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Research Article

Colloidal Silica Production with Resin from Sodium Silicate and Optimization of Process

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Abstract - Colloidal silica is a stable and homogeneously dispersed form of amorphous silicon dioxide (SiO_2) nanoparticles in water. Colloidal silica has been the focus of research due to large surface area, biocompatibility, low toxicity and chemical and thermal stability. It has been used in a wide variety of industrial applications, including pulp and paper, chromatography, electronics, foods, and colloids, as well as in the ceramics and glass industry. In this study, colloidal silica was produced using cationic resin and sodium silicate and process conditions were optimized. Temperature (50-80 °C), mixing speed (200-500 rpm) and time (20-120 min.), which significantly affect the particle size, were selected as parameters. Particle size distribution (PSD) analyzes of colloidal silica particles were performed to determine appropriate levels of the parameters. The most suitable process conditions are 50°C temperature, 40 min. and 300 rpm. The average particle size of colloidal silica produced in optimum conditions was measured as 80 nm.

Keywords: Colloidal Silica, Optimization, Sodium Silicate, Particle Size

1. Introduction

Silica and silica-derived materials are widely used in the glass, ceramics and silicon industries due to their availability and easy recovery. Colloidal silica, generally known as silica sol used as a term corresponding to a stable distribution of amorphous silica particles. Silica sols first synthesized in 1861 by Graham who investigated the production of dilute silica sol by dialysis by adding hydrochloric acid to an aqueous solution of sodium silicate. Silica sol including 10% SiO_2 was marketed for the first time in 1933 [1]. Colloidal silica is the form of amorphous silicon dioxide nanoparticles in the range of 1-100 nm, distributed in a stable and homogeneous manner in water. Sodium silicates, unlike colloidal silica, consist of silicate monomers. Sodium silicates have a SiO_2/Na_2O ratio of approximately 2.0-3.4. The SiO_2/Na_2O ratio of colloidal silica is greater than 50. Sodium silicate solutions having 3 modules are commonly used in colloidal silica production. Reactions used in the chemical preparation of colloidal silica can be divided into three main groups: reduction-oxidation reaction, hydrolysis of salts and ion displacement reactions [2].

Colloidal silica particles have many advantageous properties such as almost spherical shaped particles and narrow size distribution. However, it is one of the least understood systems due to the complexity of the physical and chemical nature of the surface. Although, there are Si–O–H and Si−O−Si functional groups on the colloidal silica surface, the distribution and amount of groups have a significant effect on the surface charge and structure [3]. The interest in research on colloidal silica, an inorganic and nanoparticulate structured material, stems from its numerous advantages, including its large surface area, low toxicity, apparent biocompatibility, optical transparency, chemical, thermal and colloidal stability [4]. Colloidal silica has been used for many years in a wide variety of industrial applications such as chromatography, healthcare, paper and pulp, electronics, foods, gels and colloids, as well as in the ceramics and glass industry [3-5]. Its current usage areas have expanded and it can be used as a coating material for various core materials, as drug carriers, as a support material for

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biosensors and catalysts, and as an additive for paints-varnishes-coatings with the surface modification of colloidal silica [6-7].

Colloidal silica is widely used in the casting industry in the production of ceramic molds, in the construction industry in concrete production, in thermal insulation, in the paper industry to ensure the non-slip properties of paper, in the paint industry to increase the scratch resistance of the paint applied to the surface, and in the beverage industry as a clarifying agent to ensure clarity. SiO_2 content of colloidal silica used in the industry changes between 15-50% and the particle size varies between 5-60 nm. When the colloidal silica use increased in novel application areas, products with different particle size distributions are needed in those areas [8]. The main producers of colloidal silica in the world market are in countries, including the United States, Germany, Japan and China. Considering the production data of 2012, commercial silica production has a market value of 3.6 billion US dollars with a production of 2.4 million tons [1].

