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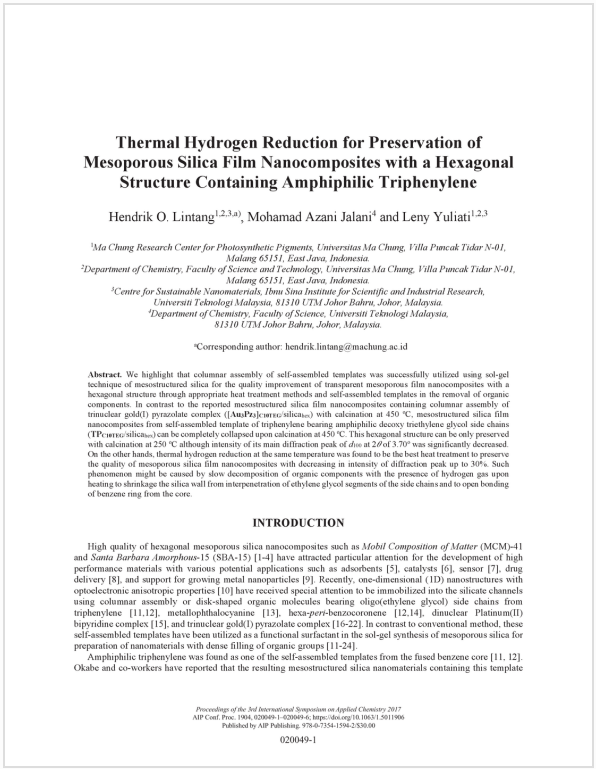
File size:	306.38K
Page count:	6
Word count:	3,160
Character count:	17,373
Submission date:	17-Jan-2018 01:09PM (UTC+0700)
Submission ID:	903507620



Thermal Hydrogen Reduction for Preservation of Mesoporous Silica Film Nanocomposites with a Hexagonal Structure Containing Amphiphilic Triphenylene

Hendrik O. Lintang[1,2,3,a)], Mohamad Azani Jalani[4] and Leny Yuliati[1,2,3]

[1]*Ma Chung Research Center for Photosynthetic Pigments, Universitas Ma Chung, Villa Puncak Tidar N-01, Malang 65151, East Java, Indonesia.*
[2]*Department of Chemistry, Faculty of Science and Technology, Universitas Ma Chung, Villa Puncak Tidar N-01, Malang 65151, East Java, Indonesia.*
[3]*Centre for Sustainable Nanomaterials, Ibnu Sina Institute for Scientific and Industrial Research, Universiti Teknologi Malaysia, 81310 UTM Johor Bahru, Johor, Malaysia.*
[4]*Department of Chemistry, Faculty of Science, Universiti Teknologi Malaysia, 81310 UTM Johor Bahru, Johor, Malaysia.*

[a]Corresponding author: hendrik.lintang@machung.ac.id

Abstract. We highlight that columnar assembly of self-assembled templates was successfully utilized using sol-gel technique of mesostructured silica for the quality improvement of transparent mesoporous film nanocomposites with a hexagonal structure through appropriate heat treatment methods and self-assembled templates in the removal of organic components. In contrast to the reported mesostructured silica film nanocomposites containing columnar assembly of trinuclear gold(I) pyrazolate complex ($[Au_3Pz_3]_{C8PEG}$/$silica_{hex}$) with calcination at 450 °C, mesostructured silica film nanocomposites from self-assembled template of triphenylene bearing amphiphilic decoxy triethylene glycol side chains (TP_{C8PEG}/$silica_{hex}$) can be completely collapsed upon calcination at 450 °C. This hexagonal structure can be only preserved with calcination at 250 °C although intensity of its main diffraction peak of d_{100} at 2θ of 3.70° was significantly decreased. On the other hands, thermal hydrogen reduction at the same temperature was found to be the best heat treatment to preserve the quality of mesoporous silica film nanocomposites with decreasing in intensity of diffraction peak up to 30%. Such phenomenon might be caused by slow decomposition of organic components with the presence of hydrogen gas upon heating to shrinkage the silica wall from interpenetration of ethylene glycol segments of the side chains and to open bonding of benzene ring from the core.

