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High performance shear thickening fluid based on calcinated colloidal silica microspheres

Sheng-Biao Zheng^{1,3}, Shou-Hu Xuan², Wan-Quan Jiang¹ and Xing-Long Gong²

¹Department of Chemistry, University of Science and Technology of China, Hefei 230026, People's Republic of China

²CAS Key Laboratory of Mechanical Behavior and Design of Materials, Department of Modern Mechanics, University of Science and Technology of China, Hefei 230027, People's Republic of China

³College of Chemistry and Materials Engineering, Anhui Science and Technology University, Fengyang 233100, People's Republic of China

E-mail: jiangwq@ustc.edu.cn and gongxl@ustc.edu.cn

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Abstract

Here, a novel method to prepare high performance shear thickening fluids (STFs) by dispersing calcinated silica microspheres into ethylene glycol is reported. The silica particles were prepared by hydrolyzing tetraethylorthosilicate (TEOS), and then they were treated under high temperature to remove the physically adsorbed water and the -OH groups on the surfaces. The influence of the temperature on the rheological properties of the final STFs was investigated and the STF prepared under the optimum temperature exhibited the best ST effects. A possible mechanism was proposed and it was found that a proper solvation layer adsorbed on the silica surface resulted in lower critical shear rate and higher shear thickening viscosity.

Keywords: shear thickening, silica microspheres, suspensions, rheology

(Some figures may appear in colour only in the online journal)

1. Introduction

Shear thickening fluid (STF) is a type of non-Newton fluids whose viscosity usually presented a sharp increase when it encountered an unexpected impact or shear. Due to this unique shear-thickening (ST) property, it has been widely applied in liquid coupling, shock absorbers, damping devices, control devices, rotary speed limiters, and body armor [1–6]. The STF was prepared by dispersing solid particles into the carrier solvent. It was proposed that these solid particles formed ordered two-dimensional structures in the suspension. If the external shear stress reached a critical level, ordered layers broke and jammed, causing the viscosity to rise [7–9]. Moreover, the reversible ST in concentrated dispersions was ascribed to the formation of jamming clusters arising from hydrodynamic lubrication forces between particles [10–14].

The solid particles played a critical role in determining the rheological properties of the STF. Various colloidal

particles, such as clay, calcium carbonate, silica, titania, starch particles, and polymer spheres, were important starting materials for preparing the high performance STFs [1–12, 14–17]. The intrinsic characteristics of the dispersing particles showed unique influence on the rheological behavior of the relative STFs. In comparison to the hard spheres, the soft spheres lead to a smaller yield strain; thus, a larger shear rate was needed for the ST phenomenon [18]. With increasing the crosslinking density of the polymer spheres, the ST effects improve. Besides the hardness, the interparticle forces also had a strong high influence on the ST behavior [19–22]. By increasing the repulsive forces between the dispersed particles, the starting shear rate for the ST increased. As the suspension presented ST behavior at volume fractions of particles near the maximum packing, the critical shear rate decreased with increasing volume fraction. Because the interparticle forces were highly dependent on the surface state of the solid particles, the study of the surface characteristics

has proven to be an essential part to investigate the ST mechanism [23, 24]. Several methods such as tuning the pH, ionic strength, and surface charge have been developed to improve the mechanical properties of the STFs [25–29]. It was found that the ST effects were not only dependent on the hydrodynamic interactions, but also dependent on the surface forces. Therefore, the optimum ST property should be obtained by controlling the surface characteristics of the solid particles.

Monodispersed silica microspheres based STFs were favorable because of their easy preparation and excellent mechanical properties. Various studies have been conducted on silica based STFs and their ST effects were controllable by altering the concentration, solvent, and additives [30–33]. It was reported that the silanol groups on the surface of the particles could lead to short-range electrostatic interactions, which would exhibit intensive influence on the ST effects. Moreover, the calcination can eliminate not only the physically absorbed water, but also decrease the –OH groups on the surface of the silica particles [23, 34–37]. To this end, the investigation of the heating treatment on the ST behavior for the silica particles based STF thereof is meaningful.

