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Morphological and Optical Studies of Silica Powders from Waste-Materials by Milling Process for Light-Harvesting Applications

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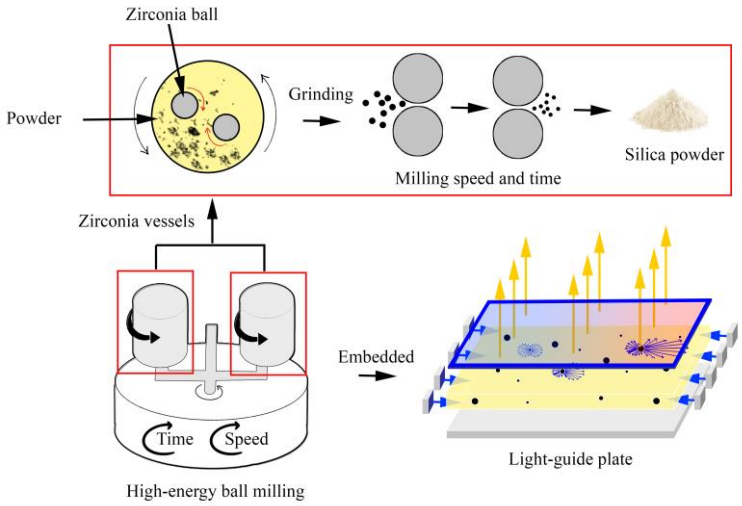
Abstract

This study focuses on the synthesis process and characterization of silica powders from wasted material obtained by milling process in which mechanical force is applied to material until it becomes silica powders with uniform distribution in their size. Effects of milling process parameters including milling speed and milling time on structural, morphological, and optical properties of the prepared powders are investigated by SEM technique and particle size analysis. Two steps of the ball milling process were conducted to further reduce the particle size of the powder. The uniform size distribution in the range of lower than 1 μm was achieved by two milling steps at milling speed 300 rpm for 30 min. Moreover, the increase of milling speed and time results to the agglomeration of fine particles forming the cluster of particles. Furthermore, the light scattering characteristic of the milled silica powders is examined in terms of size-related scattering behavior which is meaningful for light-harvesting applications including a light-guide plate with embedded light scatterers.



Keywords: Light scattering particles; Milling process; Silica powder; Waste material

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Introduction

Recently, nanomaterials and nanotechnologies have been attractive for study and development in different fields which is related and arranged on a scale less than 100 nm. The shape and size of materials in nanoscale have significantly strong influence on the physical and chemical properties of a substance due to high specific surface area. Therefore, the importance of studying characteristic properties of materials with different nanoscale sizes is still in focus since it can considerably change relevant electrical, optical, and chemical properties [1]. Various works have been dedicated to nanomaterial synthesis from commercial materials. For example, D. Ovali *et al.* reported the characterization of SiO_2-encapsulated WSi_2/W_5Si_3 nanoparticles synthesized from SiO_2 purchased from Sigma Aldrich [2]. Y. Nakashima *et al.* studied the activation of silica powder surface by a planetary ball milling [3] and I. Son *et al*. prepared highly transparent and wide viewing optical films embedded with SiO_2 nanoparticles derived from Korea Nanomaterials [4]. This research work focuses on the preparation of nanomaterial from waste-material including glass bottles. Most glass composition is made from silicon dioxide or silica whose content depends on production processes [5]. The advantages of silica include a high melting point, insoluble and durable resistance in reaction and corrosion. Silica powder has been widely used in industrial products such as electronics, optical instruments, and optical fibers manufacturing [4, 6, 7]. Meanwhile, important physical properties of silica are its transparency with strong absorptivity in the UV region that could be performed as light scattering material for the development of the light-harvesting application. Moreover, the particle size of silica powder can be scaled down to nanometer regime leading to high performance in its optical properties [8]. For the desired application especially the light guide plate module, a specific size of embedded silica nanoparticle is strongly required to enhance light scattering and improve light emitting from the plate.

Generally, silica powder could be produced by various processes including self-propagating high-temperature synthesis [9], mechanical alloying [10], plasma spray processing [11], in-situ solid-state displacement [12], hot pressing [13], and spark plasma sintering technique [14]. However, some difficulties and complex processes are encountered in silica production owing to high melting point and hardness [15]. In this research, a suitable and simple method is the key role for silica synthesis from waste material. Therefore, the high energy ball milling process is chosen as a proper process in this work, which reduces its particle size by mechanical force [16, 17]. The relevant parameters in the milling process for size reduction are focused on milling time and speed relating to particle aggregation [18]. Therefore, structural property, morphology, and particle size of silica powders after the milling process with different milling speeds and times by high energy ball milling process have been investigated systematically. Meanwhile, the possibility in light scattering mechanism of silica powder form waste material with a suitable particle size of a material is proposed by optical properties.

