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Shape Control of Silica Powder Formation

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Abstract

The purpose of the present research is the different morphologies production of crystalline and amorphous-silica powder. It's a basic material for many pharmaceutical and environmental applications as well. And, it's produced using the combination of the alkali chemical etching process and the ultra-sonication technique. The critical preparation conditions are KOH concentration (weight %) and the sonication time (hour). The paper presents the chemical mechanism of the silica particle formation as well as the different morphology. The results show the formation of crystalline and amorphous-porous-silica particles in the micrometer range with the porous order network that has pore sizes range in micrometer too. This synthetic uses commercial silicon, which could be useful for large-scale production. Also, the nano-sphere and nano-cubic shapes of silica powder are formed starting by commercial silicon powder.

Keywords

Amorphous Materials, Porous Materials, Powder Formation, SiO_2

1. Introduction

The silica (SiO_2) powder is noted as competent materials because of their unique characteristics (low density, high surface area and high specific strength) [1] [2] [3]. SiO_2 is a very interesting material for using in many fields [4] [5]. And, it's proving a promising material due to its good thermal and mechanical stability. In addition, it has many excellent physical and chemical properties [6] [7].

There're various porous-SiO_2 preparation techniques in previous studies [8]; the synthesis of porous-SiO_2 thin films is described with respect to pore structures [9]. It's derived from the presence of active (OH-groups) on the particle surface. So, the (Si-O-Si) linkages between neighboring particles are produced as a result of the hydrogen-bond formation. In case of the (OH-groups) deactivation, the nanoparticles aggregation is stopped [7]. There are several chemical techniques used for controlling the particle shapes, and size of fine SiO_2 particles [1] [8]. In this work, it demonstrated a combination between wet alkali chemical etching (ACE) process and ultra-sonication technique that controls the SiO_2 shapes.

2. Materials & Methods

The combination of ACE process and ultra-sonication technique is attractive because of its simplicity. The preparation of porous-SiO_2 different morphologies is started by 7 g commercial polycrystalline Si-powder (99%, Sigma-Aldrich). It's added to the NPA (30 Vol %), KOH_{conc} (3, 4.5, and 6 weight %) and at different time values (2, 3, 4, and 5 hours). The product is filtered, washed, and then slowly dried at room temperature. The structure of the nano-porous-SiO_2 is characterized using X-ray diffraction (XRD; Cu Kα radiation of 1.5405 Å, Shimadzu 7000 diffractometer). The scanning electron microscope (SEM; JEOL-JSM-5300) and transmission electron microscope (TEM; JEOL, Japan), present the nano-porous-SiO_2 morphology. In addition, the formation of nano-porous-SiO_2 chemical bonds is determined by Fourier Transform Infrared spectroscopy (Shimadzu FTIR-8400s, Japan).

3. Results & Discussion

XRD is considered as a reference technique for the product powder characterization [10]. **Figure 1** shows the diffraction features of the polycrystalline-Si, (SiO_2/Si), crystalline-SiO_2 and amorphous-SiO_2 using a combination between ACE and ultra-sonication process [at different values of KOH_{conc} and (t)]. **Figures 1(a)-(c)** show the diffraction patterns at 28.22˚, 47.19˚, 56.03˚, 68.97˚, 76.25˚, 87.93˚, and 94.8370˚ (JCPDS card nos. 01-079-0613 and 00-027-1402) [11], which correspond to planes (111), (220), (211), (400), (331), (422), and (511), respectively. As shown in **Figure 1(b)** (t = 3 hours), amorphous-SiO_2 appears as a broadened peak at $2\theta = 21.5˚$ for producing (SiO_2/Si). And, **Figure 1(b)** and **Figure 1(c)** (t = 4 hours) show the enhancement broadening peak that corresponds to the crystalline-SiO_2 [12].

Finally, **Figure 1(b)** and **Figure 1(c)** (t = 5 hours), the SiO_2-peak high intensity appeared at $2\theta = 20˚$ that corresponds to the complete transformation to amorphous-SiO_2 [13]. Then, the preparation conditions during the crystalline-SiO_2 formation are [4.5 and 6 weight % KOH at t = 4 hours]. But, the amorphous-SiO_2 is formed at t = 5 hours. From the reported data, (t) has a great effect in the controlling process during the SiO_2 type preparation.

