



## Original Research Paper

## An advanced fabrication route for alkali silicate glass by non-firing process

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## ABSTRACT

An amorphous silica powder surface was treated separately and combining mechanical and chemical treatment processes. The raw silica powder was mechanically modified using a planetary type mill at a rotation speed of 200 rpm for 15 min using a 5 mm size ball. The raw and the mechanically modified silica powders were hydrothermally treated for 5, 15 and 24 h lengths of time. Silanol surface groups successfully produced by the treatments were measured by diffuse reflectance infrared fourier transform (DRIFT) spectroscopy. The results show that the combination of mechanical and chemical, so call mechanochemical treatment, is the most efficient in enhancing the quantity of surface silanol groups. The mechanochemically treated powder was used for the fabrication of glass. Silica glasses were successfully fabricated without firing by mixing the treated powders with KOH 5 M solutions. Morphology and transparency of the obtained glasses were analyzed using SEM and UV–VIS–NIR techniques. Possible mechanism reactions of powder surface activation occurring during the mechanochemical and condensation processes are also discussed.

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## 1. Introduction

Silica (SiO<sub>2</sub>) is one of the most abundant minerals on the earth's surface and is an important technological material. It can have application in numerous areas especially in high-frequency devices, glass, etc. [1–3]. Firing ceramics is the most common technique in materials fabrication. However, this method has been disadvantageous in terms of its high cost and possible harmfulness to the environment. Recently, in order to reduce the cost and energy for post-forming processes, Elad-ua et al. [4] proposed a novel ceramic fabrication method without firing and/or debinding processes. In this method, the surface of a raw powder is mechanically activated using a planetary ball-mill. Deposition of different compounds on the surface of SiO based materials is usually achieved by adsorption, the adsorption efficiency increases proportionally to the quantity of surface active sites. The mechanical treatment of silica leads to the appearance not only of strongly distorted Si–O–Si fragments of the siloxane bonding, but also of ≡Si–, –SiO– and –Si· radicals. When the mechanical treatment proceeds, various active sites are produced [2]. The functionalization of the silica particle surface can be used to enhance and/or control the overall properties of the particles for

desired applications. The nature of the functional groups on the particle surface usually plays a critical role in the surface properties of particles such as hydrophobicity and chemical reactivity. Surface ≡Si–OH groups can form as a result of rehydroxylation of dehydroxylated silica when it is treated with water or aqueous solutions. The surface silicon atoms tend to have a complete tetrahedral configuration and in an aqueous medium their free valence becomes saturated with hydroxyl groups [5]. Intensive research has been carried out on the chemical modification of silica: organic molecules or radicals have been attached to the surface of silica. In these reactions the surface hydroxyl groups play an active role. The surface properties of silica depend on the presence of silanol groups. It is thus necessary to obtain both qualitative information on the chemical properties of the silica surface and quantitative data on the surface concentration of silanol groups. The reaction between the surface silanol groups (condensation) leads to the formation of siloxane bonds and molecular water (≡Si–OH) + (≡Si–OH) → (≡Si–O–Si≡) + H<sub>2</sub>O [5–8]. In the present work, mechanically treated amorphous silica powder was used for hydrothermal surface treatment in order to obtain high concentration of surface silanol groups. The treated silica powders were used for fabrication of glass without firing process. FTIR, SEM, UV–VIS–NIR spectrophotometer were used for characterization. Possible mechanism reactions of powder surface activation occurring during mechanochemical and condensation processes are also discussed.

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## 2. Experimental procedure

The amorphous silica powder (SFP-20MX, Denki Kagaku Kogyo Kabushiki Kaisha, Tokyo, Japan) used in the present investigation.

The raw silica powder was milled using a Germany type laboratory planetary mill for 15 min at a rotation speed of 200 rpm with a 5 mm size ball. These conditions were chosen based on the results of a previous work by the authors [9].

A 1:1 by weight ratio of powder and deionized water was used for the hydrothermal treatment process. The obtained slurries were placed in an autoclave and kept in a furnace at 130 °C for 5, 15 and 24 h. After these lengths of time the slurries were taken out and dried in vacuum drier for 7 h to evaporate physically adsorbed water.

DRIFT spectra were obtained using a FT-IR spectrophotometer (FT/IR 6200, JASCO Corp., Tokyo, Japan). This technique is very useful for studying the structure configuration of the silica surface before and after treatments. A 100% powder filled micro sample cup was used to obtain absorbance spectra in the 4000–400  $\text{cm}^{-1}$  region. The spectra were recorded under  $10^{-3}$  Pa of vacuum. Background grounded KBr powder spectra were obtained and used to normalize the sample spectra. All spectra were measured with a total of 512 scans and a resolution of 4  $\text{cm}^{-1}$ .

