

[Article ID] 1003– 6326(2001)05– 0768– 06

Aggregation/dispersion of ultrafine silica in flotagent solution^①

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[Abstract] The aggregation/dispersion of ultrafine particles is of interest for both fundamental and practical perspective. These behaviors of ultrafine silica in flotagent solution and the heter coagulation of silica and alumina were examined using particle size analyzer, electrokinetic potential, contact angle measurements. The flotation reagents have a pronounced effect on the aggregation or dispersion behaviors of ultrafine silica suspensions. Collector dodecylamine chloride renders silica surfaces hydrophobic and the aggregation between silica particles takes place. Modifier tripolyphosphate makes the silica surface completely hydrophilic and enhances the stability of silica suspension. These experimental results can be explained based on the extended DLVO theory by considering polar interfacial interaction between particle surfaces.

[Key words] aggregation; dispersion; silica; flotagent; polar interfacial interaction

[CLC number] TD923

[Document code] A

1 INTRODUCTION

The stability of ultrafine particle suspension is very important in many area of technology such as flotation, selective flocculation. Silica is the dominant component of most silicate gangue minerals in flotation such as quartz, kaolinite, illite, and pyrophyllite. In this regard, the studies on the nature of interaction between silica particles in some reagent solution will improve our understanding of the aggregation/dispersion behavior for silica system.

The interfacial interaction forces between particles govern the aggregation/dispersion of ultrafine particle suspension. These interaction forces have been recognized to include van der Waals forces, electrostatic forces, structural forces arising from the interactions between hydrophobic or hydrophilic particle surfaces, magnetic forces in some magnetic systems and steric forces between particles adsorbing macromolecules^[1~4]. DLVO theory is only of limited utility in describing suspension stability^[5,6]. It has been found that there may exist non-DLVO forces between two surfaces in some polar media such as water, which are repulsive between hydrophilic surfaces, and attractive between hydrophobic surfaces^[4,7,8].

For hydrophilic surfaces, the existence of non-DLVO forces was recognized much earlier^[9]. More recently, many investigators conducted surface force measurements for hydrophilic systems using various surface force apparatus (SFA)^[10,11], and atomic force microscopy (AFM)^[12,13], and confirmed the existence of the repulsive hydration force in the sym-

metric hydrophilic systems. Many investigators demonstrated that the effective range which the hydration forces operate is less than 5 nm^[13~15]. It has also been reported that there are measurable long range attractive hydrophobic forces between two hydrophobic surfaces such as methylated silica surfaces, natural coal surfaces^[16], and solid surfaces covered with surfactants^[17,18]. The various SFA and AFM are also used for the measurement of hydrophobic force curves^[4,19,20].

The origin and nature of these forces have long been controversial. For hydrophilic system, it appears that a change in the structure of water may produce repulsive force^[13,21,22]. Israelachvili believed that the repulsive hydration forces arise from strong H-bonding surface groups, such as hydrated ions or hydroxyl (—OH) groups, which modify the H-bonding network of liquid water adjacent to them^[23]. For hydrophobic systems, some investigators considered it to be related to the metastability of the water film between hydrophobic surfaces^[24,25]; while others believed that it is entropic in origin, arising mainly from the configurational rearrangement of water molecules in the vicinity of hydrophobic surfaces^[26,27]. Some authors proposed the capillary force due to cavitation in the vicinity of hydrophobic surfaces^[28,29].

van Oss et al^[30,31] reported that these structural interaction forces, which are of polar either in the attractive (hydrophobic interaction) or in the repulsive mode (hydration pressure) represented energies that may be up to two orders of magnitude higher than those commonly encountered in traditional DLVO

① **[Foundation item]** Project (G1999064901– 1) supported by the National Key Fundamental Research Program; Project (59925412) supported by National Distinguished Youth Scholar for Science Fund **[Received date]** 2000– 11– 03; **[Accepted date]** 2001– 03– 23

theory. They proposed a polar interfacial interaction theory. Skvarla and Kmet^[8] used this approach to discuss the aggregation and dispersion of fine magnetite in sodium oleate solution. HU et al^[32] investigated the interactions between fine hematite particles in sodium oleate solution and between fine wolframite particles in styryl phosphonic acid solution in the light of the van Oss theory.