Materials and Methods

Materials Preparation

Silica powder was prepared from a wasted glass bottle by the coarse grinding process until its particle size was less than 100 μm. After that, as-prepared silica powder was loaded in zirconia

vessels and operated by a tilted planetary ball mill machine (planet M2-3F). The weight ratio of silica powder and zirconia ball (diameter 5 mm) was fixed at 1:10 in both zirconia vessels. Milling process of coarse silica powder was operated at milling speed of 500 rpm for different times of 30, 60 and 90 min to obtain solid powder and labeled as the first milling condition (R1). For next milling process, the prepared silica powder R1 was operated for the second milling process and was labeled as R2 with milling speed 300 and 500 rpm and milling times of 30, 60 and 90 min. The details of the milling condition are shown in Table 1.

Table 1 Milling conditions for the preparation of silica powders.

Milling condition	Time (min)
R1 at 500 rpm	30
	60
	90
R2 at 300 rpm	30
	60
	90
R2 at 500 rpm	30
	60
	90

Characterization of the Silica Powder

Morphologies of coarse and milled silica powders were monitored by a field emission scanning electron microscope (FE-SEM, Thermo Scientific, Apreo S). The sample was placed on the carbon tape and sputter-coated with gold for conduction and then placed on the holder. XRF analysis (Bruker, S8 Tiger) was carried out to determine the relevant elements of the powders. The particle size of after-milled powders prepared with different speeds and times was determined by particle size analyzer (Delsa Nano C, Beckman Coulter). All samples for measurement were dispersed in water. Meanwhile, optical reflectance in diffuse reflectance mode of the samples was analyzed by UV-Visible Spectrophotometer (U-400, Hitachi).

Results and Discussion

The size of milled silica powder from waste material dispersed in water was measured by particle analyzer and the corresponding results are depicted in Fig. 1. For first milling round (R1), the particle size distribution of silica powders milled at 500 rpm is found to be in the range of 1 micron, especially at milling time 30 and 90 min. Meanwhile, the condition of milling time at 60 min can significantly reduce the particle size to be less than 1 μm. This result indicates that particle size of silica powder is still in the range of microscale, whilst repeated milling process or second milling round (R2) with different milling speeds and times should be required for further particle size reduction. For R2 operating at 500 rpm (same milling speed as R1), the 50% decrease in particle size in a range of 700 – 1,000 nm was obtained when the sample was milled again for 30 min. Meanwhile, the increase in size distribution was occurred by prolonging milling time for 60 and 90 min that could be due to high thermal energy supplied in the system resulting to the agglomeration of the milled particles. In the case of R2 operated at 300 rpm, particle size

distribution is nearly less than 1,000 nm for all milling time conditions when comparing to the first milling round. As the milling time increases, particle size lower than 700 nm is obtained and formation of a large particle is initiated (more than 1 μm). The particle size of silica powder at milling speed 300 rpm is smaller than that of sample milled at 500 rpm due to excess energy supplied by high-speed milling resulting to particle aggregation. Regarding this result, the particle size distribution of silica powder at a milling speed of 300 rpm for 30 min in the second milling round shows the highest percentage in the range of 700 – 1,000 nm which is considered an optimized milling condition. However, some errors on particle size analysis were occured due to the agglomeration in the solution. The increase in the milling speed and time is the key role for the variation in particle size distribution, especially the generation of thermal energy inside the vessels provided by high energy ball milling process leading to an agglomeration of particle size [19].

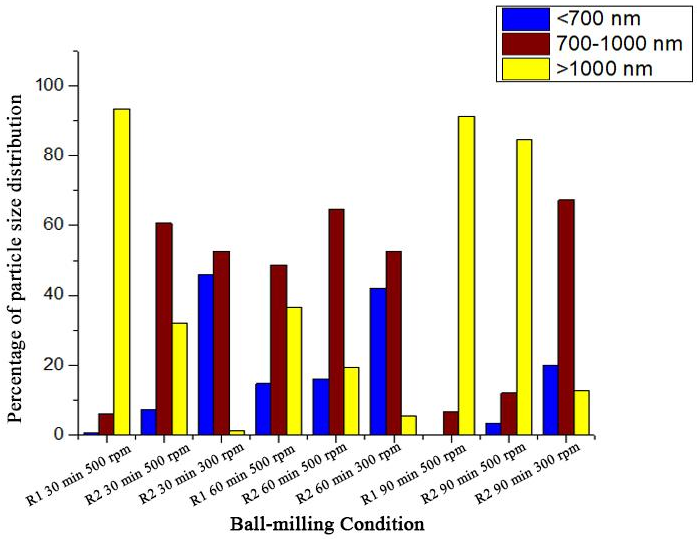

Fig. 1 Particle size analysis of silica powders milled at various milling speeds and times by high energy ball milling process. (The conditions of R1; operated at 500 rpm and R2; operated at 300 and 500 rpm with milling time mill 30, 60 and 90 min.)