In this work, high performance STFs were prepared by using the calcinated silica particles as the dispersing phase. The influence of the heating temperature on the rheological properties of the STF was investigated and the optimum treating parameter was obtained. The inner structures and surface states of the calcinated silica microspheres were studied using Fourier-transform infrared spectroscopy (FT-IR), a scanning electron microscope (SEM), transmission electron microscope (TEM) and X-ray photoelectron spectroscopy (XPS) analysis. Finally, a possible mechanism for the improving ST effect was proposed. These results supplied valuable information for understanding the structural-dependent rheological properties in the concentration suspension.

2. Experimental section

The materials used in this study included tetraethylorthosilicate (TEOS, CP), ethanol (AR), ammonia (AR) and ethylene glycol (EG, AR) and were all purchased from Sinopharm Chemical Reagent Co., Ltd (China). Deionized water was made in the laboratory.

2.1. Preparation of monodispersed silica microspheres

Monodispersed silica microspheres were synthesized by hydrolyzing TEOS in a water-ethanol mixture containing ammonia according to the Stöber process [38]. The synthesis was conducted in a 2000 mL three-necked flask, which was fitted with a mechanical stirrer. Then, 1140 mL ethanol, 92 mL ammonia, and 49 mL deionized water were first added to the flask and stirred for 10 min in the 35 °C water bath. Then 54 mL tetraethyl orthosilicate was slowly introduced by dripping. After stirring for 6 h, 27 mL tetraethyl orthosilicate and 25 mL distilled water were added again. Following stirring for another 8 h, the white colloidal suspension was

obtained. Suspension was centrifuged and then the silica microspheres sediment was redispersed in ethanol and sonicated for 30 min. The silica nanoparticles were washed three times with ethanol and three times with deionized water. At last, the silica microspheres obtained were dried in a vacuum oven at 323 K and the final obtained particles were defined as p-SiO₂.

2.2. Calcination of monodisperse silica microspheres

The silica microspheres were calcined in a muffle furnace at 473 K, 673 K, and 873 K for 6 h at a heating rate of 10 °C min⁻¹. Then the calcination-treated silica nanoparticles were obtained and defined as c-SiO₂.

2.3. Preparation of calcinated silica microspheres based STF

The c-SiO₂ microsphere powders and ethylene glycol were added into the ball mill and then ground for 24 h at room temperature to insure even dispersion of the microspheres. Here, the ball mill is better than ultrasonic dispersion, magnetic stirring dispersion, and other methods because dispersing the silica particle clusters in EG and the surface roughness of the silica microspheres were not changed during the milling treatment. The volume fraction of the silica suspensions were kept at 61.2%. The resulting dispersions were sonicated for 1 h to remove entrained air bubbles. Then, the c-SiO₂ based STF was prepared. Here, a p-SiO₂ based STF was also prepared under a similar process.

2.4. Characterization

Morphologies of p-SiO₂ and c-SiO₂ were characterized by SEM, (Sirion 200, FEI, America) and TEM (JEM-2100, JEOL, Japan). The thermogravimetric (TG) measurements were carried out by the thermogravimetric analyzer (TGA Q5000IR, TA, America) over the temperature range from room temperature to 1073 K at 10 K min⁻¹ of the heating rate. The sample chamber was purged with dry nitrogen at 10 mL min⁻¹. FT-IR of these silica samples were characterized by FT-IR spectra, which was performed on Nicolet 8700 infrared spectrophotometer (Nicolet, USA). The surface chemistry of the p-SiO₂ and the c-SiO₂ were characterized by x-ray photoelectron spectroscopy (XPS, Thermo-VG Scientific ESCALAB 250Xi). Rheological measurements were conducted on a stress-and-strain-controlled rheometer (Anton-Paar MCR 301, Austria) with 25 mm diameter, 0.02 radian cone, and plate geometry. The small angle of the cone minimized edge failure of the sample and hence increased the limit on the highest achievable shear rate. All tests were performed in an Anton-Paar MCR 301 rheometer with a fixed gap size of 0.05 mm, due to the cone and plate geometry measurement tool, and measurement temperature was maintained at 25 °C with an automatic temperature control device.