At present work, the hydrophobic interaction and hydration repulsion between silica particles, and the aggregation/dispersion of ultrafine silica are studied based on the polar interfacial interaction theory.

2 EXPERIMENTAL

2.1 Materials

Quartz ($d = 10 \mu\text{m}$) was purchased from Harric Scientific Co.. Ultrafine silica powder ($< 1 \mu\text{m}$, 99.9% purity) was purchased from Sigma Chemical Co.. The chemicals used in the present study include: technical grade sodium tripolyphosphate (GFS Chemicals), reagent grade dodecylamine hydrochloride from ACROS Organics, glycerol (EM Science), diiodomethane (Aldrich Chem. Co.), formamide (Mallinckrodt Inc.). HCl and NaOH (AR) were used for pH modification. A Milli-Q water system (Millipore) supplied with distilled water provided high-purity water with a resistivity of $+ 18 \text{ M } \Omega$ and a surface tension of 72 ± 0.2 at $23 \text{ }^\circ\text{C}$.

2.2 Zeta potential, particle size and contact angle measurements

The zeta potential and particle size of silica suspensions were measured by standard procedures with a Malvern Zeta Sizer III using a quartz capillary cell with a 4 mm diameter. The ultrafine alumina was added into a 100 mL beaker containing an aqueous solution of known composition. The solid concentration was about 0.02%. The suspension were agitated for 10 min and transferred to the capillary cell with a syringe, and then the zeta potential measurements were made in the electrophoretic mode and the distribution of particle size was measured in the size analyzer mode. The sessile drop technique was used for contact angle measurements with a NRL goniometer (Rame-Hart, Inc.)^[5].

3 RESULTS AND DISCUSSION

3.1 Aggregation/dispersion behavior of silica in absence and presence of flotagent

The particle size distribution curves of silica suspension in the absence and presence of flotagents are, respectively, given in Fig. 1 and Fig. 2. Fig. 1 shows that the distribution of coarser particle silica is increased in the surfactant solution than in deionized water. The average particle size of silica suspension is

about $3.03 \mu\text{m}$ in deionized water and increased to $4.83 \mu\text{m}$ in 10^{-4} mol/L dodecylamine chloride (DDA) solution at pH 6.5~6.7, indicating that the aggregation between silica particles occurs. Instead, the coarser parts in silica suspension are decreased in modifier solution. It can be seen from Fig. 2 that the average particle size of silica suspension is decreased from $3.03 \mu\text{m}$ to $2.44 \mu\text{m}$ by addition of 10^{-4} mol/L tripolyphosphate (TPP).

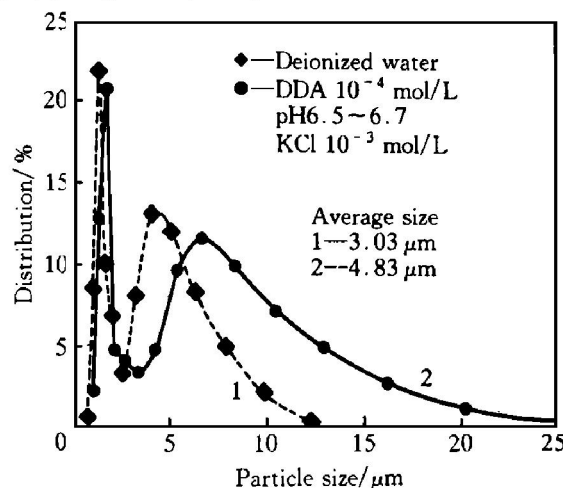


Fig. 1 Effect of dodecylamine chloride on particle size distribution of silica suspension