To produce colloidal silica different methods have been suggested such as sol-gel method, hydrothermal reaction and chemical vapor deposition (CVD) [9]. However, colloidal silica can be synthesized by hydrolysis and condensation of Tetraethyl orthosilicate (TEOS) or Tetramethyl orthosilicate (TMOS) known as polymeric sol-gel method, peptization silica gel or powder, oxidation of silicon powder and electrodialysis of hydrous silicates [7-10]. The sol–gel method having the advantage of controlling particle size and morphology by systematic control of reaction parameters at ambient temperature is widely used. Colloidal silica can be produced from sodium silicate solution by ion exchange method without using expensive starting materials and emitting toxic wastes [11-9]. The ion-exchange method has the advantages of being the most used process and being able to control the particle size and distribution. However, the regeneration of ion exchange resins is seen as a significant problem in financial and environmental terms. In addition, any Na ions remaining in the product may reduce the purity and stability of the colloid [7].

The ion exchange method is a process in which sodium ion is removed from liquid sodium silicate solution by using an ion exchange resin. Liquid sodium silicate is used as starting material by diluting to 2-6 %wt SiO_2 content. Active silicic acid is produced by removing sodium ions from diluted sodium silicate solution by passing it through cation exchange resin column [10]. Colloidal silica is produced industrially from sodium silicate solution [4]. Sodium ion is removed from diluted sodium silicate by passing it through ion-exchange cationic resin and active silicic acid is produced [6]. The ion exchange reaction between sodium ions and hydrogen ions of the cation exchange resin takes place as shown in Eqn.-1 [5].

$$Na^+ + RzSO_3^-H^+ \rightarrow H^+ + (RzSO_3)Na \qquad \textbf{(Equation-1)}$$

In equation 1, SiO_3H is the sulfonic acid group and Rz is the styrene-divinyl benzene copolymer. Since the ionic bond strength of sodium ion is stronger than that of a hydrogen ion, it is easily replaced by sodium ions in water glass solution [11]. The silica sol then forms a dilute silica sol via nucleation from active silisic acid. It includes the stages of growing acid and discrete silica particles by polymerization and concentrating the dilute silica sol [1-10]. Different raw materials can be used in the production of colloidal silica. Examples of these materials include silicon metals, ethyl silicate, silicon tetrachloride, silica powder and sodium silicate [1]. Sodium silicate is generally preferred as a starting material in the industry due to its lower cost compared to other silicates available on the market [8]. Also, colloidal silica produced by the ion exchange method have homogeneous particles, higher silica content, and lower residual sodium ions [11]. In general, colloidal silica is stable in the pH range of 10-11, and the gelation time is short in the pH range of 3-8. Therefore, before using the colloidal silica in some new applications, its surface must be modified, especially if it is going to be used in the acidic range [12]. Although extensive research has been carried out on the production of colloidal silica, studies are still being carried out to determine the stability of the silica sols produced. Tsai, 2004 [13] produced colloidal silica by using a continuous process column via sodium silicate. Differently, in our study, colloidal silica was produced in a batch process by using the separated part of the starting material as appropriate to industrial production, diluted sodium silicate, instead of KOH, without the need for the use of additional materials.

In this study, colloidal silica was produced from sodium silicate solution using ion exchange method. Before running the experiments, the amount of ion exchange resin was determined by measuring pH of the solution. Temperature (50-80 °C), mixing speed (200-500 rpm) and time (20-120 min.), which affect significantly particle size, were selected as parameters and they were optimized. PSD analyses of colloidal silica particles were performed to determine appropriate levels of the parameters. Formed products were characterized by using XRD and FTIR analyses.

2. Materials and Methods

2.1. Materials

In this study, sodium silicate ($Na_2O.3SiO_2$ having Na_2O, 8%; SiO_2, 27%, Özkimsan) and Purolite C160H cation exchange resin were used as received without any treatment. The regeneration process was carried out using HCl (37%, Merck). Deionized water was used for all experiments.

