



Research Report

Synthesis of Highly Monodispersed Mesoporous Tin Oxide Spheres

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■ABSTRACT■ We describe the synthesis of highly monodispersed mesoporous tin oxide spheres (MMTOS) via a two-step nanocasting process using monodispersed mesoporous silica spheres (MMSS) as a starting template. Through the oxidation of tin (II) chloride on the surface of monodispersed starburst carbon spheres (MSCS), which were produced using MMSS as a host, tin oxide nanocrystals accumulate into MSCS mesopores. Calcination of the composite MSCS/SnO₂ yields MMTOS that consist of SnO₂ nanocrystals. Although the starburst structure of MSCS is lost during calcination, the monodispersed spherical shape is retained. Additionally, fabrication of a three-dimensional colloidal photonic crystal is presented. Due to its uniformity, MMTOS can self-assemble into a close-packed array that has a stop band in the visible region. Using the combination of MMTOS and incorporated materials, it is expected to be possible to fabricate novel photonic devices such as optically amplified solar cells and zero-threshold lasers, by exploiting the unique properties of photonic crystals and their conductivity characteristics.

■KEYWORDS■ Tin oxide, Porous materials, Photonic crystal, Sphere, Hard template, Electronic conductivity, Close-packed opal, Carbon, Nanocasting

1. Introduction

Photonic crystals are very attractive for optical applications since light emission is suppressed in periodic structures.^(1,2) Some of the most promising photonic crystals are colloidal crystals consisting of periodic arrays of monodispersed colloidal spheres formed by self-assembly. They are expected to be used in optical applications such as low-threshold lasers,^(3,4) waveguides,⁽⁵⁾ and optical fibers⁽⁶⁾ because light propagation is confined by the photonic band gap created by periodic modulation of the refractive index. A number of studies have reported fabrication and application of colloidal photonic crystals, but few studies have used porous materials.

We have succeeded in synthesizing monodispersed mesoporous silica spheres (MMSS).⁽⁷⁻¹⁰⁾ MMSS with highly monodispersed particle diameter and pore size could be used to form colloidal crystals with uniform mesopores through self-assembly. The color of the MMSS array varies depending on the relative pressure of water or benzene vapor.^(10,11) Laser oscillation has been generated by using the stop-band effect to confine emission from dye molecules embedded in mesopores of MMSS.⁽¹²⁾ MMSS can adsorb various nanoparticles and organic dyes.⁽¹³⁻¹⁵⁾ Colloidal photonic crystals containing nanoparticles or organic dyes fabricated

from MMSS are expected to exhibit novel photonic properties. However, the application of MMSS and its composites is limited its low electrical conductivity.

A method has recently been reported for preparing mesoporous metal oxide such as CeO₂,⁽¹⁶⁾ Co₃O₄,⁽¹⁷⁾ Fe₂O₃,⁽¹⁸⁾ TiO₂,⁽¹⁹⁾ SnO₂⁽²⁰⁾ using a nanocasting process that employs mesoporous silicas or carbons as templates.⁽²¹⁾ Mesoporous tin oxide is expected to be used in various applications including dye-sensitized solar cells, Li battery electrodes, and sensors.

Mesoporous tin oxide has been prepared using a soft-templating method that employs an alkylammonium salt as a template and tin (IV) chloride or tin tetraethoxide as a precursor.^(22,23) A highly crystalline mesoporous tin oxide film has been obtained by using a heat-resistant block-copolymer as a template.⁽²⁴⁾ Mesoporous tin oxide spheres consisting of tin oxide nanoparticles were obtained by removing organic components from a sol of tin oxide nanoparticles and furfural.⁽²⁵⁾ Another type of mesoporous tin oxide has been synthesized by using SBA-15 as a hard template and tin (IV) chloride as a tin oxide precursor.⁽²⁶⁾ However, no study has reported the synthesis of monodispersed mesoporous tin oxide spheres (MMTOS) with a sufficiently high monodispersity to permit them to self-assemble into colloidal crystals.

Colloidal photonic crystals fabricated from MMTOS

are expected to exhibit relatively high electrical conductivity, light confinement, and reduced group velocity of light and thus have the potential to form novel photonic devices. Kuo et al. have fabricated a tin oxide array with an inverse opal structure.⁽²⁷⁾ However, colloidal photonic crystals with an opal structure produced from mesoporous tin oxide spheres have not been reported.