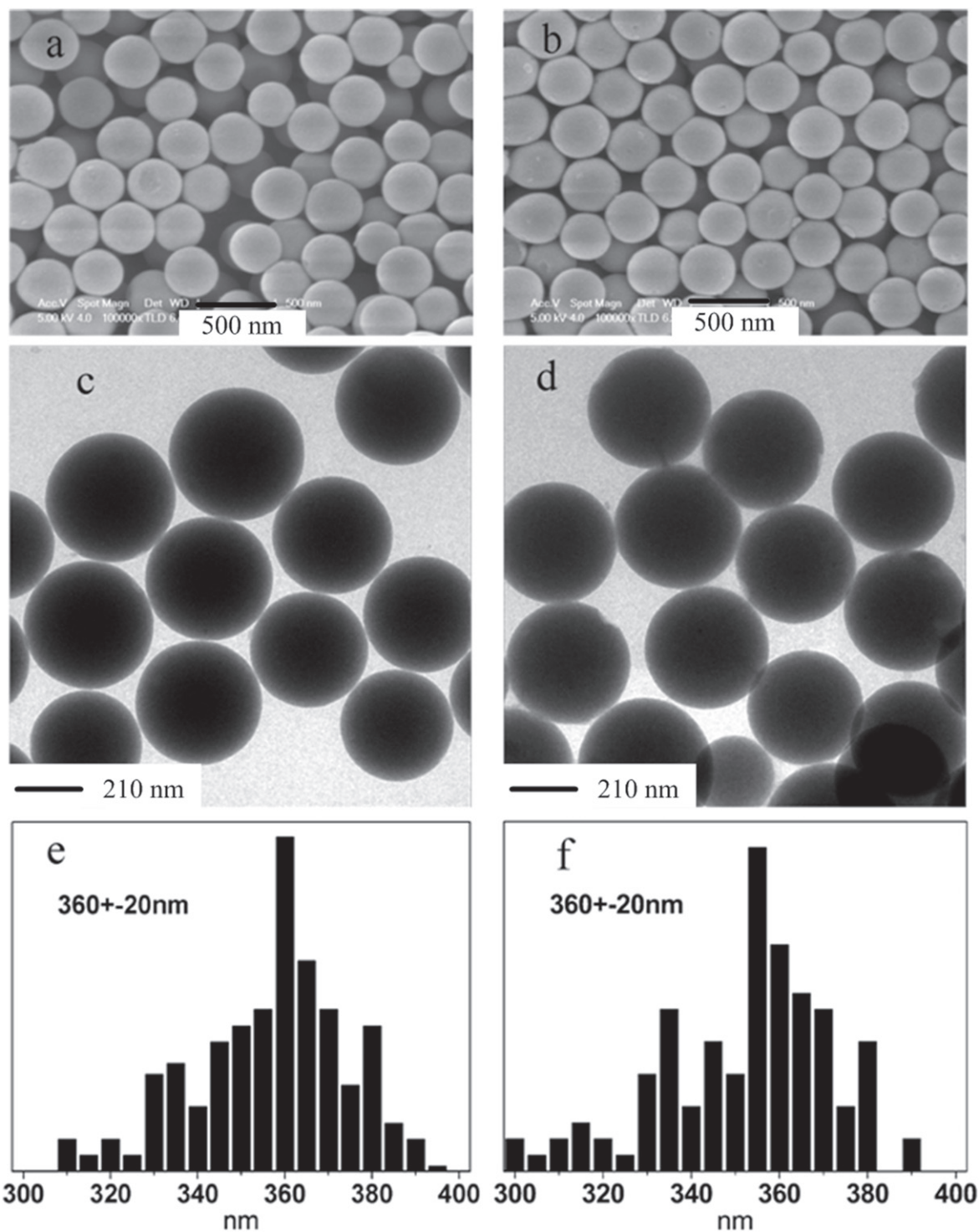


Figure 1. (a), (c) SEM and TEM images of the as-prepared p-SiO₂ and (b), (d) of c-SiO₂ (827 K) microspheres. (e), (f) are the particle size distribution histogram for p-SiO₂ and c-SiO₂ (827 K) microspheres, respectively.

3. Results and discussions

The monodispersed silica microparticles were prepared according the famous Stöber method by hydrolyzing TEOS in a water-ethanol mixture containing ammonia. Figure 1(a) presents the typical SEM image of the as-prepared p-SiO₂ microspheres, in which we can find that all the particles are spherical and the average diameter of the microspheres is

about 360 ± 20 nm, according to statistics from the SEM photograph. Here, the calcination treatments show little effect on the morphology of the final particles. The c-SiO₂ possess uniform distribution, are well dispersed on the copper grid without large aggregations, and demonstrate that no melt is occurring during calcination. To investigate the inner nanostructure of the p-SiO₂ and c-SiO₂ microspheres, the TEM was further applied to analyze the morphology.