2.2. Methods

The sodium silicate solution was diluted with deionized water to obtain a solution having 3% SiO_2. The prepared dilute sodium silicate solution was divided into two parts by volume in a ratio of 1:7. The excess volume was treated with ion exchange resin to obtain active silicic acid. pH of the solution was measured at certain periods during the ion exchange process to determine the optimum amount of resin. The 40 g of resin was used in all experiments after determining the minimum resin required that lowers the pH to the range of 2-3. The mixture was filtered under vacuum of 700 mmHg to separate the resin from the solution. pH was measured at regular intervals by adding active silicic acid solution to the slightly dilute sodium silicate solution allocated at the beginning. Thus, because of the nucleation and growth of silica particles, colloidal silica formation was achieved in the stable pH range (pH=10-11). To concentrate the colloidal silica, mixture was evaporated by heating for a certain period of 20-120 min., at different temperatures of 50-80 °C and at stirring rate of 200-500 rpm. The used Purolite C160H cation exchange resin was regenerated by using 2 M HCl to exchange Na^+ ions to H^+ ions and reuse of the resin in the ion-exchange process [11]. A block diagram used for colloidal silica production was shown in Figure 1. Particle size analyses of colloidal silica samples prepared under above parameters were performed. The most suitable production conditions of colloidal silica nanoparticles were determined.

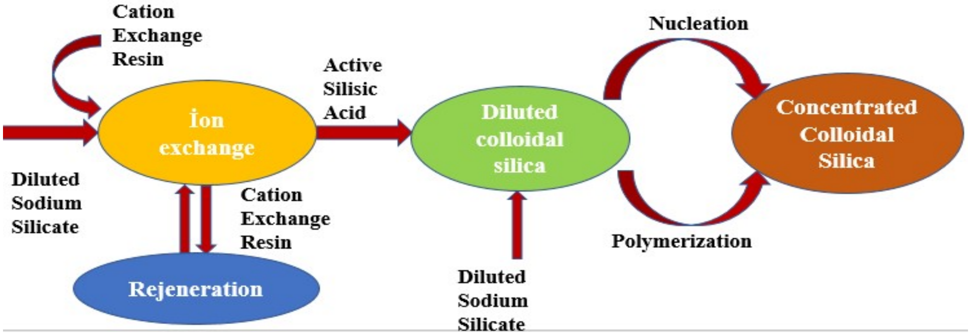

Figure 1. Block diagram of colloidal silica production process.

2.3. Characterization

Investigation of the amorphous structure of colloidal silica samples was investigated by using X-ray diffraction diffractometer (XRD) (BRUKER D8 ADVANCE TWIN-TWIN). Fourier transform infrared (FTIR) spectrum of silica nanoparticles were obtained by using FTIR spectrometer (FT/IR-4700 typeA). Solid silica particles after the drying at 105ºC was examined in FTIR spectroscopy and XRD. In the study, the particle size distribution (PSD) analysis was performed by dynamic laser light scattering technique (Nano ZS, Malvern Co. UK). Before PSD analyzes samples were used by shaking the briefly in a tube shaker. Three readings were made for each sample and the average results were recorded.

3. Results and Discussions

Infrared spectroscopy is a characterization method used to analyse organic or inorganic compounds qualitatively and quantitatively. It is used to determine the chemical bonds in the molecule with the absorption peaks that occur because of the vibration of the bonds between the atoms that make up the substance [14].

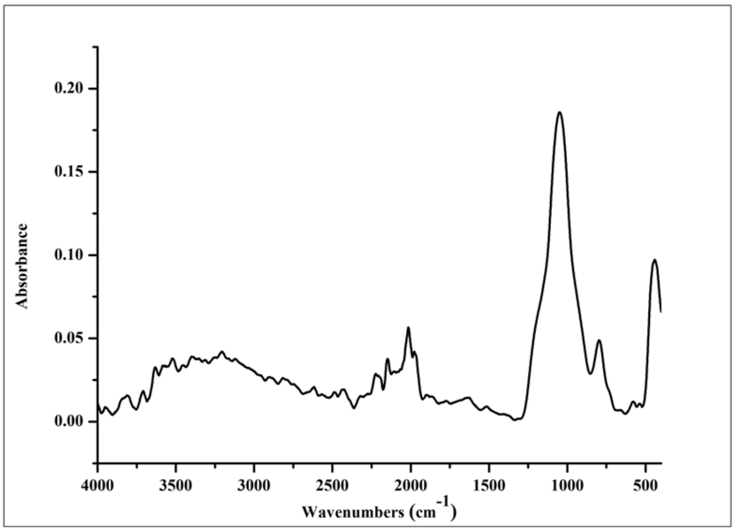

Figure 2. FTIR spectrum of colloidal silica.