The present paper describes the synthesis of MMTOS.⁽²⁸⁾ It also presents fabrication of a three-dimensional colloidal photonic crystal. MMTOS have been synthesized by a two-step nanocasting process; specifically, we prepared size-controlled MMSS and then fabricated a carbon replica using the MMSS as a template for MMTOS. A three-dimensional colloidal photonic crystal fabricated from MMTOS exhibited a stop band in the visible light region.

2. Experimental

2.1 Chemicals

Tetramethoxysilane and octadecyltrimethylammonium chloride were purchased from Tokyo Kasei Co., Ltd. 1,3,5-trimethylbenzene, furfuryl alcohol, 48% hydrofluoric acid, concentrated hydrochloric acid (HCl), and Tin dichloride (SnCl_2) with purity of 99.9% were obtained from Wako Junyaku Co., Ltd. Docosyltrimethylammonium chloride was purchased from Lion Akzo Co., Ltd. Poly(diallyldimethylammonium chloride) (abbreviated as PDADMAC, $M_w = 200,000$ -350,000, 20 wt% aqueous solution) and poly(sodium 4-styrenesulfonate) (abbreviated as PSS, $M_w = 200,000$, 30 wt% aqueous solution) were purchased from Aldrich Chemical Co., Inc. All chemicals were used without further purification.

2.2 Synthesis of mesoporous tin oxide spheres

Scheme 1 depicted the strategy we used to synthesize MMTOS.

MMSS was prepared using previously reported procedure^(7,8) employing octadecyltrimethylammonium chloride as a templating agent and tetramethoxysilane as a silica source. The mesopores of MMSS were expanded by solvothermal treatment involving either surfactant exchange with docosyltrimethylammonium chloride⁽²⁹⁾ or incorporation of swelling agent 1,3,5-trimethylbenzene.⁽¹⁴⁾

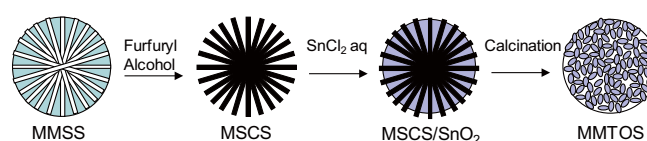
In the first nanocasting process from silica to carbon,

monodispersed starburst carbon spheres (MSCS) were synthesized by using pore-expanded MMSS as a hard template^(30,31) and furfuryl alcohol as a carbon source.⁽³²⁾ Furfuryl alcohol was introduced into the MMSS until their mesopores were filled with the alcohol. They were then polymerized in air at 423 K. The sample was heated at 773 K in a N_2 atmosphere. The remaining pores were filled with furfuryl alcohol again. The sample was polymerized at 423 K in air and carbonized at 1173 K in N_2 flow to obtain pore-expanded MMSS/carbon composites. The silica templates were dissolved using hydrofluoric acid, leaving a carbon material.

In the second nanocasting process, SnO_2 nanoparticles were introduced into MSCS templates using a modified version of Cao's procedure.⁽³³⁾ 100 mg of MSCS was dispersed into a solution containing 250 mL of distilled water, 4 mL of concentrated HCl, and 5.0 g of SnCl_2 . This solution was stirred for 4 h at room temperature, filtered, and rinsed twice with distilled water to obtain a MSCS/ SnO_2 composite. The carbon template was then removed by calcination at 623 K for 12 h or 823 K for 6 h in air to obtain MMTOS.

2.3 Preparation of colloidal crystals

100 mg of MMTOS with an average diameter of 320 nm was dispersed into 25 g of 1 g/L PDADMAC aqueous solution containing 0.5 mol/L sodium chloride by stirring for 20 min. PDADMAC-coated MMTOS



Scheme 1 Nanocasting route used to obtain monodispersed mesoporous tin oxide spheres (MMTOS). Monodispersed mesoporous silica spheres (MMSS) are used as the starting material. Monodispersed starburst carbon spheres (MSCS) obtained from MMSS are immersed in an aqueous solution of SnCl_2 . Oxidation of Sn^{2+} produces SnO_2 in the spaces of MSCS. The resultant MSCS/ SnO_2 composite is calcined to produce MMTOS composed of SnO_2 nanocrystals.