The elemental composition of the silica powders milled at different milling times was quantitatively determined by XRF. Table 2 shows the major SiO_2 compound of milled silica powder of 69.46%, 70.30% and 69.91% for the samples with milling time of 30, 60 and 90 min, respectively. This result suggests that the milling process does not have significant effect on the change in major compound of the powder. SEM images of silica powders prepared at different milling times and milling speeds are shown in Fig. 2. The morphologies of silica powder in the first milling round at milling time of 30 and 90 min reveal the different features as seen in Fig. 2(a) and (d). Size analysis of the sample milled for 30 min is consistent with distribution in its size of approximately 1 – 20 μm. After milling was repeated (R2), the particle size of silica powder is found to be in fine powder dispersing on the surface of the larger particle. Interestingly, the sample milled for 90 min exhibits inconsistent and non-uniform size as seen in SEM images and particle size analysis because the small particle could tightly cling to each other leading to

agglomeration of small particles. SEM analysis suggests that excessive milling time in the milling process could lead to particle agglomeration and a significant increase in particle size.

Table 2 XRF measurement of waste materials milled at different milling times.

Time	Content of Elements (%)											
	SiO_2	Na_2O	CaO	MgO	Al_2O_3	SO_3	Cl	K_2O	TiO_2	Fe_2O_3	Y_2O_3	ZrO_2
30	69.46	12.39	12.62	2.09	1.40	0.23	0.03	0.13	0.09	0.25	0.07	1.24
60	70.30	12.51	12.63	2.09	1.38	0.22	0.02	0.13	0.09	0.25	0.02	0.36
90	69.91	12.22	12.96	2.09	1.36	0.24	0.03	0.14	0.09	0.26	0.04	0.66

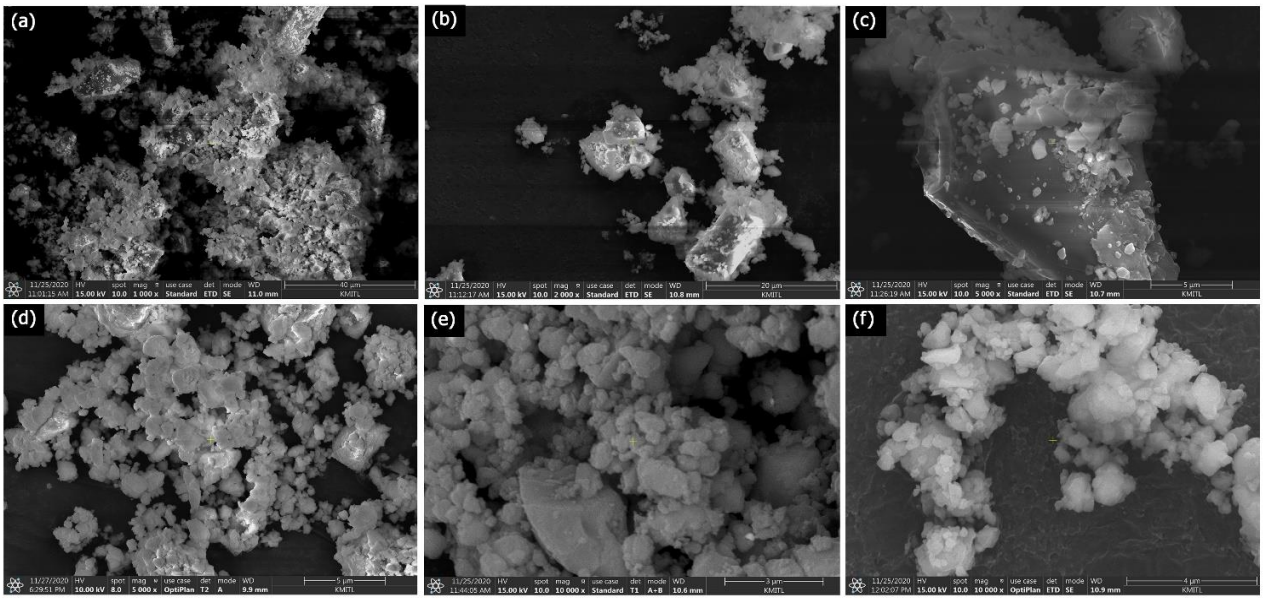

Fig. 2 SEM images of silica powder prepared with different milling speeds and milling times at R1 (a) 500 rpm 30 min and (d) 500 rpm 90 min and R2 (b) 500 rpm 30 min, (c) 300 rpm 30 min, (e) 500 rpm 90 min and (f) 300 rpm 90 min.