The FTIR spectrum of the produced colloidal silica is given in Figure 2. FTIR spectrum of colloidal particles consists of asymmetric vibrations of Si–O 1090 cm^{-1} and Si-O-Si 1080 cm^{-1} [15], peaks arising from the asymmetric vibration of Si–OH (950 cm^{-1}) [16]. These peaks are overlapping in the spectrum shown in Fig. 2. In the absorption band, the peak of the symmetric vibration of Si–O is seen at 795 cm^{-1}. Absorption bands between 850-1260 cm^{-1} are defined as the overlap of various SiO_2 peaks and peaks arising from Si-OH bonds [17]. An intense characteristic absorption band was observed between 3000-3800 cm^{-1}, which is assigned to the hydrogen bonds and O–H stretching of water. Additionally, this band is supported by the shear bending vibration of molecular water seen around 1635 cm^{-1} [17].

XRD is the most suitable characterization method to study the crystal structure of mesoporous materials and colloidal materials. The sample to be analysed is usually in the form of fine-grained powders. XRD analysis of crystalline compounds shows a diffraction pattern consisting of a narrow and prominent peak. Amorphous materials, on the other hand, do not form clear peaks. Instead, it produces spotted peaks or short rows of bumps in the diffraction pattern [1].

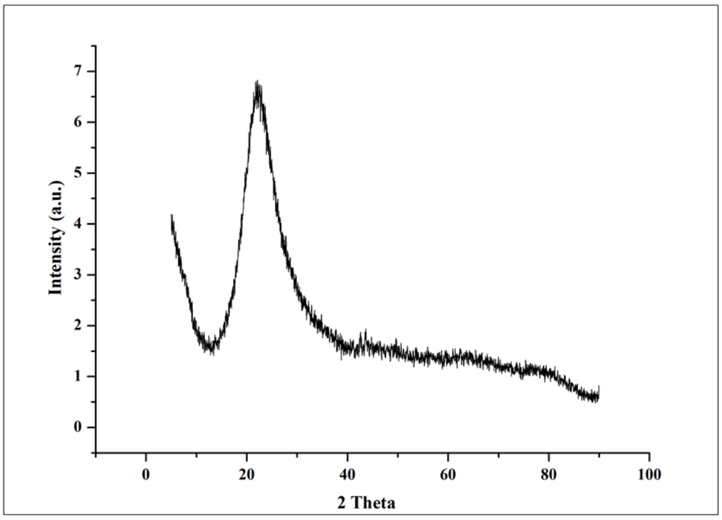

Figure 3. XRD spectrum of solid product obtained from colloidal silica.

As depicted in Figure 3 shows a broad peak observed between 15-30° at the 2θ angle confirms the amorphous nature of colloidal silica [16-18]. An amorphous peak was seen at $2\theta = 21.8^\circ$ [16]. They reported that the amorphous peak of colloidal silica was observed at $2\theta = 21.8^\circ$ [19], and in their study where they produced amorphous silica with the sol-gel procedure, they stated that the amorphous peak was recorded at $2\theta = 23^\circ$ at the end of the heat treatment.

The pH change versus resin amount during the formation of active silicic acid during the ion exchange process is given in Figure 4. The amount of resin to be used is determined as 40 g from the chart.