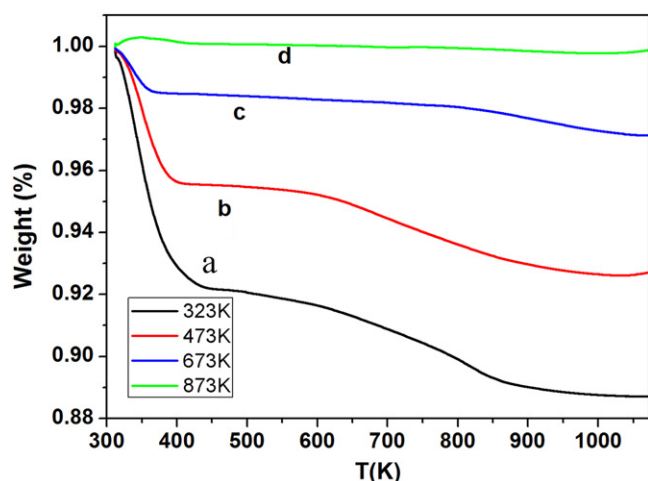


Figure 2. TG curve of the silica nanoparticles (a)–(d).

Observably, both the two particles are of same size and size distribution, which agrees well with the SEM analysis.

In this work, the c-SiO₂ microspheres were obtained by calcinating the p-SiO₂ under different temperatures. Although few changes were found in their morphologies, some differences were present in their TG curves (figure 2). For p-SiO₂, the TG curve demonstrated an 11 wt% weight loss, which must be responsible for the release of the adsorbed water and diminishing of the -OH groups on the surfaces. In contrast, the weight loss for the c-SiO₂ (473 K), c-SiO₂ (673 K), and c-SiO₂ (873 K) were 7, 2, and 0 wt%, respectively. Under higher pre-calcination temperature, more adsorbed water would be released, thus the relative c-SiO₂ possessed a smaller weight loss in the TG curve. Because of the small weight loss, no critical reduction of the diameter size was found for the silica microspheres. However, the densities of the particles were decreased after the calcination treatment. The densities of the silica particles were obtained from solution (ethanol) densitometry measurements at various solid weight fractions using a volumetric balance assuming ideal mixing. The results indicated that the p-SiO₂ particle density was $\rho = 1.78 \pm 0.01 \text{ g cm}^{-3}$, and the calcined silica particle densities were 1.70 ± 0.01 (473 K), 1.66 ± 0.01 (673 K), and $1.57 \pm 0.01 \text{ g cm}^{-3}$ (873 K), respectively. The decreased apparent density resulted from the presence of porous structure in calcined particles due to removal of the residual organic ethoxyl from the incomplete hydrolyzing TEOS, the internal water, and the deeper silanol groups during the heating process.

The p-SiO₂ and c-SiO₂ microspheres could be dispersed into ethylene glycol to form the relative STF. Here, to compare the calcination influence on the ST effects, all four samples were prepared by keeping the volume fraction as a constant (61.2 v/v%). Figure 3 showed the relative rheological behaviors under both steady shear and oscillatory shear. Taking the p-SiO₂ based STF as an example, its viscosity undergoes a shear-thinning and ST process. Under lower shear rate, the viscosity decreased and it was always present in the traditional STFs. As soon as the shear rates increased to

123 s^{-1} , the viscosity sharply increased, indicating typical ST behavior. The c-SiO₂ (473 K) and c-SiO₂ (673 K) based STFs exhibited a similar phenomenon. Unfortunately, only very weak ST was found in c-SiO₂ (873 K). The shear-rate-dependent viscosity demonstrated that the calcination shows high influence on the ST effects of the STF thereof. First, with an increase of the treating temperature, the critical shear rates for the relative STFs first increased and then decreased. The critical shear rates of the suspensions were 123 s^{-1} (323 K), 10 s^{-1} (473 K), 15 s^{-1} (673 K), and 30 s^{-1} (873 K), respectively. Second, the maximum viscosity of ST dramatically increased from 40 pa·s (323 K) to 265 pa·s (473 K), decreased to 40 pa·s (673 K) and 11 pa·s (873 K) at corresponding calcined temperature. Based on the preceding results, we can conclude that the 473 K is the optimum treating condition to improve the ST effects.