INTRODUCTION

High quality of hexagonal mesoporous silica nanocomposites such as *Mobil Composition of Matter* (MCM)-41 and *Santa Barbara Amorphous*-15 (SBA-15) [1-4] have attracted particular attention for the development of high performance materials with various potential applications such as adsorbents [5], catalysts [6], sensor [7], drug delivery [8], and support for growing metal nanoparticles [9]. Recently, one-dimensional (1D) nanostructures with optoelectronic anisotropic properties [10] have received special attention to be immobilized into the silicate channels using columnar assembly or disk-shaped organic molecules bearing oligo(ethylene glycol) side chains from triphenylene [11,12], metallophthalocyanine [13], hexa-*peri*-benzocoronene [12,14], dinuclear Platinum(II) bipyridine complex [15], and trinuclear gold(I) pyrazolate complex [16-22]. In contrast to conventional method, these self-assembled templates have been utilized as a functional surfactant in the sol-gel synthesis of mesoporous silica for preparation of nanomaterials with dense filling of organic groups [11-24].

Amphiphilic triphenylene was found as one of the self-assembled templates from the fused benzene core [11, 12]. Okabe and co-workers have reported that the resulting mesostructured silica nanomaterials containing this template

Proceedings of the 3rd International Symposium on Applied Chemistry 2017
AIP Conf. Proc. 1904, 020049-1–020049-6; https://doi.org/10.1063/1.5011906
Published by AIP Publishing. 978-0-7354-1594-2/$30.00

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Submission date: 17-Jan-2018 01:09PM (UTC+0700)
Submission ID: 903507620
File name: AIP_Conf._Proc._2017_1904,_020049-1_020049-6.pdf (306.38K)
Word count: 3160
Character count: 17373

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were reported to be collapsed in the calcination at 450 ºC for 3 hours if they did not use any acceptor molecules such as 7,7,8,8-tetracyanoquinodimethane (TCNQ), 2,4,7-trinitro-9-fluorenone (TNF), chloranil (CA), 2,3,6,7,10,11-hexacyano-hexaazatriphenylene (HAT), and 1,2,4,5-tetracyanobenzene (TCNB) for self-assembly with TP as a donor to form charge-transfer complexes in the sol-gel synthesis [11]. These acceptors compounds are generally expensive and less stable as well quite difficult to be found in the market or synthesized. However, there is no any report to synthesize the mesoporous nanocomposites with preservation of the original quality from columnar assembly of triphenylene in its mesostructured silicates with another method. Hence, this research aims to synthesize mesoporous silica nanocomposites containing columnar assembly of triphenylene for preservation of its original quality without using an acceptor molecule. Particularly, by using thermal hydrogen reduction at lower temperature (250 ºC), we have successfully preserved the quality of mesoporous silica film nanocomposites with less decreasing in its diffraction intensity. Such simple physical heat treatment even at low temperature was encouraged from our report in the synthesis of gold nanoparticles in the silicate nanochannels from immobilized columnar assembly of trinuclear gold(I) pyrazolate complex [22].

EXPERIMENTAL METHODS

General

Monitoring of chemical reactions was performed on thin-layer chromatography (TLC) using Merck pre-coated silica gel plates with thickness in 0.2 mm by spotting small amount of starting compounds and desired product. Mass spectrum was measured on AB Sciex MALDI-TOF/TOF™ 5800 spectrometry for the measurement of molecular weight and monoisotopic pattern with ionization in positive ion and reflection modes. Calcination was performed using Nabertherm model electronic muffle furnace, while thermal hydrogen reduction was performed using Carbolite model quartz tube furnace. X-ray diffraction (XRD) measurements with CuKα radiation (40 mA and 40 kV) were performed using Bruker D8 Advance diffractometer (a step size of 0.02º min^{-1} and scan speed of 2º min^{-1}). Ultraviolet-visible (UV-vis) spectra were measured on Thermo Scientific GENESYS 10S with scan speed of 100 nm min^{-1}.

Synthesis of Amphiphilic Triphenylene

Triphenylene bearing amphiphilic decoxy triethylene glycol side chains (**TP_{C10TEG}**) was prepared using Williamson ether substitution reaction in a round-bottom flask under an inert condition [11, 25] as shown in Figure 1. 2,3,6,7,10,11-hexahydroxytriphenylene (**TP_{OH}**) was bought from Tokyo Chemical Industry Co., Ltd. while amphiphilic decoxy triethylene glycol bromide (**C10TEGBr**) was prepared in three step stepwise reactions from triethylene glycol monomethyl ether. Typically, the mixture of **TP_{OH}** (80.00 mg, 0.25 mmol) and **C10TEGBr** (1.00 g, 2.61 mmol) in the presence of potassium carbonate (K_2CO_3; 600.00 mg, 4.35 mmol) was refluxed using N,N-dimethylformamide (DMF) in 10 mL under an inert condition for 66 hours at 90 ºC. After completion of the reaction as confirmed by checking using TLC, insoluble substances were filtrated from the reaction mixture. Under a reduced pressure, evaporation of the filtrate gave the desired product as a residue. Column chromatography was used for the purification consisting of silica gel with 80:1 of dichloromethane (CH_2Cl_2) to methanol (MeOH) as an eluent. The desired product was collected as purified fraction, evaporated and then dried under a reduced pressure. By using vacuum pump under reduced pressure, the final desired product as oily liquid was freeze-dried in benzene to give 298.00 mg (30% yield, 0.14 mmol) with a brownish color.

