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Fabrication of Mesoporous Silica/Alumina Hybrid Membrane Film Nanocomposites using Template Sol-Gel Synthesis of Amphiphilic Triphenylene

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Abstract. Herein we reported that by introducing a one-dimensional (1D) substrate with a porous structure such as anodic aluminum oxide (AAO) membrane, mesoporous silica/alumina hybrid nanocomposites were successfully fabricated by using amphiphilic triphenylene (TP_{C₁₁H₇}) as a template in sol-gel synthesis (TP_{C₁₁H₇}/silica₆₀). For the optical study of the nanocomposites, TP_{C₁₁H₇}/silica₆₀ showed absorption peak at 264 nm due to the ordered and long-range π - π stacking of the disk-like aromatic triphenylene core. Moreover, the hexagonal arrangement of TP_{C₁₁H₇}/silica₆₀ was proven based on their diffraction peaks of d_{100} and d_{010} at $2\theta = 2.52^\circ$ and 5.04° and images of transmission electron microscopy (TEM), respectively. For fabrication of mesoporous silica/alumina hybrid membrane, TP_{C₁₁H₇}/silica₆₀ was drop-casted onto AAO membrane for penetration into the porous structure via gravity. X-ray diffraction (XRD) analysis on the resulted hybrid nanocomposites showed that the diffraction peaks of d_{100} and d_{010} of TP_{C₁₁H₇}/silica₆₀ were still preserved, indicating that the hexagonal arrangements of mesoporous silica were maintained even on AAO substrate. The morphology study on the hybrid nanocomposites using TEM, scanning electron microscope (SEM) and field emission scanning electron microscope (FE-SEM) showed the successful filling of most AAO channels with the TP_{C₁₁H₇}/silica₆₀ nanocomposites.

Keywords: silica, alumina hybrid thin film, nanocomposite membrane, sol-gel, amphiphilic triphenylene.

1. Introduction

Ordered mesoporous silica (OMS) materials have gained much attention for the last few decades. In the early 1990s, Kuroda and his co-workers pioneered the synthesis of mesoporous silicate called FSM (Folded Sheet Materials)-16 [1] and then followed by the report of the mesoporous M41S family such as MCM (Mobil Composition of Matter)-41 by Kresge *et al.* in 1992 [2]. In particular, mesoporous silica is an ordered and well-defined pore structure with large surface areas, good thermal stability, and narrow pore size distribution [3]. Generally, mesoporous silica has been synthesized through silica

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1. Introduction

Ordered mesoporous silica (OMS) materials have gained much attention for the last few decades. In the early 1990s, Kuroda and his co-workers pioneered the synthesis of mesoporous silicate called FSM (Folded Sheet Materials)-16 [1] and then followed by the report of the mesoporous M41S family such as MCM (Mobil Composition of Matter)-41 by Kremer *et al.* in 1992 [2]. In particular, mesoporous silica is an ordered and well-defined pore structure with large surface areas, good thermal stability, and narrow pore size distribution [3]. Generally, mesoporous silica has been synthesized through silica

formation catalyzed by either acid or base medium with the presence of amphiphilic template surfactants. Since most of OMS materials were produced in the powdered form [4, 5], it is interesting to investigate the properties of mesoporous silica synthesized as thin film nanocomposite membranes.

Recently, there are many works reported on the synthesis of thin film nanocomposites by utilizing the columnar assembly of functional amphiphilic organic moieties in the channels of mesoporous silica [6-14]. Also, in 2002 Okabe *et al.* showed that amphiphilic triphenylene derivatives could be used as a self-assembled template to synthesize mesostructured silica films [15]. However, their silica films depend on the donor-acceptor system and ethanol vapour-mediated maturing for the synthesis of nanocomposites. Theoretically, triphenylene is a disc-shaped molecule of a flat polycyclic aromatic hydrocarbon (PAH) consisting of four fused benzene rings with a planar structure and three-fold rotation axes. Moreover, triphenylene derivatives possessed graphite-like conducting pathway [16] that ideal to be used in energy devices like battery due to its good ionic conductivity properties. Nevertheless, previous studies reported that as thin films, the formed mesostructured silica tends to have channel direction oriented in parallel to the substrate [17] and this phenomenon is not optimal for energy storage purposes. Xie *et al.* stated that a one-dimensional (1D) arrangement is crucial in energy conversion and storage as it can help to minimize the path of carrier transport, increase the carrier collection ability, and act as an electron expressway in the axial axis [18]. Thus, the development of mesoporous silica thin film with 1D alignment is really important, and the formed silica nanochannels must be arranged perpendicularly to the substrate. 16