Figure 2 shows the chemical bonding formation of SiO_2 types; the broadening bands (820 and 1300 cm^{-1}) assign to Si-O-Si asymmetric-stretching-vibrations [14], and the narrow bands (1089 and 1093 cm^{-1}) correspond to the SiO_2 presence [15]. The bands (801 and 803 cm^{-1}) are due to symmetric-stretching-vibrations of siloxane-groups (Si-O-Si), as shown in **Figure 2(a)** and **Figure 2(b)**, respectively.

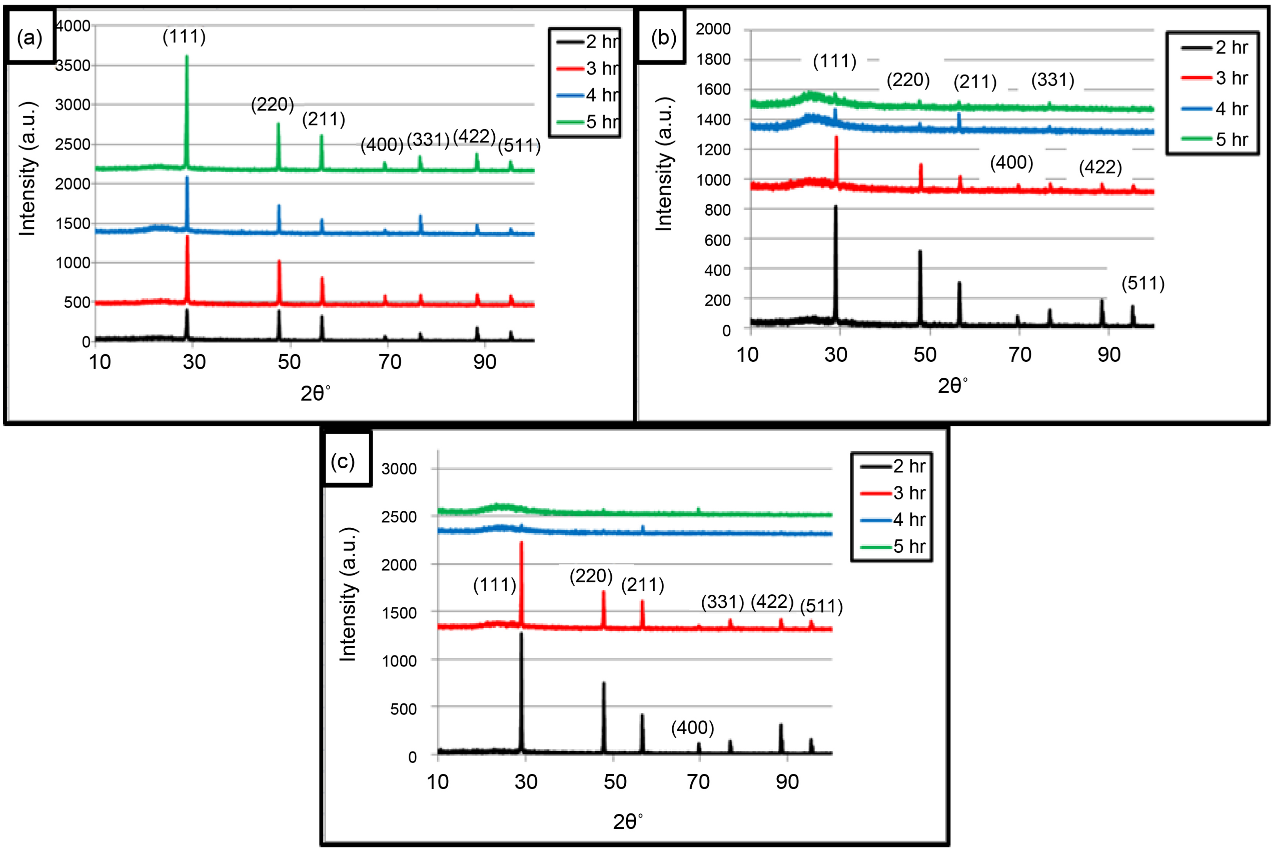

Figure 1. XRD of Si-polycrystalline, SiO_2/Si crystalline SiO_2 and amorphous-SiO_2 powders obtained with different KOH_{conc} and t; (a) 3 wt% KOH, (b) 4.5 wt% KOH, and (c) 6 wt% KOH.