KOH 5 M (5 mol/l) solution was used for solidification of silica powder with 40% weight in a 60 mm length  $\times$  15 mm width mold. Before casting to the mold, the slurry was mixed in a mini electrical mixer for 5 min with a rotation speed of 2000 rpm. After pouring the slurry, the mold was covered and kept 24 h in an oven at 40 °C for solidification. The solidified body was de-molded and dried for 24 h at 80% RH and 60 °C. The thickness of the bodies was always 3.5 mm.

Glassy morphology observation was carried out using FE-SEM (JSM-7000F, JEOL Ltd., Tokyo, Japan). Samples were dried in vacuum dryer for 24 h before observation.

Transparency was observed using UV–VIS–NIR spectrophotometer (UV-3150, Shimadzu Corp., Kyoto, Japan) in the range of the visible light length 800–300 nm.

Experimental procedures can be summarized as shown below:

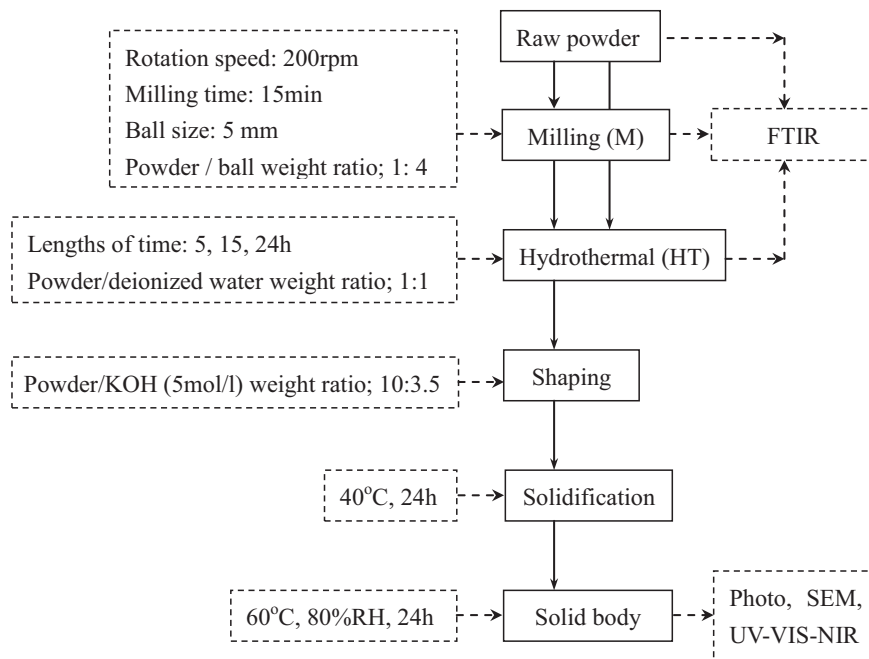
## 3. Results and discussions

In contrast to standard methods of surface chemistry, which usually measure average properties of collective chemical functional groups at interfaces, infrared techniques measure directly specific properties of individual types of functional groups. Three independent parameters characterize each infrared absorption band: frequency, intensity and width. Changes in these parameters with different conditions and in the presence of various adsorbed molecules offer numerous possibilities for studying the chemistry of surface functional groups [10]. On the other hand, absorption coefficients of surface groups are not known well enough for accurate estimates of absolute concentrations. Even though qualitative observations of intensity already provide information on changes in relative concentration of surface groups during treatments, band ratios within each spectrum can be used to obtain quantitative changes [11].

Fig. 1a shows the common scale DRIFT spectra of the raw and mechanically treated (M) silica powders in the SiOH (silanol) and SiH (silane) stretching band frequency regions 4000–2000  $\text{cm}^{-1}$ . The presence of OH stretching vibration is revealed by the broad band between 2800 and 3800  $\text{cm}^{-1}$  (dotted arrow). Three peaks assigned to related silanol groups are considered in this region. A sharp peak at 3750  $\text{cm}^{-1}$  is due to isolated SiOH groups. The highest band at 3670  $\text{cm}^{-1}$  is assigned to weakly hydrogen bonded SiOH groups and adsorbed water molecules. The shoulder peak at 3440  $\text{cm}^{-1}$  is due to water molecules hydrogen bonded to each other and to SiOH groups [10,12]. The band at 2250  $\text{cm}^{-1}$  is assigned to SiH bond vibrations [13]. This band at 2250  $\text{cm}^{-1}$  appears not to be affected by the DRIFT accessory and can be used as a reference for each individual spectrum. The amount of OH in raw or treated silica powder surfaces is then assessed in the form of an integrated OH/SiH band frequency absorbance ratio. The results are shown in (b). It is observed that OH surface groups could be produced by mechanical treatment and the amount of the groups increases 1.5 times compared to the as received silica powder.

The common scale DRIFT spectra of the raw and hydrothermally (HT) treated silica powders in the 4000–2000  $\text{cm}^{-1}$  range are shown in Fig. 2a and the OH/SiH absorbance ratios in (b). The

Experimental procedures can be summarized as shown below.



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