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were obtained after removal of excess PDADMAC by centrifuging twice at 5000 rpm for 10 min and rinsing with water. The PDADMAC-coated spheres were then dispersed in a solution containing 25 g of 1 g/L PSS aqueous solution and 0.5 mol/L sodium chloride. PSS was deposited onto the PDADMAC-coated spheres after the above rinsing. Furthermore, polyelectrolyte-coated MMTOS spheres were dispersed in water (1 wt%) by stirring for 1 h. The suspension was dropped onto a flat quartz plate to form a close-packed array.

2.4 Characterization

The samples were obtained by scanning electron microscope (SEM; Akashi-Seisakusho, Sigma-V) and transmission electron microscope (TEM; JEOL, JEM-2000EX) to determine their particle size and morphology. The average particle diameter and coefficient of variation (CV) were estimated from 50 particles in an SEM image. The MSCS/SnO₂ composites and MMTOS were analyzed by X-ray diffraction (XRD; Rigaku, RINT TTR) with Cu K α radiation. The pore structures of the samples were analyzed by nitrogen adsorption-desorption measurements (Bell. Co., BELSORP-mini II) at 77 K. The samples were degassed at 423 K in 10⁻⁶ Torr for 2 h prior to each adsorption measurement. The specific surface area and the pore size distribution were calculated from the adsorption branch by the Brunauer,

Emmett, and Teller method and the Barrett, Joyner, and Hallender (BJH) model, respectively. The total pore volume was determined from the amount of adsorbed nitrogen at $P/P_0 = 0.95$.

3. Results and discussion

3.1 Morphology change during preparation

Figure 1 shows SEM images of pore-expanded MMSS, MSCS, the MSCS/SnO₂ composite, and the resultant MMTOS obtained by calcination at 623 and 823 K. **Table 1** lists the average particle sizes and CV of these samples. The MSCS were 7.5% smaller than

Table 1 Properties of samples.

Samples	Particle size /nm	CV /%	Pore volume /mLg ⁻¹	Specific surface area /m ² g ⁻¹	Pore diameter /nm	Crystalline size /nm
Pore-Expanded MMSS	940	3.2	1.74	912	6.8	-
MSCS	860	3.3	0.84	1560	1.8	-
MSCS/SnO ₂ composite	860	7.4	0.087	129	< 1.0	2.1
MMTOS calcined at 623 K	700	3.7	0.19	103	6.3	4.7
MMTOS calcined at 823 K	690	4.6	0.17	35	18	10.2

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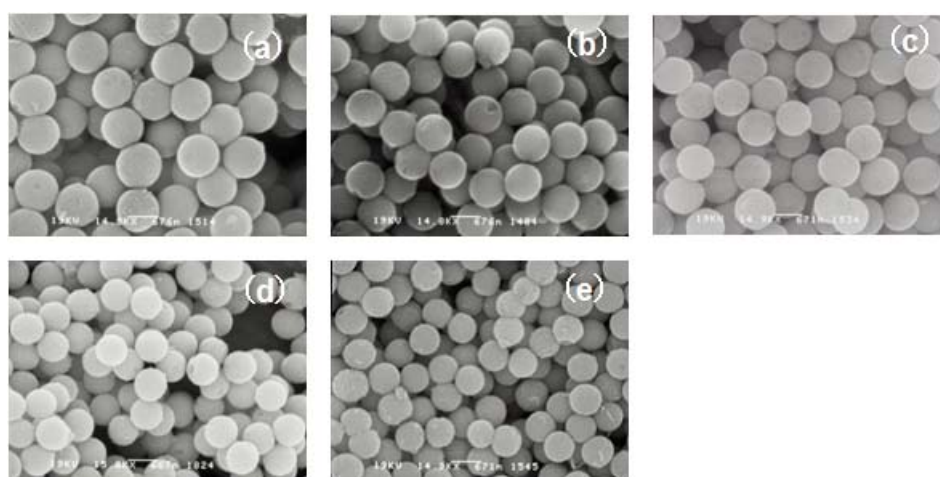


Fig. 1 SEM images of (a) pore-expanded MMSS, (b) MSCS, (c) MSCS/ SnO₂ composite, and MMTOS calcined at (d) 623 and (e) 823 K.

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the pore-expanded MMSS due to shrinkage during carbonization in the first nanocasting step. The particles also shrank by about 20% during calcination of the MSCS/SnO₂ composite during the second nanocasting step; the average particle size decreased from 860 nm to 700 and 690 nm when calcined at 623 and 823 K, respectively. However, the spherical shape and the monodispersity of MMTOS were preserved even after calcination. The MMTOS calcined at 623 and 823 K had CVs of 3.7 and 4.7%, respectively.