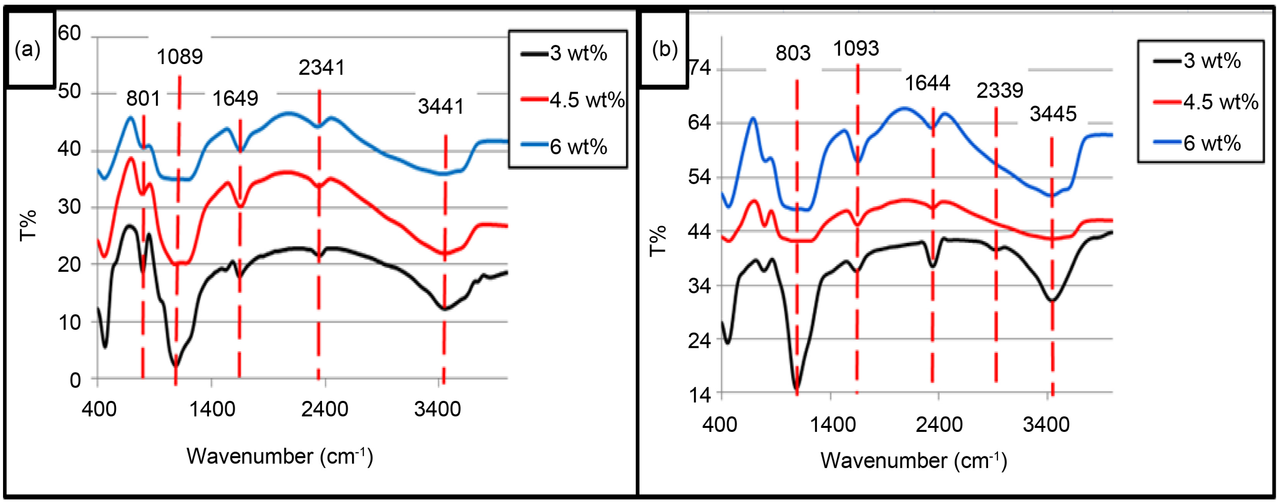

Figure 2. FTIR of the precipitated SiO_2-nanoparticle obtained with different KOH_{conc} prepared by ACE; (a) $t = 4$ hr, (b) $t = 5$ hr.

The O-H bending-vibration bands of the adsorbed water are located at 1649 and 1644 cm^{-1}, and the O-Hstretching-vibration bands at 3441 and 3445 cm^{-1} (at $t =$ 4 and 5 hours). The presence of water molecules proves that the amorphous-SiO_2 chemical formula is close to SiO_x yH_2O form [16]. In addition, the shoulder at 2341 cm^{-1} is assigned to the stretching-vibrations of Si-OH groups in the amorphous-SiO_2 structure [15], which also confirm the XRD data as shown in **Figure 1**.

Figure 3 presents SEM-micrographs of SiO_2 powder morphology; **Figure 3(e)** shows the inversely proportional relation between (t) and SiO_2 construction. At 6 weight % KOH; **Figures 3(a)-(d)** correspond to $t = 4$ and 5 hours, respectively. The pores interconnection is the most important structure characteristics of porous-SiO_2 prepared in this work, by which the type of application is determined. Then, at the increasing of (t) values, the verification of SiO_2 became more obvious. This relation proves the porosity percent enhancement at increasing (t). **Figure 3(c)** shows the particle size value (0.59 - 1.16 μm) and the pore size that's nearly in the same range. But, at $t = 5$ hours (**Figure 3(b)**), the SiO_2 particle size range decrease to (0.08 - 0.34 μm). So, SEM-images are in line with XRD data, as a result of complete conversion to the amorphous-SiO_2. So, the product powder is suitable for using as heavy metal removal and dye removal as shown in last literatures [17].