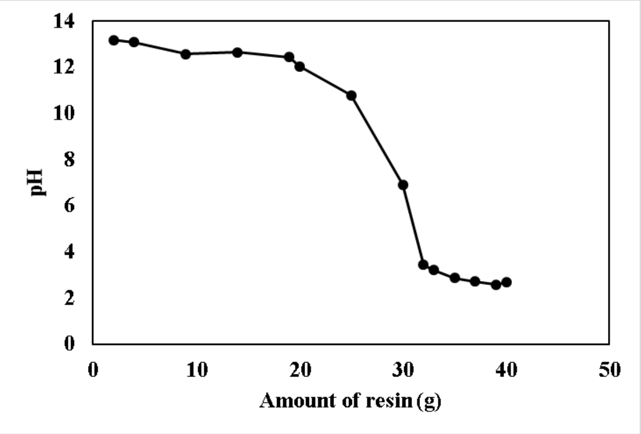

Figure 4. pH change for different amounts of resin during the formation of active silicic acid.

By adding active silicic acid solution onto dilute sodium silicate, colloidal silica formation was achieved in the stable pH range (pH = 10-11) [19] as a result of the nucleation and growth of silica particles. Generally, colloidal silica is stable in the pH range of 10-11. Colloidal silica, which is unstable in the pH range of 3-8, has a short gelation time [6]. To concentrate the colloidal silica, heating was carried out for a certain period (20-120 min.) at different temperatures (50-80 °C) with stirring of (200-500 rpm). Experiments were carried out at 4 different levels with time, mixing speed and temperature parameters using sodium silicate solution and cationic resin. Average particle size distribution analyses of the produced colloidal silica samples were performed and the change in particle size distribution of colloidal silica in response to heating time is given in Figure 5.

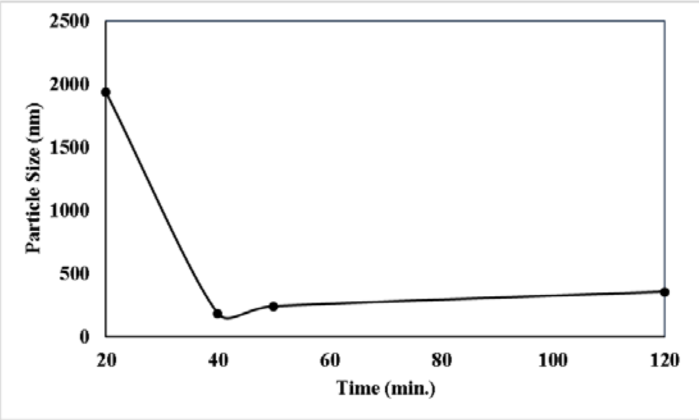

Figure 5. Particle size distribution of colloidal silica produced in different times.

As seen in Figure 5, the particle size corresponding to 20 minutes of heating time has the highest value. As a result of the sudden growth of colloidal silica produced over time, unstable particles larger than the desired particle size were formed. Minimum particle size was determined at 40 minutes as 184 nm for the colloidal silica obtained. After the heating period, the increase in time caused the particle size to increase. In line with these data, it was observed that the value closest to the desired particle size was obtained at 40 minutes of heating.

The temperature range to be used for colloidal silica production was chosen as 50-80 °C. It has been stated that when temperatures exceed 80 °C, deterioration occurs in the amorphous structure of SiOHs in colloidal silica. It has been determined that colloidal silica produced below 50 °C gels because it is not in a stable structure. Figure 6 shows the change in particle size distribution of the produced colloidal silica with temperature.

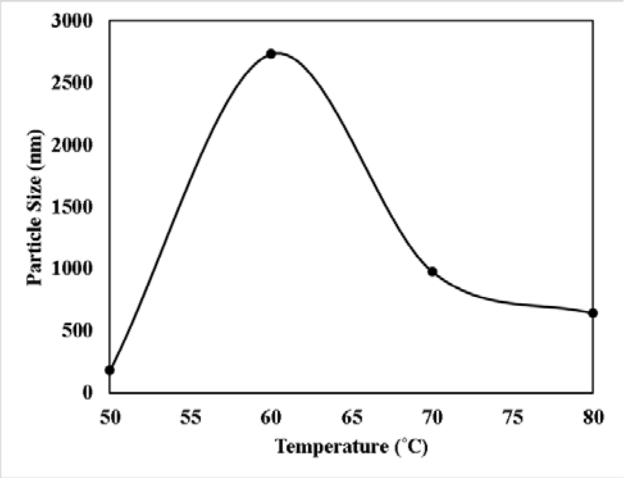

Figure 6. Particle size distribution of colloidal silica produced at different temperatures.