3.2 Structure of SnO₂ in the nanopores of MSCS

Figure 2(a) shows an XRD pattern for the MSCS/SnO₂ composite. Very broad, weak peaks are observed at 2θ of 27, 34, and 53°, which correspond to diffractions from the 110, 101, and 211 planes of SnO₂ crystal, respectively. The SnO₂ nanocrystals are thought to deposit on the surface of MSCS through oxidation of Sn²⁺ to Sn⁴⁺ in the acidic aqueous solution. The crystallite size estimated by the Scherrer equation was about 2.0 nm. No particles are observed on the outer surface of MSCS in the SEM image for MSCS/SnO₂ composites (see Fig. 1). The MSCS/SnO₂ composites had an average diameter of 860 nm, which is almost equal to that of MSCS (see Table 1). These results suggest that SnO₂ nanocrystals were precipitated only inside MSCS.

Figure 3(a) shows the nitrogen adsorption-desorption isotherms for MSCS and MSCS/SnO₂

composite. Table 1 lists the total volume, the specific surface area, and the averaged pore diameter calculated from the isotherms. MSCS consists of carbon nanorods with a starburst structure and nanopores between the carbon nanorods.⁽³⁰⁾ The average size of the nanopores was estimated to be 1.8 nm by the BJH method, which corresponds to the size of SnO₂ nanocrystals in the MSCS/SnO₂ composite estimated

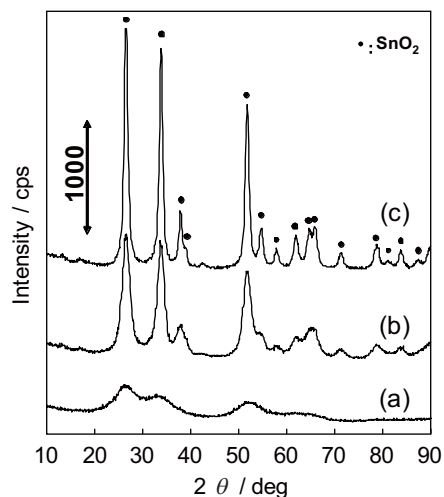


Fig. 2 XRD patterns of (a) MSCS/SnO₂ composite and MMTOS calcined at (b) 623 and (c) 823 K.

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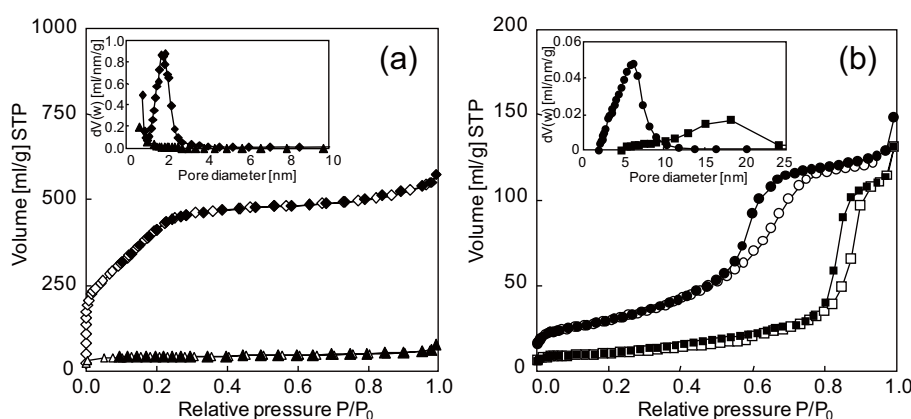


Fig. 3 Nitrogen isotherms of (a) MSCS (filled and open diamonds) and MSCS/SnO₂ composite (filled and open triangles) and (b) MMTOS calcined at 623 K (filled and open circles) and 823 K (filled and open squares). The insets show pore size distributions for these materials evaluated by the BJH method from the adsorption branch.

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from the XRD pattern. The total pore volume of the MSCS/SnO₂ composite was 0.087 mL/g, which is almost 10% of that of MSCS. No adsorption step due to homogenous nanopores in MSCS was observed. These results reveal that most mesospaces in the MSCS in the MSCS/SnO₂ composite were filled with SnO₂ nanoparticles.

3.3 Crystal growth of SnO₂ during calcination for removal of carbon

Figure 2 also shows XRD patterns for the MMTOS calcined at 623 and 823 K. XRD peaks for MMTOS became sharper with increasing calcination temperature. The crystallite sizes increased from 2.0 nm (for the MSCS/SnO₂ composite) to 4.7 and 10.2 nm on calcination at 623 and 823 K, respectively.