The basic chemical reactions of SiO_2 powder synthesis are presented in the following mechanism:

$$\mathrm{Si} + 2\mathrm{KOH} + 2\mathrm{H_2O} \rightarrow \mathrm{Si(OH)_4} + 4\mathrm{e}^- + 2\mathrm{K}^+ + \mathrm{H_2} \quad [18] \tag{1}$$

At high pH range (alkaline medium) [1]:

$$\mathrm{Si(OH)_4} + \mathrm{OH}^- \rightarrow \mathrm{Si(OH)_3O}^- + \mathrm{H_2O} \tag{2}$$

$$\mathrm{Si(OH)_3O}^- + \mathrm{Si(OH)_3O}^- \rightarrow \mathrm{Si(OH)_3\text{-}O\text{-}Si(OH)_2O}^- + \mathrm{OH}^- \tag{3}$$

The SiO_2 morphology [nano-porous-sphere and nano-porous-cubic] is visualized by TEM-images. **Figure 4(a)** and **Figure 4(c)** show the nano-porous-sphere SiO_2 (90 nm), with different geometric projections, that's synthesized at the

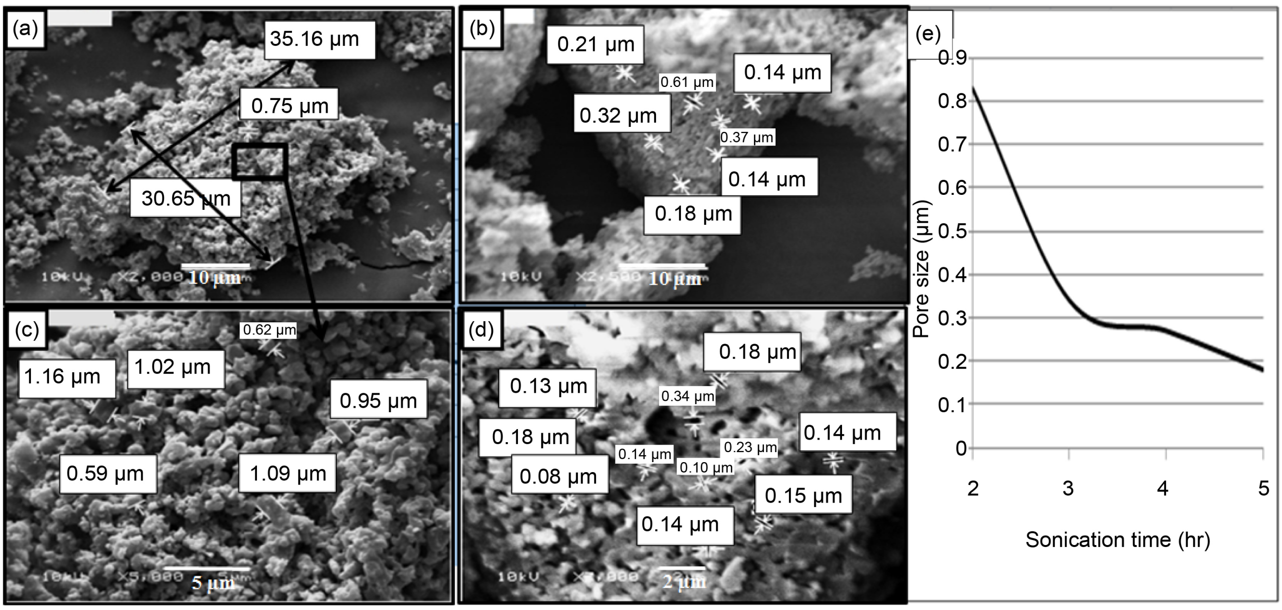

Figure 3. SEM images of porous-SiO_2 obtained with (a) and (c) 6 wt% KOH and $t = 4$ hr, (b) and (d) 6 wt% KOH and $t = 5$ hr. (e) The relation between t (hr) and the pore size (μm).