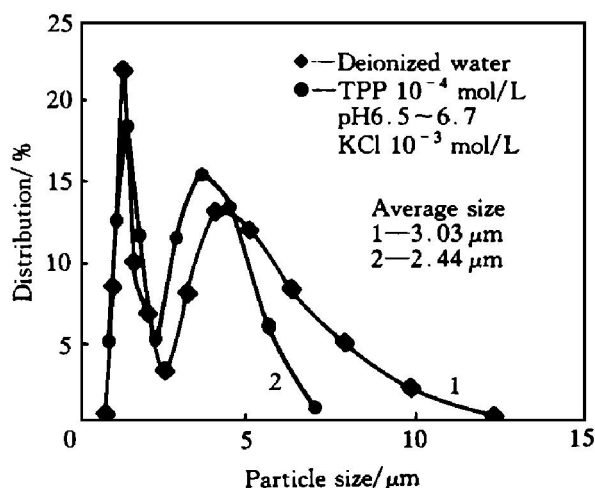


Fig. 2 Effect of tripolyphosphate on particle size distribution of silica suspension

3.2 Electrokinetic behavior

The zeta potentials of silica in the presence and absence of collectors are shown in Fig. 3 as a function of pH. It follows that the silica is negatively charged at wide pH range and PZC occurs at about pH 1.8. The addition of 10^{-4} mol/L dodecylamine chloride decreases the negative zeta potential of quartz and produces a PZC at about pH 3.2. When adding 10^{-4} mol/L TPP the negative zeta potential of silica moved to more negative with a value of -60.46 mV .

3.3 Wettability and surface energy

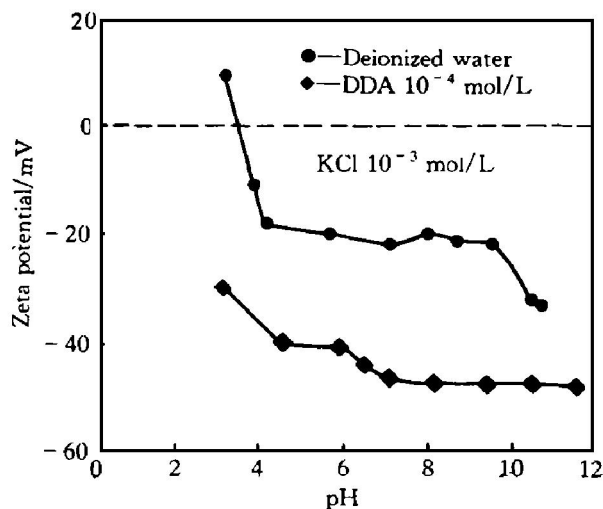


Fig. 3 Zeta potential of silica in deionized water and in dodecylamine solution vs pH

According to van Oss's surface thermodynamic approach, the surface energy of a solid as well as the solid-liquid interfacial energy is determined by two components. The first is the apolar (Lifshitz-van der Waals, LW) component γ^{LW} and the second is the polar (Lewis acid-base, AB) component γ^{AB} . van Oss et al.^[30, 31, 33-36] considered the polar interactions as electron acceptor-electron donor interactions, or Lewis acid-base (AB) interactions, which were essentially asymmetrical and could only be satisfactorily treated by taking that asymmetry into account. They designated the symbol γ_i^- to express the parameter of the polar component (γ_i^{AB}) of the surface tension of compound i , γ_i^- is due to the electron donor or proton acceptor, and γ_i^+ to express the electron acceptor or proton donor parameter of the γ_i^{AB} .

The Lifshitz-van der Waals (LW) interfacial tension (energy) γ_{SL}^{LW} can be written as

$$\gamma_{SL}^{LW} = \left[\sqrt{\gamma_S^{LW}} - \sqrt{\gamma_L^{LW}} \right]^2 \quad (1)$$

The polar component of solid-liquid interfacial tension can be written as

$$\gamma_{SL}^{AB} = 2 \left[\sqrt{\gamma_L^+ \gamma_S^-} + \sqrt{\gamma_S^+ \gamma_L^-} - \sqrt{\gamma_S^+ \gamma_L^-} - \sqrt{\gamma_S^- \gamma_L^+} \right] \quad (2)$$

And related to contact angle by using the Young-Dupre equation,

$$\gamma_L(1 + \cos \theta) = 2 \left[\sqrt{\gamma_S^{LW} \gamma_L^{LW}} + \sqrt{\gamma_S^+ \gamma_L^-} + \sqrt{\gamma_S^- \gamma_L^+} \right] \quad (3)$$

Thus, by contact angle (θ) measurements with three different liquids (of which two must be polar) with known γ_L^{LW} , γ_L^+ , γ_L^- values, using Eqn. (3) three times, the values γ_S^{LW} , γ_S^+ and γ_S^- of any solid can be determined. Similarly, by contact angle measurements of a liquid on various solids (of which two must be polar), the γ_L^{LW} , γ_L^+ and γ_L^- can be determined. It is always necessary to determine (or to

know) the value of γ_L .

