Investigating the Effect of Temperature on the Formation of Silica Nanocolloids Synthesized by Ion Exchange Method

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INTRODUCTION

Silica colloids consist of amorphous SiO_2 fine particles and usually spherical silica dispersed in a liquid phase. The formation process of colloidal silica was introduced in 19th century [1]. Researchers have used several methods, including ion exchange [2], electrodialysis of aqueous silicates, condensation of silane [3], peptization of silica gel [4] and direct oxidation of silicon to produce silica colloids [5]. Among these methods, the ion exchange method has been selected due to its ability to be industrialized and produced on a large scale and simple equipment. Also among the precursors used for silica nano colloids including silicon tetrachloride [6], tetraethoxysilane (TEOS) [7], sodium silicate [1] and silica powder [8], sodium silicate was selected due to its low price and convenient availability, which also has the ability to produce silica colloids by ion exchange method.

OBJECTIVES

In the current research, silica colloids were produced by ion exchange method using sodium silicate as the precursor. Afterwards, the effect of temperature on the particle size and the stability of the prepared silica colloids was investigated. The samples were characterized by Inductively Coupled Plasma Optical Emission spectroscopy (ICP-OES), Zeta potential, Fourier-transform infrared spectroscopy (FTIR), Energy-Dispersive Xray Spectroscopy (EDX), Scanning Electron Microscopy (SEM) and Dynamic Light Scattering analysis (DLS).

Table 1: ICP-OES test results on sodium silicate and silicic acid

Materials	Sodium ions (mg/Kg)
Sodium silicate	9580
Silicic acid	74

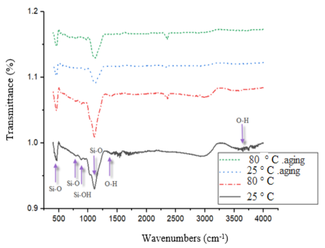

Figure 1: FTIR spectra of silica colloid samples at 25°C and 80°C before and after aging

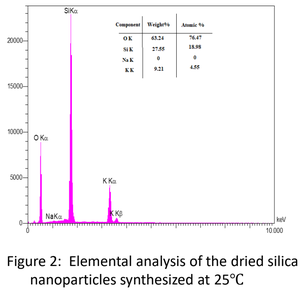

Figure 2: Elemental analysis of the dried silica nanoparticles synthesized at 25°C

MATERIALS & METHODS

Sodium silicate containing 7.5–8.5 %Na_2O and 25.5–28.5 %SiO_2 was used to prepare silica nanocolloids. 13 grams of sodium silicate was diluted with 40 ml of deionized water. Then, active silicic acid was produced by passing dilute sodium silicate through ion exchange resin. The sodium ions of the diluted sodium silicate solution were substituted by hydrogen ions on the exchange sites of cationic resin. 1 gram of potassium hydroxide (KOH) was added to 10 ml deionized water and the mixture was heated at different temperatures of 25°C and 80°C. Afterwards, silicic acid was titrated to the prepared KOH solution at a constant rate of 0.5 ml/min. The stability of silica colloids is very important. Accelerated aging test can be done by lowering the solution pH and increasing the store temperature. By mixing with the cation exchange resin, potassium ions were removed from the products and the pH value was reduced from 10-11 to 2-3 and then separated. The acidic slurry is placed in the oven at 65 ° C for 7 days. The solution is stable, if it don't turns to a gel state during the accelerated ageing test, otherwise, it shows an unstable behaviour. Moreover, the synthesized silica nanocolloids were characterized by Inductively Coupled Plasma Optical Emission spectroscopy (ICP-OES model ELAN DRC-2), Zeta potential (model SZ-100), Fourier-transform infrared spectroscopy (FTIR model Bruker Tensor II), Energy-Dispersive Xray Spectroscopy (EDX model TESCAN-Vega3JK), Scanning Electron Microscopy (SEM model TESCAN-Vega3JK) Dynamic Light Scattering analysis (DLS model SZ-100). In order to prepare the sample for SEM test, the silica colloid formed at 25 ° C was diluted with water and completely dispersed by ultrasonic device. Then some of the resulting solution was poured on the slide and dried at room temperature.