Sol-Gel Synthesis of Thin Mesostructured Silica Film Nanocomposites

Thin mesostructured silica film nanocomposites (**TP_{C10TEG}/$silica_{hex}$**) was prepared using sol-gel synthesis of **TP_{C10TEG}** in the acidic ethanolic solution without the presence of any acceptor [11]. In that reaction, ethanol (EtOH) in 0.01 μmol (595.60 mg) in an acidic solution consisting of 30.60 μmol of hydrochloric acid (HCl; 3.90 mg) in 0.26 μmol of water (H_2O; 45.40 mg) was used for self-assembly and oligomerization with 0.17 μmol of **TP_{C10TEG}** (36.30 mg) in the presence of 5.10 μmol of silica source (162.00 mg) from tetrabutyl orthosilicate (TBOS). The mixture was prepared and wrapped in small sample bottle at room temperature. The final composition is calculated to be 1: 30: 150: 770: 1.8 for [**TP_{C10TEG}**]/ [TBOS]/ [H_2O]/ [EtOH/ [HCl]. After 12 hours, a transparent thin film on top of a quartz substrate was fabricated from viscous solution of the mixture using spin-coater with a speed of 3000 rpm in 15 seconds. For enhancing the structure, as-synthesized thin film was dried at room temperature for 24 hours. For structural

analysis, heat treatments of TP_{C10TEG}/$silica_{hex}$ was performed using calcination at various temperatures from 250 until 450 °C and thermal hydrogen reduction at 250 °C in the presence of hydrogen gas.

RESULTS AND DISCUSSION

Amphiphilic TP_{C10TEG} was synthesized by reacting TP_{OH} and **C10TEGBr** under a basic condition (K_2CO_3) in DMF at 90 °C using Williamson ether substitution reaction for 66 hours as shown in Figure 1. Six hydroxyl groups of TP_{C10TEG} will be substituted with amphiphilic alkyl bromide. To confirm this reaction, the characterization using ^1H– and ^{13}C–NMR spectroscopy and MALDI-TOF/TOF spectrometry were reported to confirm the structure of TP_{C10TEG}. ^1H– and ^{13}C–NMR spectra were clearly confirmed the number of proton and carbon including their chemical shifts and integrations as reported previously [25]. Moreover, by using MALDI-TOF/TOF mass spectrometer, the mass spectrum showed the same monoisotopic pattern compared to the prediction one with a molecular formula of $C_{120}H_{217}O_{30}$ at 2137.5376 Dalton. The calculated molecular weight for the same molecular formula was 2137.2607 Dalton, which is very closed to the observed one. Therefore, this result indicates that TP_{C10TEG} as oily liquid was successfully synthesized with a brownish color in 30%. Of interest, the thin film on a quartz substrate showed optical properties with the presence of absorption peak as a broad spectrum from 200 to 270 nm, indicating π-π stacking from the self-assembly of TP_{C10TEG} molecules with a columnar structure from fused benzene core [25].

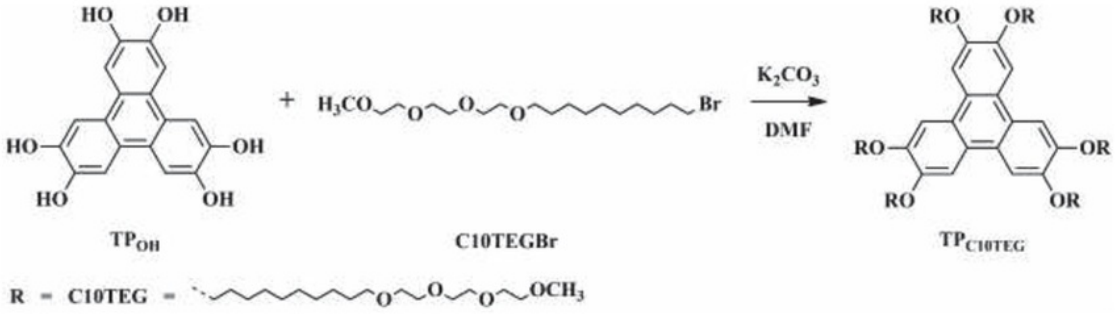

FIGURE 1. Synthesis and molecular structure of amphiphilic triphenylene bearing decoxy triethylene glycol (TP_{C10TEG}) from Williamson ether substitution reaction between 2,3,6,7,10,11-hexahydroxytriphenylene (TP_{OH}) and amphiphilic decoxy triethylene glycol bromide (**C10TEGBr**) as shown in reference [11].

