International Journal of Advancements in Technology
ISSN: 0976-4860

Sun Kang, Int J Adv Technol 2017, 8:3
DOI: 10.4172/0976-4860.1000188

Research Article

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The Effect of Recycled Solvent on the Structure of Photonic Crystal

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Abstract

Minimizing or avoiding the creation of waste solvents can be most effective in protecting environment and managing the cost. Since fabrication of silica spheres (approximately 1.0 g of silica spheres with diameter of approximately 300 nm) for photonic crystal requires relatively large amount of solvents (100 ml 2-propanol and 100 ml NH4OH solution), it will be ideal to use recycled solvents for reducing waste solvents. Monodisperse silica spheres were synthesized with Stöber synthetic process. The diameter of the spheres was uniform when the ratio of 2-propanol and NH4OH was 1:1. To investigate the effect of recycled solvents, the ratio of 2-propanol:NH4OH maintained 1:1. The spheres were separated by filtration or centrifuge. The mixture of solvents was recycled repeatedly after separating the spheres. The spheres became irregular shape when the mixture of solvents recycled by filtration more than twice. The solvent was completely dried and collected the residual compound in the mixture of solvents, which was comprised of mainly –OH and Si-O-Si. However, there was no irregular shape and residual solid in the mixture of solvents after 4 times recycling by centrifuge. These results imply that the filtering process removes NH4OH due to the low pressure during the filtering process, but the centrifuge method requires no low pressure process and maintains similar NH4OH concentration after repeated recycling process. Therefore, the recycled solvent mixture can be repeatedly used as long as similar NH4OH concentration maintains.

Keywords: Recycled solvent; Photonic crystal; Irregular structure; NH_4OH concentration

Introduction

Chemical process industries have produced waste solvents, which are of great concern to the industries and to the public and have been suggested many technologies and implemented to reduce waste and associated environmental impacts [1]. Engineers and technologists develop technologies with inherently superior from the point of view of their productivity; the environment has rarely been more pressing; this is true for energy production, use, chemical and other materials processing for many manufacturing industries [2].

Many of the solvents used in manufacturing processes are harmful to the environment and human [3-5]. When environmentally harmful solvents are produced, the following two options can be chosen: firstly, replace the solvents with less or not harmful solvents which are as effective and secondly recycling the solvents that minimize the generation of hazardous waste and result in substantial cost savings [6]. Reducing hazardous wastes has been an important issue for research and manufacturing facilities and can be achieved by changing processes or substituting non-hazardous material instead of hazardous ones. Once the solvent contaminates with undesired material, the solvent is no longer capable of being used for specific operations and should be disposed as hazardous waste or should be recycled. Many researchers have focused on reusing the waste water by ozone treatment [7], nanofiltration and reverse osmosis [8], biological treatment [9] and electrochemical oxidation [10]. Wang et al. successfully recycled not only solvent but also pigment and plasticizer by combined method of selective dissolution and evaporation method for the waste plasticized polyvinylbutyral films [11].

Photonic crystals consisting of periodically arranged materials with notably different refractive indexes have been novel architectures with unique optical properties. The colloid diameter of silica spheres should be smaller than 350 nm to obtain visible range stop band [12]. Experimentally, large volume of solvents requires fabricating small diameter of silica spheres in the range of smaller than 350 nm. Therefore, repeated use of the recycled solvents or reducing the volume of the solvents is the way for reducing the waste hazardous solvents and protecting the environment and human. Reducing the volume of the solvents may not possible for fabricating required diameter of silica spheres. Therefore, repeated use of this mixture solution can reduce the production of environmentally hazardous chemical waste and the cost of the production. In this paper, we report the effect of NH4OH concentration on the diameter of silica spheres. The solvents were recycled by filtering or centrifuge process. The effect of the reduction of NH4OH during the recycling process and the resulting shape of silica spheres were reported in this investigation.

Materials and Methods

Materials

Ammonium hydroxide (NH4OH, 28%), 2-propanol (99%), tetraethyl orthosilicate (TEOS, 98%) were purchased from Sigma Aldrich Co Ltd and used without further purification.