Clearly, the calcination plays a critical role in determine the rheological properties of the final suspension. After calcination, the change of the silica is solely the silanol groups and physically adsorbed water on the surface of the silica particle. It was reported that the surface characteristic highly influence the ST effects through the silanol groups. The coverage of the silanol (Si-OH) groups on the fumed silica was about 2.5 silanol groups nm^{-2} , while the value was 8.2 silanol groups nm^{-2} for the silica particles prepared via the Stöber process [34]. Zhuravlev reported the number of silanol groups per square nanometer is 4.6, this number was suggested as a physicochemical constant of dehydrated but fully hydroxylated amorphous silica, independent of origin and structural characteristics of the silica [35–37]. The silica surface silanol groups are the main centers of adsorption, bonding water molecules with hydrogen bonds on the silica. Therefore, the calcination affected the ST effects via changing the surface functional group on the silica microspheres.

FT-IR spectra of the raw and calcined (under various temperatures) silica nanoparticles were shown in figure 4. Both silanol groups (chemical bonded) and physically adsorbed water (hydrogen bonded) exist on the surface of silica. They are different from the bonding form; thus, water on the surface could be dehydrated with increasing calcined temperature. It was reported that the silanol groups and physically adsorbed water on surface were 8.2 nm^{-2} and 9.8 nm^{-2} , respectively [34]. From the TG curve, we find the physically adsorbed water was removed mainly at 350 K. Therefore, in comparison to the p-SiO₂, the infrared absorption of H₂O at 3465 cm^{-1} and 1633 cm^{-1} for c-SiO₂ (473 K) microspheres decreased obviously, indicating that the silanol groups are hardly decreased but the physically adsorbed water was mostly removed. The removal of the physically adsorbed water facilitated the adsorbing ethylene glycol to form a solvation layer on the surface of silica nanoparticles, which gives rise to short-range, non-DLVO repulsions ('solvation forces') and stabilizes the silica particles.

The XPS is a very good technique for detecting the surface state of the particles [39, 40]. Figure 5 presents the typical XPS spectra of the silica particles treated under different calcinations temperatures, which indicates all the particles have a similar surface state. The intense peak at 285 eV,