Figure 2 shows XRD patterns for thin film TP_{C10TEG}/$silica_{hex}$ without and with thermal treatments. Before thermal treatments (Figure 2[A] and2[B]), XRD peak of spin-coated film (a) showed characteristic diffraction peaks (d) of d_{100} and d_{200} at 2θ of 2.70° and 5.25° for mesostructured silica nanocomposite with a hexagonal structure. By using the Bragg' law, d-spacing of TP_{C10TEG}/$silica_{hex}$ at 2θ of 2.70° was calculated to be 3.27 nm [11,16,25]. Moreover, the absence of d_{110} indicates that the orientation of c-axis for the hexagonal unit cell to the quartz surface is parallel for thin film sample as obtained from the fabrication. Such diffraction peaks with that geometry have generally found for mesostructured and mesoporous silica nanocomposites when the materials were fabricated as thin film onto any substrates.

Heat treatment for thin film samples can be easily and physically performed to clarify the formation of a hexagonal structure of mesostructured materials compared to other techniques using instrumentation such as transmission electron microscopy involving sample preparation using either focus-ion beam (FIB) or ultramicrotome or using chemical treatments such as extraction of template. After heat-treatment, if the materials can still preserved the diffraction peaks for hexagonal structure, the as-fabricated mesostructured materials have actually owned that geometry. On the other hands, disappearance of its diffraction peaks indicates that the as-fabricated mesostructured materials have a lamellar structure. For investigation of hexagonal structure in mesostructured silica TP_{C10TEG}/$silica_{hex}$, thermal treatment such as calcination at 450 °C (1 °C min^{-1} as the heating rate) for 3 hours was carried out. As reported by Okabe *et al.* [11], TP_{C10TEG}/$silica_{hex}$ without an acceptor molecule for the sol-gel synthesis with TP_{C10TEG} provided disruption of hexagonal structure up to 100% after calcination at 450 °C for 3 hours (Figure 2[A](f)). As shown in Figure 2[A] with variation calcination temperature from 400 (e), 350 (d) and 300 °C (c), this phenomenon was also

observed even the calcination temperature was reduced to 300 ºC with the same reaction time. Such results indicate that hexagonal structure in the as-synthesized mesostructured silica nanocomposite is not strong enough to preserve its own structure. Interestingly, when the calcination temperature was reduced to as low as 250 ºC (closed to temperature for decomposition of organic components in $\mathbf{TP_{C10TEG}}$) (Figure 2[A](b)), the hexagonal structure can be only preserved up to 20% (80% decreasing in intensity) from the calculation of the intensity ratio. Moreover, it gave shifting of d_{100} from 2θ of 2.70º with d-spacing of 3.27 nm to 2θ of 3.70º with d-spacing of 2.39 nm as shown in Table 1. Such low calcination temperature helps the mesostructured to preserve the hexagonal structure from heat treatment.

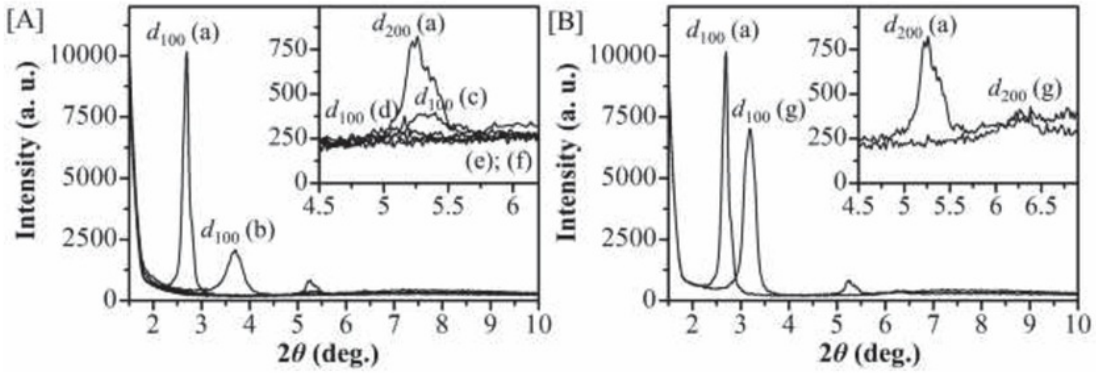

FIGURE 2. XRD diffractograms of $\mathbf{TP_{C10TEG}}$/silica$_{hex}$ [A] after calcination for 3 hours at (b) 250, (c) 300, (d) 350, (e) 400 and (f) 450 ºC as well as [B] thermal hydrogen reduction for 2 hours at (g) 250 ºC. XRD pattern of (a) $\mathbf{TP_{C10TEG}}$/silica$_{hex}$ before both thermal treatments in [A] and [B].