The interfacial interaction energy parameters were obtained by using the following relationship^[31]. For the LW interactions between similar solid surface (1) in a liquid (3),

$$\Delta G_{131}^{LW} = -2 \left[\sqrt{\gamma_S^{LW}} - \sqrt{\gamma_L^{LW}} \right]^2 \quad (4)$$

and for the AB interactions between similar solid surface (1) in a liquid (3),

$$\Delta G_{131}^{AB} = -4 \left[\sqrt{\gamma_L^+ \gamma_L^-} + \sqrt{\gamma_S^+ \gamma_S^-} - \sqrt{\gamma_S^+ \gamma_L^-} - \sqrt{\gamma_S^- \gamma_L^+} \right] \quad (5)$$

When $\Delta G_{131}^{AB} < 0$, it defines the hydrophobic AB or hydrophobic structural interactions. When $\Delta G_{131}^{AB} > 0$, it defines quantitatively the hydrophilic AB or hydrophilic structural interactions.

Table 1 presents the average contact angles for quartz surfaces in various solutions. In deionized water, the water contact angles with quartz is zero, showing a hydrophilic surface. In the DDA cationic surfactant solution, the water contact angles with quartz at pH > 2 are increased and thereby hydrophobicity enhanced. Treated in modifier solution of 10⁻⁴ mol/L TPP, quartz surfaces exhibit zero contact angles with water and under this condition quartz become very hydrophilic. Different contact angles of various liquids on quartz surface at different conditions as seen in Table 1 show that the polar interfacial interaction changes with the conditions.

Table 1 Advancing contact angles of various apolar and polar liquids at quartz surface for different solution conditions

Solution conditions		Average contact angle θ / (°)				
Reagent / (mol·L ⁻¹)	pH	W	GL	FM	DM	
Deionized water		0	21.2	0	30.7	
DDA: 5 × 10 ⁻⁵	6.7~6.8	45	40	29	39.9	
	10 ⁻⁴	6.7~6.8	57	51	41	40
	5 × 10 ⁻⁴	6.5~6.7	81	76	65	49
TPP 1 × 10 ⁻⁴	9~9.2	0	22.8	0	33.4	

The values for the various components of surface energy of quartz corresponding to the solution conditions in Table 1 are calculated on the basis of Eqn. (3) and presented in Table 2. With the data in this table and in the light of Eqs. (4) and (5), the energy parameters of Lifshitz-van der Waals and interfacial polar interactions in various systems between quartz particles are calculated and shown in Table 3. It can be seen from Table 2 that at different solution conditions the Lifshitz-van der Waals component of interfacial tension γ_S^{LW} shows not too much difference, however, the electron-donor γ_S^- and electron-acceptor γ_S^+ components are evidently different, which will result in different polar interfacial interactions between silica particles. As illustrated in Table

3, the Lifshitz-van der Waals interaction energy parameters are negative at various solution conditions, indicating an attractive van der Waals forces. However, the polar interfacial interaction energy parameters are positive for silica in deionized water and in tripolyphosphate solution, and negative in dodecylamine chloride solution. These results demonstrate that the repulsive polar interfacial interaction forces may exist between silica surfaces in deionized water and TPP solution, and attractive interaction forces exist in DDA solution.