Repeated use of the mixture solvents recycled by filtering process

Mixture of 2-Propanol (100 ml) and NH4OH solution (100 ml) was added to the 250 ml round bottom flask. TEOS (3.5 g) was added to the mixture of the solution, and the solution was stirred for 6 h at room temperature. The spheres were separated by filtering with nylon filter having 200 nm pores. The filtrate solution was charged to the 250 ml round bottom flask. TEOS (3.5 g) was added to the round bottom flask with vigorous stirring. The stirring continued for 6 h at room temperature. The spheres were separated by filtering. This process repeated to fabricate the silica spheres as shown in Figures 1a and 1b. The same process was repeated 4 times. After total 5 times reaction, the solvent was dried, and residual compound was collected. FTIR spectrum was obtained using Nicolet iS5.

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Received: June 07, 2017; **Accepted:** July 07, 2017; **Published:** July 14, 2017

Citation: Sun Kang K (2017) The Effect of Recycled Solvent on the Structure of Photonic Crystal. Int J Adv Technol 8: 188. doi:10.4172/0976-4860.1000188

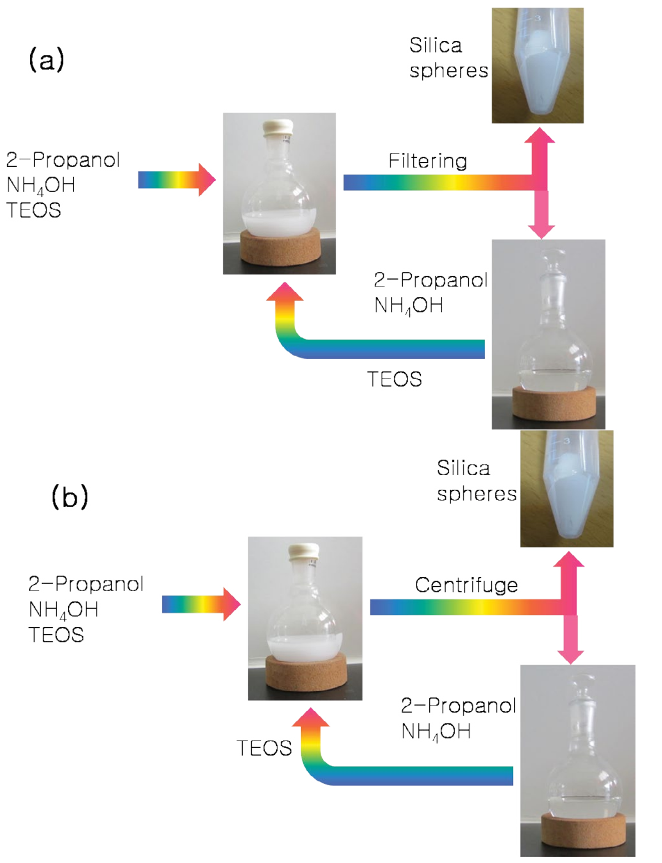


Figure 1: The graphical representation of the solvent recycling process with (a) filtering method and (b) centrifuging method.

Repeated use of the mixture solvents recycled by centrifuge

The same processes were performed compared with section 2.3 except that the solvent was collected by centrifuge process. The collected solvent mixture was repeatedly used 4 times. The final solvent was dried to collect residual solid in the solvent. However, there was no residual compound in the recycled solvent.

Measurements

Silica spheres and residual compounds collected by drying 5 times used solvent with filtration method were dispersed in methanol. The solution was dropped and dried to a KBr plate, and the FTIR spectra were obtained using FTIR spectrometer (Nicolet iS5). Silica spheres were dispersed to methanol. The solution was dropped and dried to a silicon wafer. Field emission scanning electron microscope (FESEM) images and energy dispersive spectroscopic data (EDS) were obtained using field emission scanning electron microscope (JOEL ISM-7401F).

Results and Discussion

Repeated use of the mixture solvents recycled by filtering process

Since most of the solvents are derived from petroleum sources, recycling solvent is valuable. Although many processes (including reverse osmosis or ion exchange) are not suitable for recycling solvents, distillation is a proven method for recycling solvents. However, distillation requires large amounts of energy. Therefore, repeatedly using the solvent is ideal to increase the productivity, if there is no contamination on the solvent. After fabrication of silica spheres, the spheres were filtered with a nylon filter having pore size of 200 nm, and the filtrate solvent was reused to make silica spheres without any treatment. The spheres fabricated with recycled solvents for 0, 1, 2, 3, and 4 times is shown in Figure 2. Two or three connected spheres are observed as shown in Figures 2c-2e shows many connected, irregular-shaped, and aggregated spheres. Undesired shapes of the spheres were drastically increased with the increase of the solvent recycling time. Therefore, using recycled solvent to fabricate monodisperse silica spheres is not appropriate in this process.