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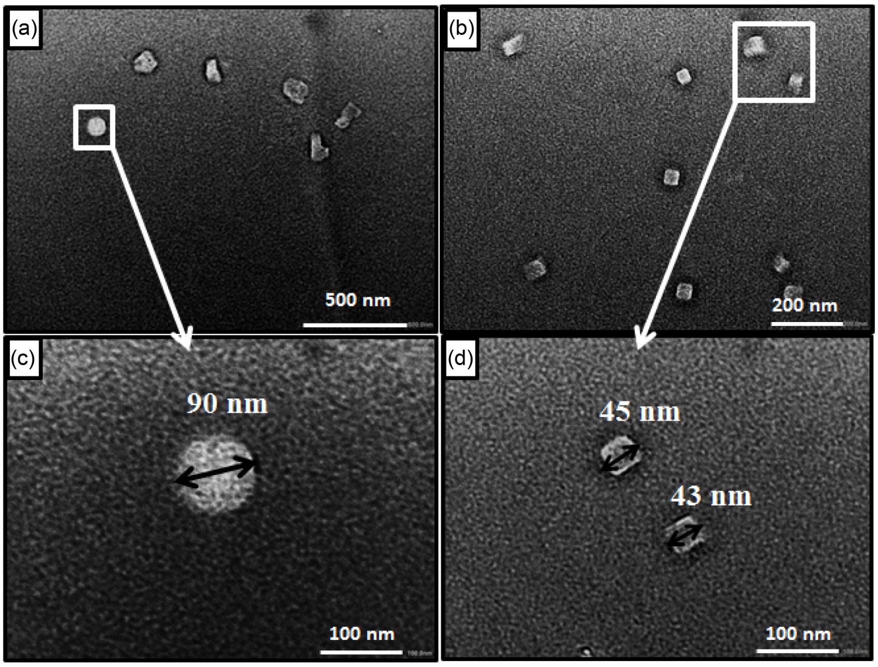

Figure 4. TEM-micrographs of SiO_2-nanoparticles obtained with (a) and (c) 4.5 wt% KOH and *t* = 3 hr, (b) and (d) 4.5 wt% KOH and *t* = 4 hr.

preparation conditions 4.5 weight % KOH and *t* = 3 hours. But, at *t* = 4 hours, the nano-porous-cubic particles (45 nm) are appearing that agree the XRD-data in Figure 1(b). Then, it has high surface area that forms anion surfactants, which is used in many industrial applications [19]. Also, the crystalline-SiO_2 [1] is recorded in Figure 4(b) and Figure 4(d). Then, the critical preparation conditions of porous-crystalline and amorphous-SiO_2 are 6 weight % KOH at (*t* = 4 and 5 hours), as shown in Figure 1(c), Figures 3(a)-(d). Either in Figure 1(b) and Figures 4(a)-(d), at the different preparation conditions [4.5 weight % KOH at *t* = 3 and 4 hours], the nano-sphere and nano-cubic-SiO_2 shapes, respectively, are described.

Thus, it's found that the sonication time is controlled in the form and size of powder produced. Due to the ultra-sonication process is a growth inhibition technique especially in presence of alkali chemical etching process. So, the enhancement volume process is intermittently synchronized with the base etching process, causing the final shape of the product to change.

4. Conclusion

The pore size of the interconnected porous-SiO_2 network in the micron range is successfully formed using commercial Si-powder as a cheap source of SiO_2 (different morphological shapes). The particle size of the crystalline-porous-SiO_2 powder is in range (0.59 - 1.16 μm), but in case of amorphous one is in range (0.08 - 0.34 μm). The successful production of SiO_2 powder is through a combination ACE and ultra-sonication technique and subsequent drying at room temperature. It provided optimal conditions for the generation of micro-porous-crystalline and amorphous-SiO_2 network. So, it's a novel method for controlling the synthesis of several SiO_2-nano-shapes {spherical (90 nm) and cubic (45 nm)} production.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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