Table 2 Values of components of surface and interfacial energies of quartz at different solution conditions

Solution condition		Surface energy γ /(mJ·m ⁻²)			
Reagent/(mol·L ⁻¹)	pH	$\sqrt{\gamma_S^{LW}}$	$\sqrt{\gamma_S^+}$	$\sqrt{\gamma_S^-}$	
Deionized water	6.7	5.77	1.82	7.27	
DDA	5 × 10 ⁻⁵	6.7~6.8	5.71	1.66	5.36
	10 ⁻⁴	6.7~6.8	5.81	1.25	4.51
	5 × 10 ⁻⁴	6.6~6.7	5.59	0.15	3.0
TPP	1 × 10 ⁻⁴	9~9.2	5.95	1.8	7.5

Table 3 Energy parameters of interactions between various particles

System conditions		Energy parameters / (mJ·m ⁻²)	
Particle (1) / Water(3) / Particle (1)	Reagent / (mol·L ⁻¹)	ΔG_{131}^{LW}	ΔG_{131}^{AB}
	Deionized water	-2.424	28.678
SiO ₂ /W/SiO ₂ , pH6.5~6.7	DDA: 10 ⁻⁴	-2.604	-8.208
SiO ₂ /W/SiO ₂ , pH9.2	TPP: 10 ⁻⁴	-3.28	31.85

3.4 Interaction forces between silica surfaces

In the classical DLVO theory, the sum of the repulsive electrostatic interaction force F_E and the attractive van der Waals force F_W arrives at a total net DLVO interaction forces (F_T^D) for a given separation as following:

$$F_T^D = F_W + F_E \quad (6)$$

The repulsive electrostatic interaction force decays approximately exponentially with the distance of separation (D). For two similar spheres of radius R , according to the constant surface potential model F_E is given by

$$F_E = 2\pi\epsilon_a R \kappa \varphi_0^2 \exp(-\kappa D) \quad (7)$$

where ϵ_a is the absolute dielectric constant of the medium, for water $\epsilon_a = 6.95 \times 10^{-10} \text{ C}^2/\text{m}$; R is radius of the particles; φ_0 is potential at the particles surface, here the measured zeta-potential was taken as the relevant potential; κ is the inverse Debye length equal to 0.104 nm^{-1} for 10^{-3} mol/L 1:1 elec-

trolyte at 298 K; D is the separation.

The van der Waals attractive force F_W can be calculated using the classical expression with known Hamaker constant. It can also be obtained according to the surface energy measurements^[26,27] as below,

$$F_W = \pi R \Delta G^{LW} \left[\frac{D_0}{D} \right]^2 \quad (8)$$

The equation describing the decay with distance of polar interfacial interaction forces for two spheres of radius R is

$$F_{AB} = 2\pi R \Delta G_{131}^{AB} \exp\left[\frac{-D}{h_0} \right] \quad (9)$$

where h_0 is the decay length, here h_0 is taken to be 1 nm for hydrophilic system and 2 nm for hydrophobic system; D_0 is the minimum equilibrium contact distance between particles, $D_0 = 0.158 \text{ nm}^{[14]}$ or $0.163 \text{ nm}^{[11]}$.

The total extended DLVO energy is

$$F_T^{ED} = F_{LW} + F_E + F_{AB} \quad (10)$$

In order to further explain the aggregation/dispersion of ultrafine silica in various solutions, we can calculate the interaction forces between the silica particles under these conditions. The electrostatic interaction forces were calculated by Eq. (7) and the data in Fig. 3. The Lifshitz-van der Waals and interfacial polar forces were calculated in the light of Eqs. (8) and (9) and the data in Table 2 and 3. The total DLVO and EDLVO forces are obtained by Eqs. (6) and (10).

The interaction force curves between silica particles in the absence and presence of DDA at pH 6.5~6.7 are shown in Fig. 4. It may be seen from Fig. 4 that no matter whether or not adding DDA, DLVO force profiles demonstrate that a low force barrier exists between silica particles at some distance and disappears at shorter distance. It suggested that almost same results be predicted for silica interactions in the