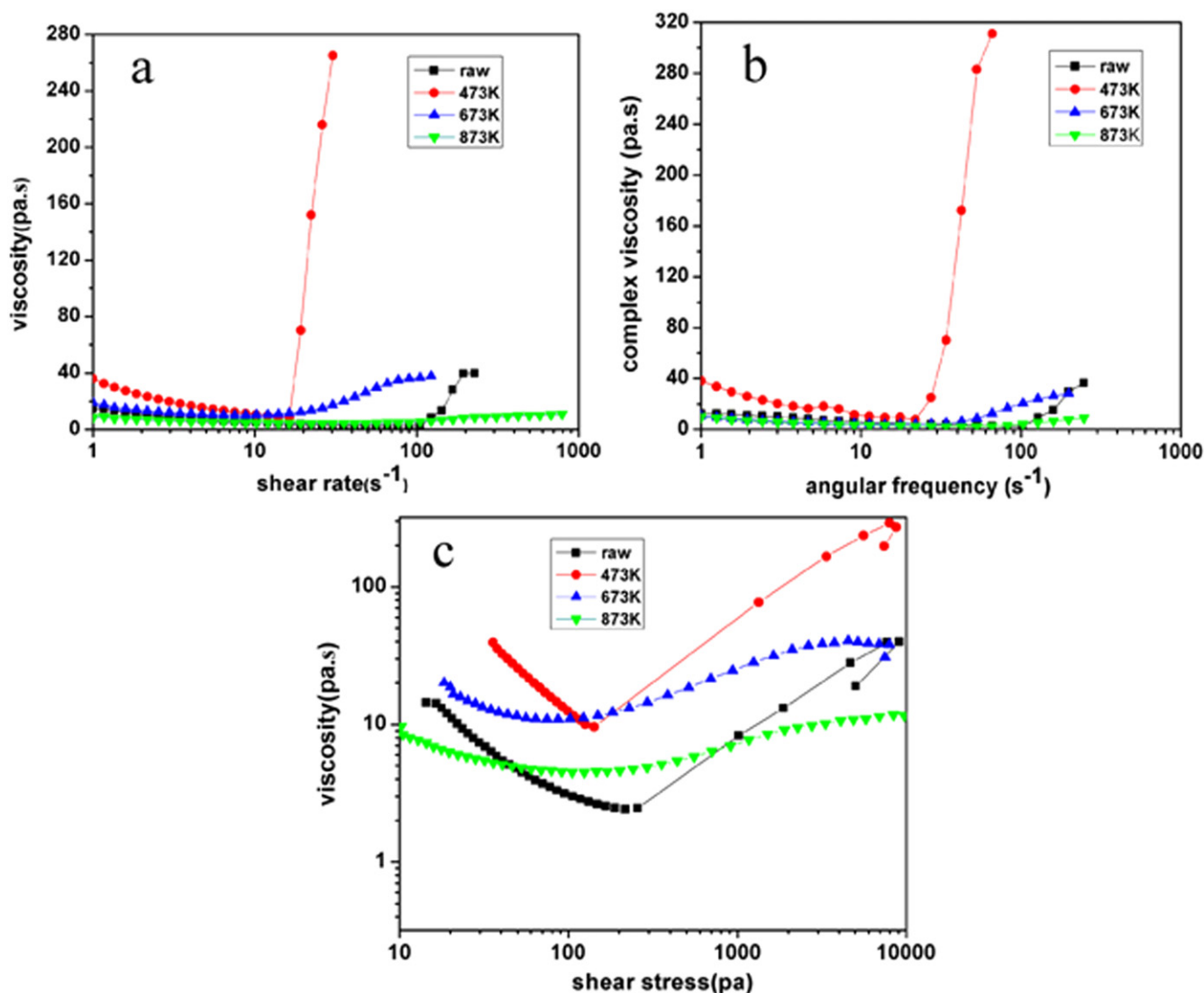


Figure 3. (a) Viscosity versus shear rate for different calcinations temperatures; (b) complex viscosity versus angular frequency for different calcinations temperatures; and (c) viscosity versus shear stress for different calcinations temperatures. The suspension volume fraction was the same 61.2%.

as a reference for other peaks, is from the C element, which resulted from the residual organic ethoxyyl due to the incomplete hydrolyzing TEOS. O 1s and its content change as a function of heating temperature are clearly observed in figure 5(b). The content of oxygen on the silica microspheres surface reached the maximum at 473 K, and then decreased with increasing calcined temperature. The atom ratio of Si to O was found to decrease from 2.2 to 2.0 with increasing heat treatment temperatures (the former ratio being higher than 2 because of the contribution of silanol groups on the surface). These XPS results suggest that the maximum of the silanol groups appears at 473 K. When the calcined temperatures rise higher, the numbers of the silanol groups on the silica microspheres surface become lower. More silanol groups lead to a more adsorbed liquid molecule, which further facilitates the thicker solvation layer. Therefore, a high ST effect was obtained.

For given particles and liquid suspension, the critical shear rate is closely related to the volume fraction; the higher volume fraction compresses particles closer and tends to form clusters at low shear rate [22]. Previous results suggested that the critical stress was independent of the volume fraction, but sensitive to particle size and the critical stress decreased with increasing particle size [41, 42]. The magnitude of the ST effect progressively increased with silica volume fraction while at the same time the point of incipient ST was shifted toward lower shear rates [19].

The formation of a solvation layer not only increases the repulsive force but also increases the solid volume fraction by increasing the effective particle size. As the calcined temperature increased from 473 K to 673 K and 873 K, the magnitude of the ST effect decreased, even lower than the raw silica suspension, at the same time the critical shear rate decreases. These results agreed well with previous reports.