It has been determined that as the temperature increases, the particle size of the produced colloidal silica increases and moves away from the reported particle size range [20]. As it was stated that the addition of alkaline (pH = 8-10.5) at moderate temperature, colloidal silica was produced at a temperature of at least 60 ˚C to ensure nucleation, polymerization and particle growth [21]. Thus, a stable dilute colloidal silica solution is formed. In this study, concentrated colloidal silica was produced by investigating the duration of the heat treatment process. A temperature of 60 ˚C increased polymerization and particle growth occurred with the rising temperature. The reason for the decrease in particle size above 70 ˚C may be that hydrolysis and condensation proceed much faster, leading to the formation of secondary particles. The temperature dependence of particle growth is related to changes in hydrolysis and nucleation rate, which decrease significantly with lowering the reaction temperature. Higher temperatures in alkaline regions increase Ostwald ripening of colloidal silica and coarsening of particles. Thus, smaller particles accumulate and combine on larger particles to form larger aggregates [22].

Table 1. Experimental data for optimization of colloidal silica process conditions.

Experiment	Concentration % wt.	Temperature (°C)	Heat. Time (min)	Mixing Speed (rpm)	Av. Part. Size (d.nm)	pH
1	3	50	50	200	242	10.5
2	3	50	120	200	357	10.6
3	3	50	20	200	1935	10.6
4	3	50	40	500	1081	10.4
5	3	50	20	400	460	9.8
6	3	50	20	300	205	9.9
7	3	50	40	200	184	10.4
8	3	50	40	300	80	10.4
9	3	50	40	400	7027	10.7
10	3	80	40	200	642	9.7
11	3	70	40	200	981	9.5
12	3	60	40	200	2735	10.3

When the average particle sizes of colloidal silica samples are compared in Table 1, the most suitable process conditions for the sample having the lowest particle size are 50°C temperature, 40 minutes of heating and 300 rpm stirring rate. Particle size analysis of colloidal silica produced under these conditions is given in Figure 7-a and Figure 7-b, based on volume and intensity, respectively. Analysis of particle size distribution and average particle size were performed. The smallest average particle size was obtained in experiment 8. The highest one was occured in experiment 9. This result shows that lower mixing speed leads to a lower average particle size [7].

Particle size analysis results of 3% colloidal silica samples prepared at four different mixing speeds at 50 °C are included. According to these results, it was determined that colloidal silica samples prepared at mixing speed of 400-500 rpm had the highest particle size. Particle size analysis of the colloidal silica sample prepared at a stirring speed of 300 rpm is within the colloidal silica particle size range previously stated [1].

Colloidal silica is obtained by stably dispersing solid silica particles in water in the particle size range of 4-100 nm [4]. When the average particle sizes of colloidal silica samples are compared, the most suitable process conditions for the sample with the lowest particle size are 50°C temperature, 40 min. heating time and 300 rpm stirring rate.

While the volumetric distribution results are proportional to the 3rd power of the particle diameter (D^3), the size distribution displayed based on intensity is proportional to the 6th power of the diameter (D^6), so the relatively large particles in the suspension can be seen more clearly in the intensity-based distribution. When the size distribution by volume given in Figure 7a is examined, the average particle size of colloidal silica produced under the most suitable process conditions was determined as 80 nm and is within the particle size range used in the industrial field (4-100 nm) [4]. It is understood from the analysis based on Intensity in Figure 7b that there are three different particle size groups in the colloidal silica sample. It was evaluated that some of silica particles formed agglomerated and the average particle size increased.