Figure 4 shows that nanoparticles smaller than 3 nm are highly dispersed in MSCS/SnO₂ composites, whereas larger nanocrystals aggregated to produce spherical MMTOS. The sizes of SnO₂ nanoparticle estimated by the TEM were 3-7 nm and 10-20 nm in the MMTOS calcined at 623 and 823 K, respectively. Calcination to remove the carbon nanorods and generate nanopores between SnO₂ nanoparticles caused the SnO₂ particles to aggregate and grow. The SnO₂ particle size is in reasonable agreement with the crystallite size of SnO₂ estimated from the XRD profile.

Figure 3(b) shows nitrogen adsorption-desorption isotherms for MMTOS calcined at 623 and 823 K. The inset shows the pore size distributions for these materials evaluated by the BJH method from the adsorption branch. Table 1 lists the total pore volume, the specific surface area, and the average pore diameter

of MMTOS. Both of the isotherms for MMTOS increase steeply due to capillary condensation of N₂ in the mesopores. The pore diameters of MMTOS calcined at 623 and 823 K were as large as 6.3 and 18 nm, respectively. The crystallite sizes of SnO₂ increased with increasing calcination temperature, resulting in larger pore diameters and lower specific surface areas. The mesopores of MMTOS are considered to be generated by the removal of carbon nanorods and subsequent aggregation of SnO₂ nanocrystals.

3.4 Particle size control of MMTOS

Using MMSS templates with various particle diameters enables us to control the particle sizes of MSCS and MMTOS via the nanocasting route. SEM images of pore-expanded MMSS, resultant MSCS, and MMTOS shown in Fig. 5 reveal that MMTOS with different particle diameters were obtained. Using our synthesis technique, it is possible to obtain MMTOS with any size between 120 and 1200 nm. This is also confirmed by high-resolution SEM observations, which reveal that MMTOS with a particle size of 320 nm consist of SnO₂ nanoparticles (see Fig. 6). Thus, our synthesis strategy can control the particle size and mesopores diameter of MMTOS. In future photonic applications, it will be important to control the size of MMTOS to tune the wavelength of the stop band of a MMTOS array.

3.5 Colloidal photonic crystal

One of the most important applications of monodispersed particles is the fabrication of colloidal

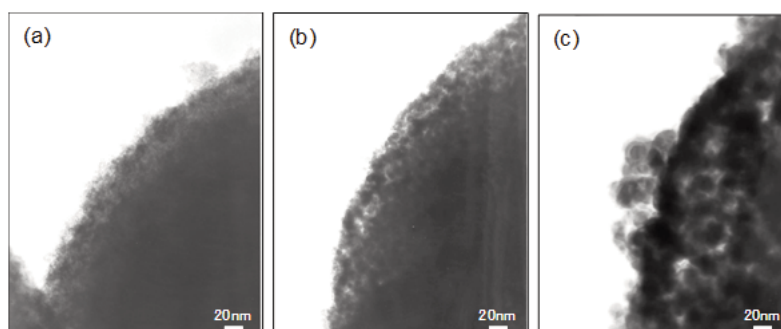


Fig. 4 TEM images of (a) MSCS/SnO₂ composite and MMTOS calcined at (b) 623 and (c) 823 K.

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photonic crystals for use in novel photonic devices. Colloidal photonic crystal have unique properties such as light confinement and reduction in the group velocity of light due to the stop band effect. Because of high mass density (7.1 g/cm^3) of tin oxide, it is more difficult to fabricate colloidal photonic crystals from

MMTOS than from polystyrene or silica colloidal particles. Therefore, MMTOS was coated with charged polymers to enhance the repulsive force between the particles prior to fabricating a colloidal photonic crystal. **Figure 7** shows a SEM image of a colloidal photonic crystal obtained from the polymer-coated