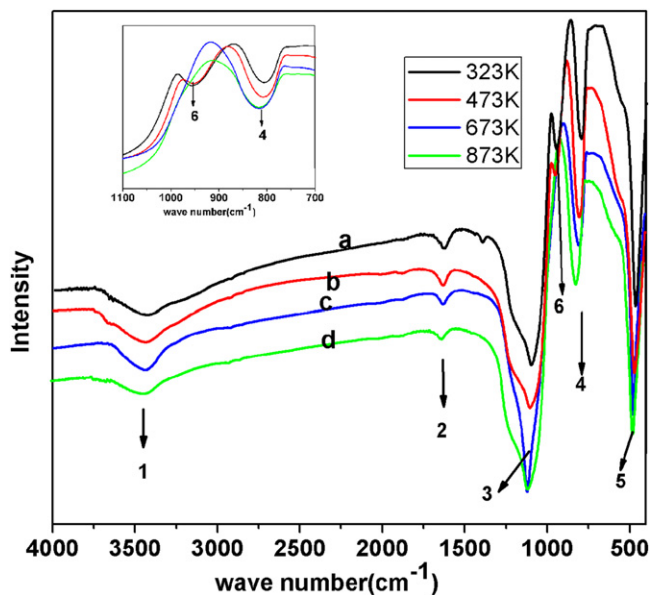


Figure 4. FT-IR spectra of the silica nanoparticles (a)–(d).

The schematic illustration of the effect of silica-calcined treatment via the change of silanol groups on the rheological properties is shown in figure 6. The chemisorption water (silanol groups) on the surface started to condense and evolve water extensively with the increasing calcined temperature, especially the temperature beyond 473 K, which is the threshold temperature for the beginning of dehydroxylation. The infrared absorption of bending vibration at 950 cm^{-1} (paired vicinal silanol groups are shown in table 1) disappeared when the calcined temperature reached over 673 K, as shown in figure 4. It indicated that the silanol groups were removed gradually with the increasing temperature of calcination treatment. The reduction of silanol groups weakened the solvation repulsive force due to the decreased solvation layer. The silanol groups on the silica surface give rise to short-range repulsive forces, and the rate of the surface silanol

Table 1. Peaks of infrared absorption and the function groups of silica particles.

Peak	Wavenumber	Molecular function group
1	3465	molecular H_2O
2	1633	molecular H_2O
3	1100	asymmetric stretching vibration of $\equiv\text{Si}-\text{O}-\text{Si}\equiv$
4	800	symmetric stretching vibration of $\equiv\text{Si}-\text{O}-\text{Si}\equiv$
5	470	bending vibration of $\equiv\text{Si}-\text{O}-\text{Si}\equiv$
6	950	bending vibration of $\equiv\text{Si}-\text{OH}$

groups was proportional to the magnitude of the short-range repulsive forces, which resulted in ST. Therefore, under low calcination temperature (473 K), there are enough silanol groups on the surface of the silica to support the ST layer, thus they presented the best ST effects. Once the temperature is too high, not only the adsorbed water but also the silanol groups gradually decreased, the hot point for the ST layer decreased, therefore, the ST effects sharply decreased.

4. Conclusions

This work reported a high performance STF that was prepared by calcinating the dispersing silica microparticles. The influences of the calcination temperature on the rheological properties of the relative STFs were discussed. It was found that the physically adsorbed water and silanol groups on the surface of the silica microspheres were highly dependent on the calcination temperature. The 473 K was proven to be the optimum treating temperature for the STF. A possible mechanism was proposed to analyze the temperature effects and it was found that the surface silanol groups on the silica particles served as the centers for the solvation layer adsorbed on the silica surface. The higher ST degree caused by the