TABLE 1. Summary for d_{100} XRD peak position and intensity for $\mathbf{TP_{C10TEG}}$/silica$_{hex}$ thin film nanocomposite before and after both heat treatments

Label	Sample	d_{100} XRD Peak Position (deg.)	d_{100} XRD Peak Intensity (a. u.)
(a)	$\mathbf{TP_{C10TEG}}$/silica$_{hex}$ film before heat treatments	2.70	10174
(b)	$\mathbf{TP_{C10TEG}}$/silica$_{hex}$ film after calcination at 250 ºC	3.70	2070
(c)	$\mathbf{TP_{C10TEG}}$/silica$_{hex}$ film after calcination at 300 ºC	5.34	368
(d)	$\mathbf{TP_{C10TEG}}$/silica$_{hex}$ film after calcination at 350 ºC	- (sample decomposed)	-
(e)	$\mathbf{TP_{C10TEG}}$/silica$_{hex}$ film after calcination at 400 ºC	- (sample decomposed)	-
(f)	$\mathbf{TP_{C10TEG}}$/silica$_{hex}$ film after calcination at 450 ºC	- (sample decomposed)	-
(g)	$\mathbf{TP_{C10TEG}}$/silica$_{hex}$ film after thermal hydrogen reduction at 250 ºC	3.20	7017

Thermal hydrogen reduction can be also performed to evaluate the quality or preservation of mesostructured materials having particular structure by flowing hydrogen gas to quartz tube furnace containing thin film sample at the center during the heat treatment at certain temperature. In contrast, calcination method just used electronic muffle furnace at certain heating temperature. Interestingly, when thermal hydrogen reduction was performed at 250 ºC (1 ºC min^{-1} as the heating rate) for 2 hours as the best temperature for calcination method, the resulting mesoporous silica showed decreasing in intensity of d_{100} diffraction peak from 10174 to 7017 (in a. u.), which is up to 30% of its original height (Figure 2[B](g)). This result provided shifting of d-spacing from 3.27 to 3.20 nm (Tabel 1). Moreover,

calculation of the reduction of *d*-spacing for both methods at 250 °C indicates that decreasing of *d*-spacing for calcination method (0.88 nm) was higher than that of thermal hydrogen reduction (0.51 nm). Hence, decomposition of organic components in the silicate nanochannels with thermal hydrogen reduction was slowly occurred with the presence of hydrogen gas upon heating so that shrinkage of the silica wall from interpenetration of ethylene glycol segments of the side chains and ring opening of bonding of benzene ring from the core of **TP** were coincidently took place without significantly disruption of a hexagonal structure.

CONCLUSIONS

Thin mesostructured silica film nanocomposite with a hexagonal structure was successfully fabricated as a transparent film onto a quartz glass using sol-gel synthesis of columnar assembled triphenylene ($\mathbf{TP}_{\mathbf{C10TEG}}$) as a template. The hexagonal structure can be easily preserved and confirmed from the as-synthesized by using thermal hydrogen reduction at 250 °C with small reduction in *d*-spacing compared to the calcination method at the same temperature. This result indicates the slow decomposition and ring opening of the organic components from $\mathbf{TP}_{\mathbf{C10TEG}}$ molecules inside the silicate nanochannels for the preservation of a hexagonal structure. Such simple method with physical treatment may shed light on the method for confirmation of particular nanostructure from thin film sample without using chemical treatment or modification in the molecular level.

ACKNOWLEDGMENTS

The authors would like to thank to Ministry of Higher Education (MOHE), Malaysia through Universiti Teknologi Malaysia (UTM) for the financial using Research University Grant Scheme (RUG) with type of Tier 1 (Q.J130000.2526.10H11). The authors would also like to appreciate the contribution of Centre for Sustainable Nanomaterials (CSNano), UTM for providing instrumentations in the characterization. Particularly, H. O. Lintang would like to thank to Ma Chung Research Center for Photosynthetic Pigments, Universitas Ma Chung, Indonesia for the additional financial support.

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