Anodized aluminum oxide (AAO) membrane was used as a template for controlling the perpendicular orientation of mesoporous silica to the substrate [19-23]. AAO was made up of porous cylindrical channels with ordered and vertical 1D arrangement [24, 25]. In 2003, the first work of filling the mesoporous silica inside the AAO was done by Yang *et al.* [26], followed by another outstanding work by Teramae *et al.* in 2004 [21]. However, in their work, they used the commercially available surfactants such as Pluronic F-127 ($\text{EO}_{106}\text{PO}_{70}\text{EO}_{106}$) and cetyltrimethylammonium bromide (CTAB) as the template for synthesis of mesoporous silica. Therefore, in our work, we have used stepwise-synthesized useful surfactants, which is amphiphilic triphenylene ($\text{TP}_{\text{C}_{10}\text{TEG}}$) as a template in the sol-gel synthesis of mesoporous silica ($\text{TP}_{\text{C}_{10}\text{TEG}}/\text{silica}_{\text{hex}}$) in the AAO. Generally, triphenylene derivatives were mainly applied for molecular electronics, field-effect transistors, and photovoltaic due to its excellent self-healing property as well as giving the anisotropic columnar assembly of liquid crystalline phase to produce 1D or charge and energy migration materials [16, 27]. However, although triphenylene derivatives have an excellent ionic conductivity, not many works have been done for investigating the potential of this nanomaterial as energy devices. Thus, the interesting utilization of this triphenylene derivative for potential application in energy aspects that mainly require fast charge transfer and / or transport that may be achieved when using triphenylene derivative. Hence, this report was done based on the idea of synthesizing a hybrid nanomaterial that has the potential to be used in energy conversion or storage. Likewise, sol-gel synthesis was utilized in this study due to its simple and easy method of synthesizing hybrid of mesoporous silica/alumina membrane nanocomposites consisting of organic functional groups with dense filling in the nanochannels [28].

2. Experimental Methods

13 General

Proton and carbon nuclear magnetic resonance (^1H - and ^{13}C -NMR) spectra were performed by a Bruker AVANCE 300 MHz NMR spectroscopy. The chemical shifts (δ) were determined using deuterated chloroform (CDCl_3) at 7.24 and 77.0 ppm for ^1H - and ^{13}C -NMR as internal standards. XRD measurements at room temperature were carried out using D8 Advance diffractometer from Bruker with $\text{CuK}\alpha$ radiation at 40 mA and 40 kV with a step size and scan speed of 0.02° and 2°min^{-1} . Thermo Scientific GENESYS 10S with 100 nm min^{-1} as a scan speed was used as an ultraviolet-visible (UV-vis) spectroscopy. For monitoring chemical reactions, thin-layer chromatography (TLC) was used on pre-coated silica gel plates of 0.2 mm in thickness from Merck. AB Sciex MALDI-TOF/TOF™

5800 spectrometry was used for the measurement of mass spectra on reflection mode with ionization in ion positive mode. For crystallization, a matrix such as dithranol (1, 8, 9- antracenetriol) in chloroform was spotted onto a conductive plate containing the samples. The AAO membrane used was commercially purchased Whatman Anodisc 25 with 25 mm in diameter and 0.2 μm pore size. JEOL JEM-2100, JSM-6390LV, and JSM-6701F microscopies were used to visualize TEM, SEM and FE-SEM images at 200, 15 and 5 kV, respectively.