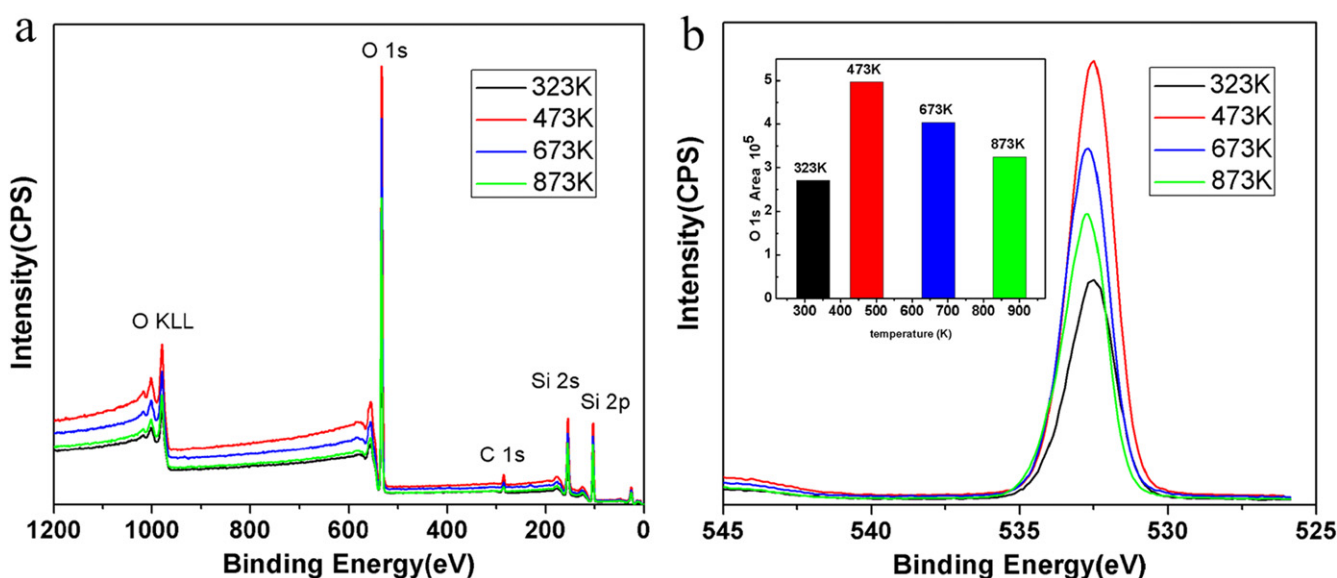


Figure 5. (a) XPS spectra of silica particles for different calcinations temperatures and (b) expanded view of O 1s.

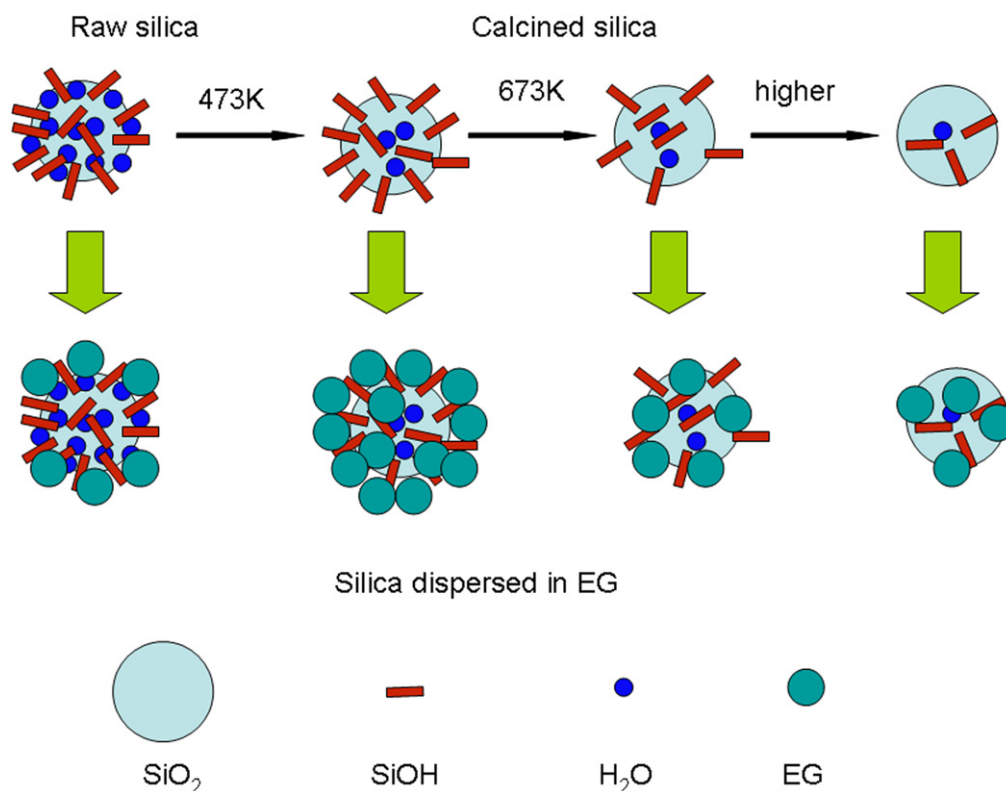


Figure 6. Effect of the calcined temperature on the structure of the silica nanospheres.

solvation layer gave rise to a repulsive interparticle force. This result gives valuable information to further understand the mechanism for the ST effects.

Acknowledgments

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