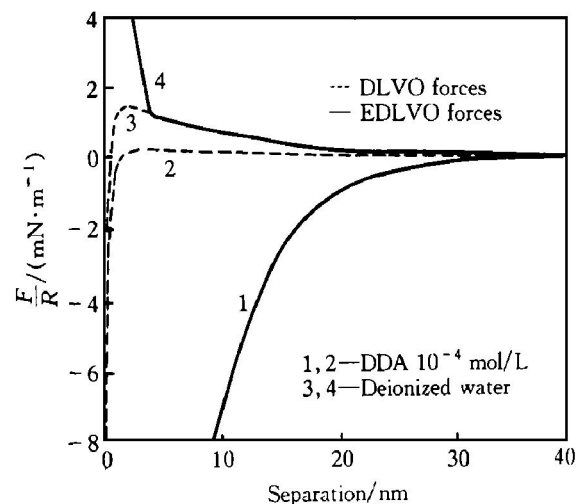


Fig. 4 DLVO and extended DLVO force profiles between silica surfaces in deionized water and dodecylamine (DDA) solution

absence and presence of dodecylamine chloride from DLVO theory, which can not explain the different aggregation/dispersion behavior of silica suspension at the different conditions. EDLVO force curves demonstrate that there is always a repulsion between silica particles at pH 6.5~6.7 in pure water and a strong attraction between silica particles in DDA solution. It means that the aggregation in ultrafine silica particle suspensions will take place by addition of 10^{-4} mol/L DDA as shown in Fig. 1. In the light of Eq. (9) and Eq. (10), the attractive interaction between silica particles adsorbed DDA can be attributed to the polar interfacial interaction due to the surface hydrophobized.

Fig. 5 shows the force profiles between silica surfaces in the absence and presence of tripolyphosphate. The DLVO force curves show a force barrier at certain distance and then become attractive at shorter distance both for pure water and TPP solution. DLVO theory predicts that the interactions between ultrafine silica particles may have a same tendency in deionized water and in TPP solution, which can not explain the dispersion of silica suspension by addition of TPP as seen in Fig. 2. According to the calculated results from EDLVO theory, the strong repulsion is always present between ultrafine silica particles at pH 6.5~6.7 in the absence and presence of TPP. The repulsive forces is much higher in TPP solution than in deionized water, indicating that tripolyphosphate can be used as the dispersant of ultrafine silica as seen in Fig. 2. In this case, the repulsive interaction is also due to the polar interfacial interactions, but it is hydration repulsive interaction.

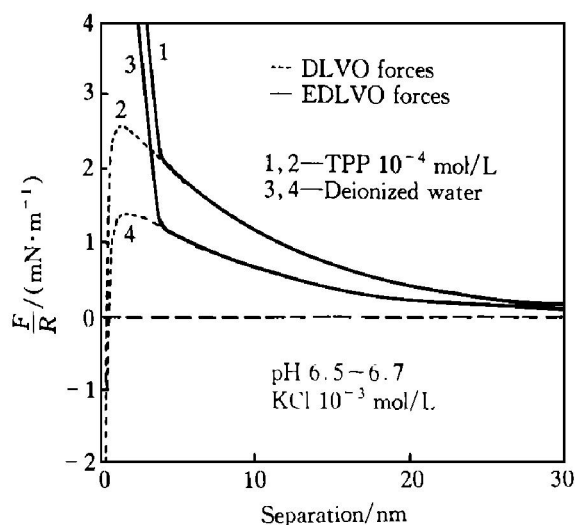


Fig. 5 DLVO and extended DLVO force profiles between silica surfaces in deionized water and in tripolyphosphate (TPP) solution

4 CONCLUSIONS

Silica surface is negatively charged at wide pH range having a PZC value 1.8. The negative zeta po-

tential of silica surface is decreased and the charge reversal occurs at pH 3.2 in 10^{-4} mol/L dodecylamine chloride, whereas in tripolyphosphate solution, silica surface becomes more negative. The particle size of ultrafine silica suspension is coarser in DDA solution than in pure water, showing the aggregation phenomena between silica particles. TPP acts as a dispersant for silica suspension. The average particle size of silica suspension is smaller in TPP solution than in pure water. Silica surface is rendered hydrophobic in DDA solution giving rise to strong attractive interfacial polar interactions (hydrophobic interaction), which is responsible for the aggregation of silica particles. Silica surface adsorbed TPP is more polarized and hydrophilic, inducing strong repulsive interfacial polar interactions (hydration repulsion), which accounts for the dispersion mechanism of TPP.

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(Edited by YUAN Sai-qian)