a nitrogen inlet. Given amount of the monomers and distilled water were firstly added into the flask. The mixture was vigorously stirred at about 300 rpm for 30 min at room temperature, and then the $\text{K}_2\text{S}_2\text{O}_8$ was added as initiator. After 10 min, the reactor was heated up to 75 °C in a water bath and maintained for 6 h. The polymerization was conducted under nitrogen atmosphere and the resultants were collected by centrifugation after the polymerization. The particles were rinsed by distilled water for three times. At last, the obtained nanoparticles were dried under vacuum at 50 °C over night.

Five kinds of PS–AA particles with different monomer molar ratios of styrene/acrylic acid at a fixed amount of styrene and six kinds of PS–AA particles with different amounts of cross-linking monomer DVB were synthesized. The ratios of styrene/acrylic acid were 3/1, 5/1, 7/1, 8/1, 9/1 and the amount of cross-linking monomer DVB were 0.1%, 0.2%, 0.3%, 0.4%, 0.5%, 0.6% according to the styrene. For simplicity, the DVB cross-linked PS–AA was defined as PS–AA–DVB.

2.3. The preparation of PS–AA based STF

Typically, the STFs were prepared by adding a given amount of PS–AA particles or PS–AA–DVB particles directly into ethylene glycol. Then the mixtures were mixed for 24 h in a ball crusher so as to obtain a uniform distribution of polymer particles within the suspension and to insure the particles were not aggregated. At last, the samples were sonicated for an hour in order to remove the air bubbles.

2.4. Rheological measurements

Rheological measurements were carried out on a stress and strain controlled rheometer (Anton-Paar MCR301) at 25 °C with cone-plate geometry having a cone angle of 0.2° and a diameter of 25 mm. Both steady-shear and oscillatory-shear tests were conducted on each sample with a gap size of 0.05 mm to measure the STF rheology under both static and dynamic loading conditions. For steady-shear test, a pre-shear of 1 s^{-1} was applied for 60 s prior to further measurement to remove loading effects.

2.5. Characterization

The field emission scanning electron microscope (FE–SEM, 20 kV) images were taken on a JEOL JSM–6700F SEM. Transmission electron microscopy (TEM) photographs were taken on a JEM–2011 with an accelerating voltage of 200 kV TEM. Infrared (IR) spectra were recorded in the wavenumber range 4000–400 cm^{-1} with a Nicolet 8700 Fourier transform infrared (FT–IR) spectrometer using a KBr wafer.

3. Results and discussion

3.1. Preparation and characterization of the PS–AA nanospheres

The PS–AA nanospheres with different inner structures were obtained by soap-free emulsion polymerization. In this work, the

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