Study on physical properties of dispersed silica

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Abstract. The paper deals with the physical properties of colloidal silica dispersed system such as distribution width (Qₐ, %) and density (qₓ, %) defined by photon cross correlation spectroscopy (PCCS) at an average size of 17.06 nm for D₅₀ % and the area of specific surface of 349.96 m² cm⁻³ depending on molar (Rₑ₂) optimal ratio of silica stone mineral to alkali ash powder. The change of crystal structure to amorphous state was determined by X-ray diffraction analysis. The results of their kinematic viscosity, liquid density, UV light adsorption inspections show possibility to produce the colloidal silica by using the wetting and ultrasonic vibration method for that fusible alloy.

1. Introduction
Silicon dioxide (SiO₂) nanoparticles aqueous dispersion is used in a wide range of environmentally friendly products in a large variety of industries: biomedicine, food and beverages field. Within this study dispersed silica particles are produced by the direct fusion of precisely determined ratios of soda ash (Na₂CO₃) and natural silica stone in electric muffle furnace at approximately 1100 °C and then it is not well dispersed typically after wetting [1–3].

But for most silica applications especially when being used in coatings to improve scratch resistance, the silica particles need to be smaller than 40 nm to fulfill this requirement and that particle agglomeration hinders each individual silica particle to interact with the surrounding media. So, the stability and agglomeration state of this aqueous dispersion nanofluids are the key parameters for their use [4–6].

Experimentally ultrasonic processing has been used to get more effective colloid size, stable and non-agglomerated dispersion system. The dispersed silica colloid particles were measured by using photon cross correlation spectrometer. The minimal particle size was reduced to 17.06 nanometers at D₅₀ % of the test. The area of specific surface (Sₓ) for Rₑ₂ was obtained 349.9 m² cm⁻³ at molar optimal ratio of 2 for silica to sodium oxide weight.
2. Experimental procedure

2.1. Material and method

Dispersed silica nanoparticles were prepared by melting process of mixes by certain ratio of grinded powder of local silica stone from Tuv province, Sereglen soum located at 47°65’03.72”N, 107°25’22.47”E area with the utilization license N.21101 and sodium oxide as from sodium ash, CAS 497-19-8, Sigma Aldrich, Na2CO3. The physical and chemical properties were designed according to molecule ratio of acid to alkali as silica to sodium oxide weight which is shown in tables 1 and 2.

| Sample no. | SiO2 | Na2CO3 | Na2O | Rm=SiO2/Na2O |
|------------|------|--------|------|--------------|
| 1          | 10.1 | 8.1    | 3.4  | 3.0          |
| 2          | 10.1 | 12.2   | 5.1  | 2.0          |
| 3          | 6.0  | 14.5   | 6.0  | 1.0          |

Dispersed silica sols as yield content, density and formula for nanofluids etc. were prepared under their molar ratio (Rm) of the weight as shown in table 2.

| Rm | Yield (%) | Density (kg l⁻¹) | Formula |
|----|-----------|------------------|---------|
| 3.0–3.2 | 25–40 | 1.26–1.45 | Na2O · xSiO2 + H2O (3.0 < x < 3.2) |
| 2.0–2.6 | 35–46 | 1.38–1.53 | Na2O · xSiO2 + H2O (2.0 < x < 2.6) |
| 1.0–1.6 | 35–55 | 1.38–1.70 | Na2O · xSiO2 + H2O (1.0 < x < 1.6) |

Chemical composition of the silica stone used in the experiment was shown in the table 3 by comparing the test results of certified reference material (CRM.N40 testing) and its certified data of CRM.N40 according to the certificate. Mineral composition of the silica stone was studied by powder X-Ray Diffraction Analysis (XRDA) with the Siemens Diffractometer D5000 equipment, the results described in figure 1.

| Sample | SiO2 | Al2O3 | TiO2 | Feox | CaO | MgO | Na2O | K2O | MnO | P2O5 | LOI |
|--------|------|-------|------|------|-----|-----|------|-----|-----|------|-----|
| Silicaste | 98.56 | 0.15  | <0.005 | 1.13 | <0.01 | <0.01 | <0.01 | 0.03 | 0.012 | <0.005 | 0.28 |
| CRM.N40 test | 87.98 | 6.15  | 0.057 | 0.27 | 0.08 | 0.16 | 0.04 | 4.51 | 0.008 | 0.076 | 0.57 |
| CRM.N40 cert | 88.20 | 6.18  | 0.058 | 0.261 | 0.11 | 0.15 | –     | 4.23 | –    | 0.077 | –   |

The dispersed colloidal silica was prepared in the following steps [7]: mixing initial components, fusing them to temperature influence as approximately 1000 °C and wetting by distilled water to transform it into liquid state. The cleaning & non-agglomeration was done using centrifugal and ultrasonic method [8] with 800D Centrifuge equipment at 2000-2500 rpm speed and SK – 120 DTH Ultrasonic with 900-watt capacity for 15 min each sample. Value of pH for synthesized sol was defined with the laboratory PH5011 pH-meter. Liquid density of colloidal silica was determined by the aerometric method described in GOST 18481-81 [9] and kinematic viscosity was done by viscos meter described in GOST 10028-81 [10]. Absorptivity of UV light for dispersed nanoparticles in distilled water was determined by spectroscopic method using Shimadzu, UV-Vis Recording Spectrophotometer, UV-2401PC. The size and the value of average suspended sol, and the width of particle size distributions etc. were defined by photon cross correlation spectroscopic (PCCS) method using Shimadzu, STMPA.
Nanophox as described in MNS:ISO 13321:2014 standard [11]. This equipment determines the particle size from few nanometers to value 10 mkm at the initial step of precipitation. Test results were calculated by using WINDOX 5 program.