FTIR analysis of silica spheres and residual in the solvent.

Figures 3a and 3b shows the FTIR spectra for the spheres fabricated with fresh solvent mixture and 4 times recycled solvent mixture. The broad and strong absorption peak at 3400 cm^{-1} represents the –OH stretching vibration. The absorption peaks at 1634, 1094, 836 cm^{-1} are due to the surface free water molecules vibration mode, Si-O-Si stretching vibration mode, and Si-O stretching vibration, respectively. The absorption peaks at 1462 and 949 cm^{-1} are attributed from –CH deformation and Si-O-C stretching vibration, respectively, which implies possible existence of unhydrolized Si-O-CH_3. The additional evidence was obtained by EDS analysis as shown in Figure 3c, which showed 6.41 wt. % of carbon. The strong absorption peak at 460 cm^{-1} indicates the rocking motion of the bridging oxygen atoms in the Si-O-Si plane. The two spectra are almost identical, which indicates there is no chemical structure difference between two spheres. The 4 times recycled solvent mixture was completely dried and approximately 40 mg of white residual solid was obtained. Since the precursor is TEOS, the residual material is expected to be a Si-O compound. Figure 3 shows FTIR spectrum of the residual material and exhibits strong absorption peak centered at 1100 cm^{-1}. The peak at approximately 3500 cm-1 corresponds to the absorption of Si–OH stretching vibration. This result indicates that the Si-O-Si and Si-OH compound remains in the solvent. This residual silica compound might be the cause of the irregular shape of the spheres after repeated use of the solvent.

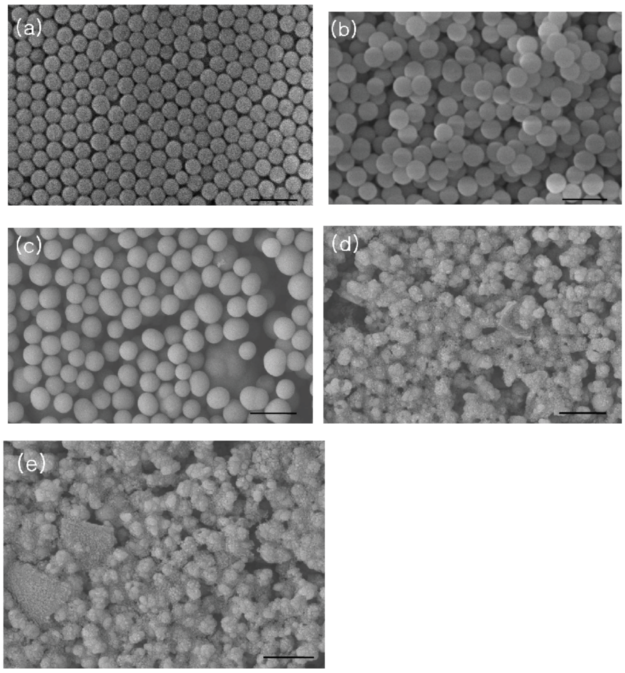
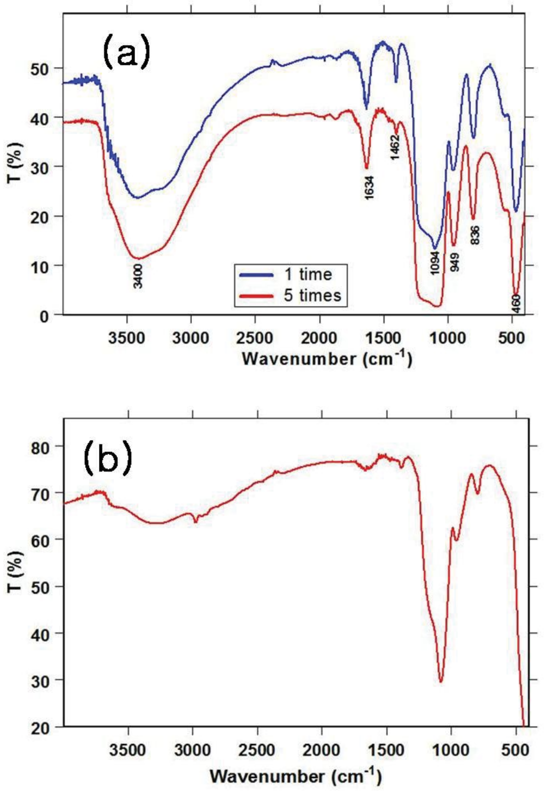


Figure 2: FESEM images of silica spheres fabricated with (a) 0, (b) 1, (c) 2, (d) 3, and (e) 4 times recycled solvents with filtering method. The bar size is 1 μm.