2.2 Synthesis of $TP_{C_{10}TEG}$

According to Okabe *et al.* [15], $TP_{C_{10}TEG}$ was prepared by reacting hydroxy type of triphenylene and amphiphilic alkyl bromide. Typically, in dry N,N-dimethylformamide solution (10 mL), the mixture of 80.0 mg for 2,3,6,7,10,11-hexahydroxytriphenylene (0.247 mmol) and 1.0 g for 1-(2-(2-(2-methoxyethoxy)ethoxy)ethoxy)-10-bromodecane ($C_{10}TEG\text{Br}$; 2.61 mmol) with the presence of 600.0 mg of potassium carbonate (4.7 mmol) was refluxed at 90 $^{\circ}\text{C}$ for 66 hours under an inert condition. Insoluble substances from the reaction mixture were filtered off, and then the filtrate was evaporated under a reduced pressure to give residue as the desired product. Silica column chromatography in dichloromethane (CH_2Cl_2) and methanol (MeOH) with a ratio of 80:1 was used as an eluent for the purification procedure. The collected fraction as the desired product was evaporated until all solvents were completely removed. This desired product was dried under a reduced pressure and followed by freeze-drying in benzene until reached a formation of dried oily liquid with a brownish color with 30% yield (298.0 mg, 0.139 mmol).

2.3 Sol-gel synthesis of $TP_{C_{10}TEG}/\text{silica}_{\text{hex}}$

For confinement of 1D columnar assembly of $TP_{C_{10}TEG}$ into the nanoscopic channels of mesoporous silica, the sol-gel synthesis was performed under an acidic solution of aqueous ethanol (EtOH, 595.6 mg, 0.01 μmol ; HCl, 3.9 mg, 30.60 μmol ; H_2O , 45.4 mg, 0.26 μmol) for a mixture of $TP_{C_{10}TEG}$ (36.3 mg, 0.17 μmol) and tetrabutoxysilane (TBOS, 162.0 mg, 5.10 μmol). This sol-gel reaction with TBOS was carried out for partial oligomerization in 12 hours at room temperature with mole ratios of $TP_{C_{10}TEG}/[\text{TBOS}]/[\text{H}_2\text{O}]/[\text{EtOH}]/[\text{HCl}]$ in 1:30:150:770:1.8. The mixture was completely wrapped with aluminum foil to avoid the interaction of the light. Spin-coater was then used to make a thin film on a quartz plate from the viscous solution with a speed of 3000 rpm for 15 s. The thin film provided colorless transparent material and was then dried for 24 h at room temperature. For the synthesis of $TP_{C_{10}TEG}/\text{silica}_{\text{hex}}$ inside channels of AAO, the sol-gel solution was drop-casted onto the AAO membrane surface and allowed gravity to flow through the channels of the membrane for the formation of mesoporous nanocomposites. The resulting thin film was dried in open air for 24 h at room temperature.

3. Results and discussion

3.1 Structure elucidation of $TP_{C_{10}TEG}$

From Williamson ether substitution reaction of triphenylene and amphiphilic $C_{10}TEG\text{Br}$, structure elucidation of the desired product for purified $TP_{C_{10}TEG}$ showed $^1\text{H-NMR}$ spectrum with δ_{H} as illustrated in Figure 1(a) with special labels for the presence of protons according to their environments (Ha-i). Particularly, it gave δ at 7.86 (Hi, singlet, 5.61H), 4.19 (Hh, triplet, 11.50H), 3.64–3.51 (Hb = $\text{CH}_2\text{CH}_2\text{OCH}_2\text{CH}_2\text{OCH}_2\text{CH}_2$, multiplet, 72.43H), 3.42 (Hc = OCH_2 , triplet, 12.42H), 3.35 (Ha = OCH_3 , s, 18.00H), 1.91 (Hg = CH_2 , triplet, 2.15H), 1.56 (Hd = CH_2 , singlet, 5.62H) and 1.40–1.28 ppm (He = CH_2 , Hf = $(\text{CH}_2)_4$, overlapped, 12.12H). For $^{13}\text{C-NMR}$, the spectrum gave δ_{C} as shown in Figure 1 (b) for the presence of carbon according to their environments (Ca-m) at 148.88 (Ck), 123.52 (Cm), 107.28 (Cl), 71.54 (Cb), 71.15 (Cd), 70.42 (Cc), 69.96 (Ce), 69.63 (Cj), 58.94 (Ca), 29.56–29.46 (Cf, Cg, Ch) and 26.12 ppm (Ci). Moreover, the mass spectrum of purified $TP_{C_{10}TEG}$ observed a monoisotopic peak at 2137.2607 Dalton for $[\text{M}]^+$ where it was almost similar to the calculated value for a monoisotopic peak with a molecular formula of $\text{C}_{120}\text{H}_{217}\text{O}_{30}$ at 2137.5376