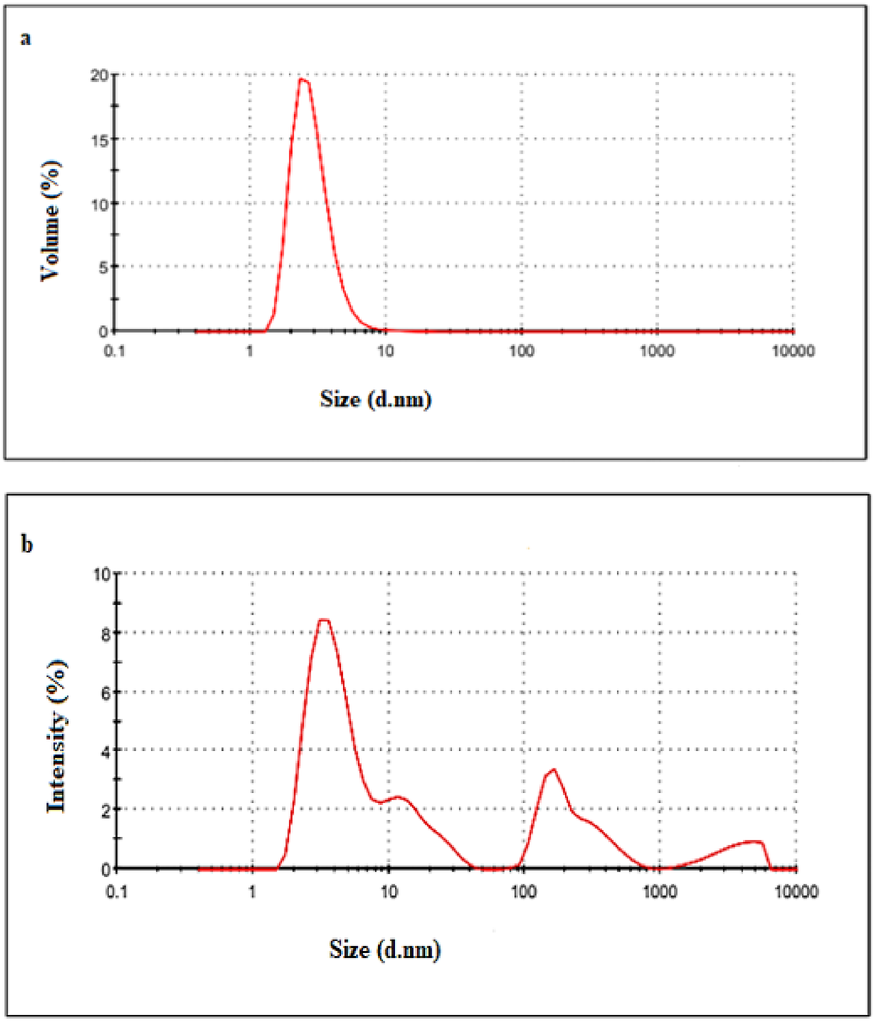

Figure 7. Particle size analysis of colloidal silica produced under optimum conditions.

4. Conclusions

Colloidal silica was produced from sodium silicate solution using ion exchange resin. Appropriate process conditions were determined by analyzing the particle size distribution of the produced colloidal silica. According to the particle size distribution range, the most suitable colloidal silica production parameters are 50°C temperature, 40 minutes time and 300 rpm stirring. The average particle size of colloidal silica produced at the mixing speed of 300 rpm was found to be 80 nm. The chemical bond structure and amorphous structure of colloidal silica were confirmed by FTIR and XRD analyses, respectively.

The study is important as it provides the advantages of producing colloidal silica in the industrially used particle size range from sodium silicate as the starting material, regenerating the resin utilized and reusing it repeatedly.

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Author contributions: Concept B. A., J. A., R. T. & M. G.; Data Collection &/or Processing - B. A., J. A., R. T., İ. E. & M. G.; Literature Search - B. A., J. A., R. T. & M. G.; Writing - B. A., J. A., R. T. & M. G.

Conflict of Interest: This paper has been presented at the UKMK-15 (15th National Chemical Engineering Congresss) held in Çanakkale (Turkey), September 4-7, 2023.)

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