![Figure 1. X-ray powder pattern of the silica stone.](image1)

3. Results and discussion
The chemical composition of silica stone was used in this study, as shown in table 3. X-ray diffraction analysis was performed for both untreated silica stone (figure 1) and the dispersed silica, either treated by different ratio for sodium ash at 1100 °C for 1 h (figure 2).

![Figure 2. X-ray pattern of colloidal silica sol with sodium ash.](image2)

Measurement of all these properties provides information about the colloidal state of nanofluids. Hence the most important variable was the solid content. As it is shown in figure 1, X-ray diffraction intensive peaks corresponding to quartz crystal of silica disappeared and decreased their reflection intensity in figure 2, because of that at x-ray pattern with sodium carbonate phase after drying this low melting alloy was dispersed from the surface of colloidal amorphous silica phase at a higher temperature by distilled water (see figure 2).

By X-ray diffraction analysis the crystal structure of quartz crystal disappeared at $2\theta = 31.8^\circ$, having confirmed its complete transition to amorphous state for this pyro chemically prepared colloidal silica.
particles. Behind it, pH value of the medium can be used as an important factor for the stability of system to control it and as a driving force in electric chemistry [12]. So, the value of pH and density (\(\rho\), g ml\(^{-1}\)), kinematic viscosity (\(\mu\)), absorbed spectroscopy investigations for UV light were measured on all synthesized colloidal silica samples and shown in table 4 and figure 3.

**Table 4.** Study on molar ratio dependence of pH, density, viscosity and UVabs for colloidal nanofluids.

| \(R_m\) | pH   | \(\rho\) (g ml\(^{-1}\)) | \(\mu\) (Pa s) | UVabs (nm) |
|--------|------|--------------------------|----------------|-----------|
| 1      | 13.53| 1.08                     | 226.62         | 303       |
| 2      | 12.94| 1.09                     | 307.85         | 361       |
| 3      | 12.3 | 1.035                    | 576.08         | 333       |

![Figure 3. Absorbed UV spectrum of colloidal silica sol.](image)

The band gap of electronic transition of vibrational fine structure for the absorptivity of UV-Vis spectra at the widest value of \(l = 361\) nm area was observed for \(R_m\) type soluble silica suspension. These results showed that dispersions can be used or diluted with suitable (compatible) solvents such as suspensions of nanoparticles in water. These dispersions of nanoparticles can sometimes settle while storage, in this case they can be mixed (shaken up) before use and that colloidal silica is used as a raw material for water glass production optimal at molar ratio \(R_m\) for silica to alkali metal oxide. In addition, this dispersed phase could not dissolve in the dispersion medium of water with negative charges observed while their aging time and dispersion was controlled with the photon cross correlation spectroscopy to achieve ultrasonic treatment. Table 5 presents in detail the experimental results of the particles with the size of the dispersed silica sol depending on its distribution width (\(Q_s\), %) and density of colloidal suspension (\(q_x\), %), and coefficient of regression \((r^2)\) for linear equation for all samples, this also shown in figure 4.

The mass median diameter as \(D_{50}\) for \(R_m1 = 43.9\) nm, \(R_m2 = 17.1\) nm, \(R_m3 = 44.8\) nm was shown in figure 4 and in table 5. Concerning the influence of the solid content, pH and salt concentration results declared that minimal average size of colloidal silica from the area of specific surface was 349.96 m\(^2\) cm\(^{-3}\) at \(R_m2\). So, to produce colloidal SiO\(_2\) particles the molar optimal ratio was concluded to be 2 when using sodium carbonate to process the natural silica stone minerals.
4. Conclusion

Colloidal silica was produced by pyro chemical of silica stone, being an abundant mineral in Mongolia with molar different ratios of sodium ash at 1100 °C. The synthesis parameters, such as molar ratio of mixing, fluid density and size of colloid particles were optimized.

X-ray powder diffraction of colloidal silica disappeared at the crystal peak of quartz peak at 2θ = 31.8° and UV-vis. Spectrum. The band gap vibration of electronic transition was observed at the widest value of l = 361 nm area for \( R_{m2} \) type soluble silica suspension.

Colloid silica nanoparticles were confirmed at the optimized parameters of \( R_{m2} \) by PCCS inspection with the area of specific surface of up to 349.96 m² cm⁻³. The average size of the colloidal silica particles was 17.6 nm at D₅₀ % value, coefficient of regression for linear equation being 0.97 and with 1.04 slope.

These findings seem to be extremely important for the industry to produce colloidal SiO₂ particles by using silica stone and sodium ash minerals in practice in areas given.

Table 5. Average size (\( X_{nm} \), %), density (\( q_x \)), area (\( Q_x \), %) and regression (\( r_2 \)) coefficient of particle size distribution width for nano dispersed colloidal silica.

| \( R_m \) | \( D_{nm} \), % | \( SV \) (m² cm⁻³) | Slope \( \sigma_x = x84 \times 50^{-1} \) | \( \alpha = \sigma_x \times 50^{-1} \) | \( r_2 \) | SMD (nm) | VMD (nm) |
|---|---|---|---|---|---|---|---|
| 1 | 37.8 | 39.2 | 43.9 | 49.2 | 50.9 | 56.5 | 137.58 | 1.12 | 0.03 | 0.98 | 43.61 | 44.15 |
| 2 | 16.5 | 16.6 | 17.1 | 17.7 | 18.1 | 18.8 | 349.96 | 1.04 | 0.06 | 0.97 | 17.14 | 17.16 |
| 3 | 37.6 | 39.2 | 44.8 | 53.3 | 53.4 | 60.9 | 135.05 | 1.19 | 0.03 | 0.96 | 44.43 | 45.22 |

Figure 4. Colloidal silica particles distribution statistical curve by cumulative percent.
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