Diffuse reflectance spectra (DRS) in the range of 250 – 800 nm of silica powders prepared at different milling times are shown in Fig. 3. It can be observed that all silica powder samples exhibit the same patterns and high transparency in the visible region. The increase of reflectance intensity is observed by prolonging milling time attributing to size reduction of fine powder after milling process and increasing light scattering phenomena of the nanoparticles. When visible light impinges on the small particles whose size is in the range of light wavelength (400 – 700 nm), greater amount of photons could be scattered from the powders resulting in the increasing reflectance intensity. Regarding this mechanism, it is suggested that the improvement of light scattering performance in light guide plate applications is expected by specific light wavelength and particle size ascribed to Rayleigh scattering [20]. However, the type of scattering depends on size of particle. If the size of particle is smaller than the incident light wavelength, the scattering phenomena can be explained by the Rayleigh scattering as described in Fig. 4(a). Mie scattering is dominant when the size of particles is larger than the incident light wavelength as shown in Fig. 4(b). These two scattering phenomenon are considered to be major mechanisms responsible to the light scattering of the scattered particles embedded inside the light-guide plate.

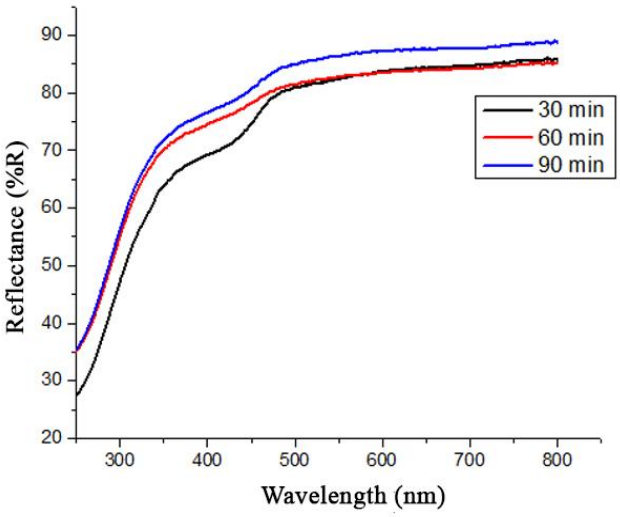

Fig. 3 DRS spectra of silica powders prepared with different milling times of 30, 60 and 90 min.

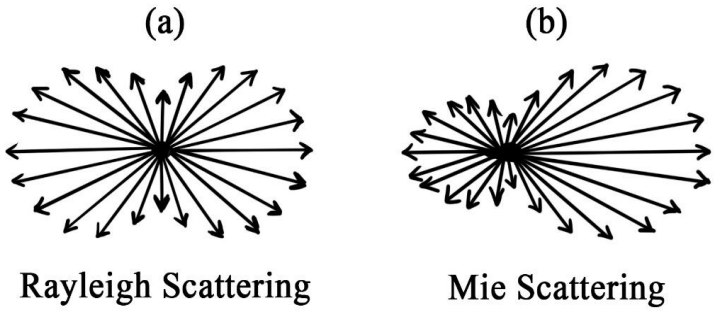

Fig. 4 Schematic illustrations of (a) Rayleigh Scattering (b) Mie Scattering.

Conclusion

Size reduction of silica powder from glass waste-material was carried out using a high-energy ball milling process. The data from SEM images and particle size analysis indicate that milling process parameters including the milling time and milling speed are considered as major factors in the reduction of particle size distribution in both milling processes including R1 and R2. The optimized milling time and speed should be acknowledged for attaining designated particle size. The first round operated at 500 rpm and milling time for 60 min is optimized show the smallest particle size in vicinity of 1 µm. For the second milling process, the powders were repeatedly milled with the same milling time at different milling speeds. The optimized condition was found at the operating speed of 300 rpm for 30 min, showing the smallest particle size compared with other conditions. Meanwhile, high reflectance spectra can be enhanced by the increase in milling time due to the size effect of silica particles on relevant light scattering phenomena.

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