(c)

Element	Weight %	Atomic %
C	6.41	9.92
O	56.26	65.37
Si	37.33	24.71

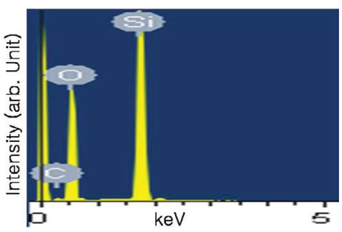


Figure 3: FTIR spectra of silica spheres fabricated with recycled solvent (a) 0 and 4 times, (b) the residual solid in the solvents recycled 4 times by filtering process, (c) EDS spectrum of silica spheres.

Repeated use of the mixture solvents recycled by centrifuge process

The solvent mixture and silica spheres were separated by centrifuge. The separated mixture solvent was reused five times to fabricate silica spheres without any treatment. Figures 4a to 4e show silica spheres fabricated with fresh solvent mixture, 1, 2, 3, and 4 times recycled solvent mixture, respectively. There are no connected, irregular-shaped, and aggregated spheres. Such a drastic difference between the spheres fabricated with the recycled solvent mixture by filtration and centrifuge is due to the NH4OH concentration. Since low pressure should be used during the filtering process, large amount of NH4OH can be removed. The silica residuals remain in the solution due to the low concentration of NH4OH. However, the NH4OH may not disappear during the recycling process by centrifuge. Therefore, maintaining the high concentration of NH4OH causes full incorporation of silica compound to silica spheres.

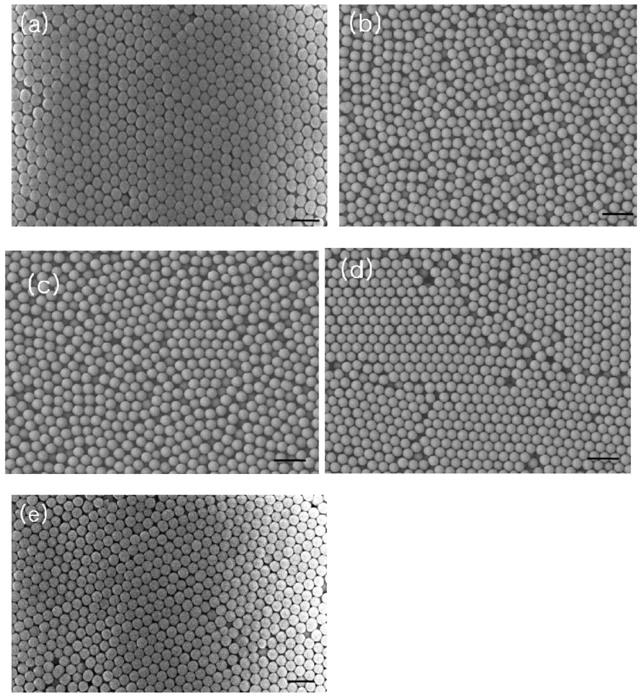


Figure 4: FESEM images of silica spheres fabricated with recycled the solvents (a) 0, (b) 1, (c) 2, (d) 3 and (e) 4 times. The bar size is 1 μm.

Conclusion

Recycling process with filtration lost too much NH4OH due to the low pressure process. Silica compound remained in the recycled solvent mixture due to the low NH4OH concentration, which led that the silica compounds were not fully incorporated to silica spheres. The residual silica compounds cause the connected, aggregated, and irregular-shaped spheres. Therefore, recycling the solvent by filtration is not appropriate method in repeated using the solvent. However, silica spheres fabricated with many times recycled solvent by centrifuge showed no defect-pattern, which was due to the maintenance of high concentration of NH4OH. This result indicates that the recycled solvent can be repeatedly used as long as the appropriate NH4OH concentration maintains.

Acknowledgment

This research is performed under the support of Kyungil University.

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