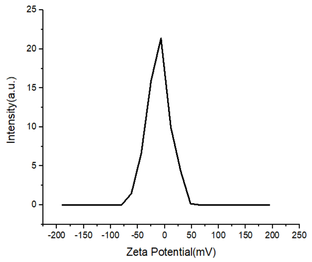

Figure 3: Zeta potential of the silica colloid synthesized at 25 °C

Table 2: DLS result of silica colloidal samples

	Mean (nm)	Mode (nm)	Average (nm)
Silica colloidal sample at 25 °C	6.9	5.3	16.7
Silica colloidal sample at 80 °C	13.6	12.3	13.5

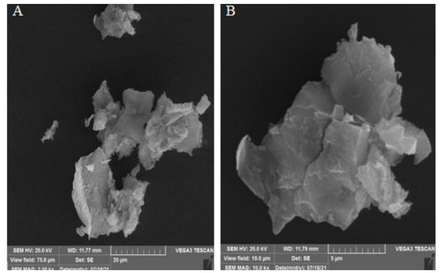

Figure 4: The SEM image of the silica colloid synthesized at 25 °C (A and B are at different magnifications)

RESULTS

ICP-OES test was performed on collected silicic acid from ion exchange column and sodium silicate as the raw material before ion exchange. The amount of residual sodium ions in the prepared silicic acid decreased from 9580 to 74mg/Kg compared to the primary sodium silicate, which confirms the formation of silicic acid (Table 1). As shown in Figures 1 and 2, the FTIR and EDX results confirmed the presence of silica in the form of silicon dioxide. Also, sodium ions are completely removed during ion exchange process. Figure 3 shows the changes in the zeta potential intensity of the synthesized silica nanocolloid at 25 °C. The same charge between the particles prevents the accumulation and maintains the Brownian motion. Colloidal stability is achieved at zeta potential values above +10 (positive charge) and -10 (negative charge) [9]. As shown in Figure 3, the absolute value of the zeta potential above -10 (Mean of zeta Potential is -6.8 mV) indicates that the resulting silica colloidal is stable. The particle size of the silica nanocolloids were measured by DLS analysis. The result (Table 2) shows that the mean particle sizes of the nanocolloids prepared at 25° C and 80 °C are about 6.9 and 13.6 nm, respectively, as tabulated in Table 2. As the temperature increases, the mean particle size rises and larger particles are obtained, this is accordance to the previous researches [2]. It should be noted that by increasing the size of the particles, silica nanocolloid tends to sediment or to turn into a gel state and is not stable anymore. Figure 4 shows the microscopic images of the dried silica nanoparticles synthesized at 25℃. It can be seen that the morphology of sample depicts mostly irregular shape with high agglomerations due to their nanoscale dimension. It is inferred that this particle size was influenced by the existence of many agglomerations of particles on the sample which was detected by SEM. Also, the silica colloid formed at 80 °C turned into a gel after one month, while the silica colloid formed at room temperature was stable.

CONCLUSIONS

Silica nanocolloids have been synthesized by ion exchange method using sodium silicate as raw material. The FTIR and EDX results confirmed the presence of silica in the form of silicon dioxide. The absolute value of zeta potential of sample at 25 °C was -6.8 mV which indicated that the prepared solution was stable. Particle size analysis showed that the mean particle size of the silica colloids at 25 °C and 80°C were about 6.9 and 13.6 nm, respectively. The morphology of sample depicted mostly irregular shape with high agglomerations due to their nanoscale dimension. As the silica colloid formed at 80 °C turned into a gel after one month, while the silica colloid formed at room temperature was stable. As a result, it can be shown that the temperature parameter is one of the most important parameters affecting the formation of stable silica colloids.

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