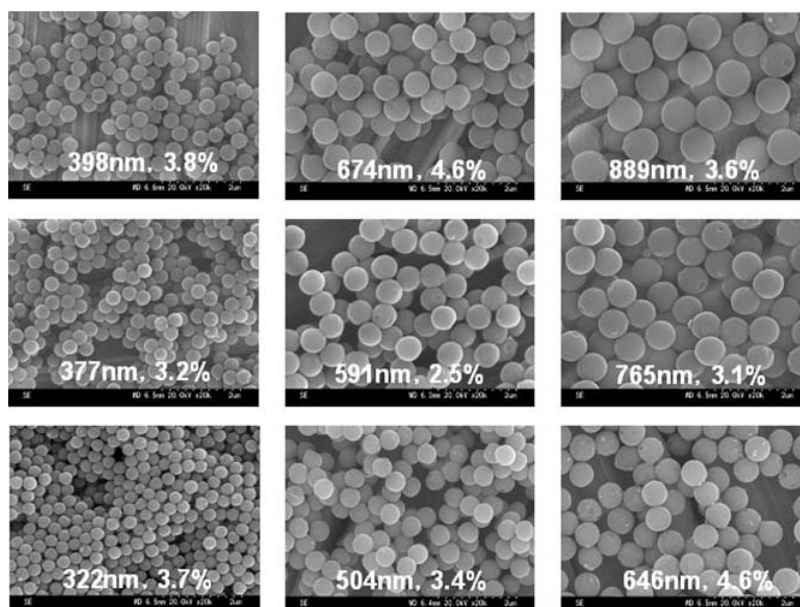


Fig. 5 SEM images of pore-expanded MMSS with different particle diameters (upper images), corresponding MSCSs (middle images) and MMTOSs (lower images). The average particle diameter and CV are given in each image.

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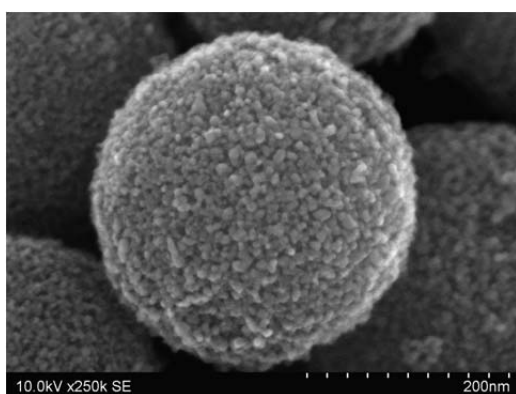


Fig. 6 High-resolution SEM image of MMTOS with a diameter of 320 nm. The image was obtained using a Hitachi S-5500N scanning electron microscope.

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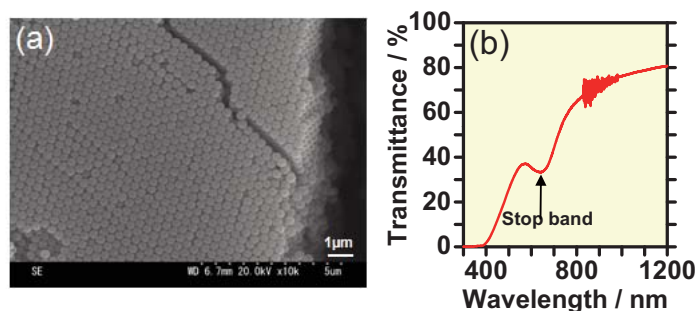


Fig. 7 (a) SEM image of a colloidal photonic crystal obtained from MMTOS with a diameter of 320 nm. (b) Transmission spectrum of colloidal photonic crystal showing a stop band around 650 nm.

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MMTOS and its visible transmission spectrum. A three-dimensionally close-packed opal structure was observed. In addition, the transmission spectrum showed a stop band around 650 nm from the colloidal photonic crystal. MMTOS has mesopores that can adsorb a variety of luminescent dyes, polymers, and nanoparticles. The wavelength of the stop band can be tuned to that of the luminescence of the incorporated materials just by changing the particle size. This raises the possibility of producing novel photonic device in the near future.

4. Summary

MMTOS have been obtained using MSCS as a host. The particle size and mesopore diameter of MMTOS can be tuned by varying the size of the parent MSCS and the calcination temperature. A colloidal photonic crystal with a stop band could be obtained from mesoporous tin oxide spheres. This new synthesis strategy is expected to be applied to other semiconductor materials such as titania to obtain interesting monodispersed mesoporous spheres and their colloidal opals. Incorporation of various dyes, polymers, nanoparticles into mesopores of MMTOS should enable novel photonic devices to be fabricated such as optically amplified solar cells or zero-threshold lasers by exploiting the unique properties of photonic crystals and the conductivity characteristics. We are currently investigating the effect of the stop band on the photocurrent in a dye-sensitized solar cell.

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