4 a. This results indicated that amphiphilic version of TP_{C10TEG} was successfully synthesized and then can be used as a template for the sol-gel synthesis of mesoporous silica nanocomposites containing columnar assembled in the nanoscopic channels.

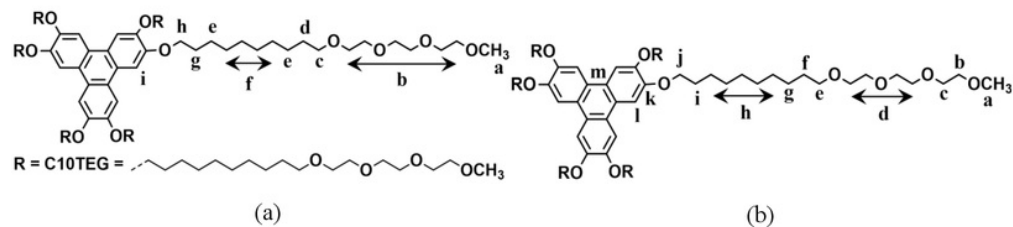


Figure 1. Representative molecular structures of TP_{C10TEG} for (a) ¹H- and (b) ¹³C-NMR spectra

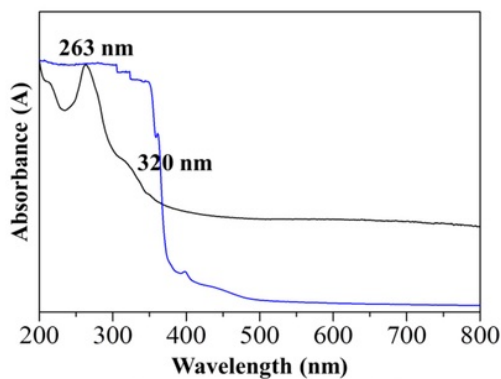


Figure 2. UV-vis spectra of the bulk TP_{C10TEG} (blue line) and TP_{C10TEG}/silica_{hex} fabricated onto quartz substrate (black line)

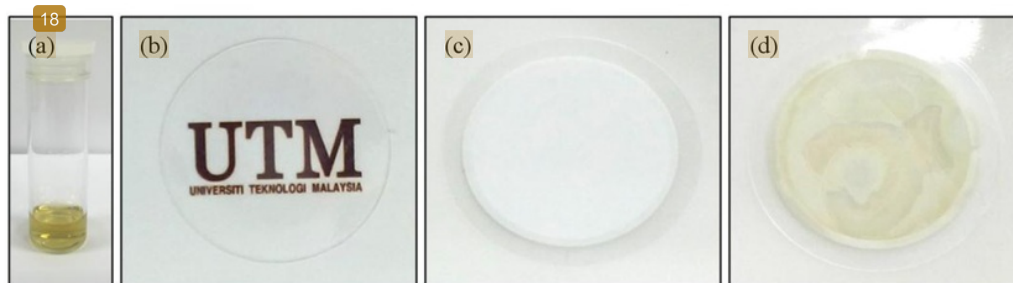


Figure 3. Photograph images of (a) sol-gel solution of TP_{C10TEG}/silica_{hex}, (b) transparent thin film of TP_{C10TEG}/silica_{hex} fabricated onto quartz substrate, (c) blank AAO membrane and (d) TP_{C10TEG}/silica_{hex} after drop-casted onto AAO membrane via a gravitational filtration

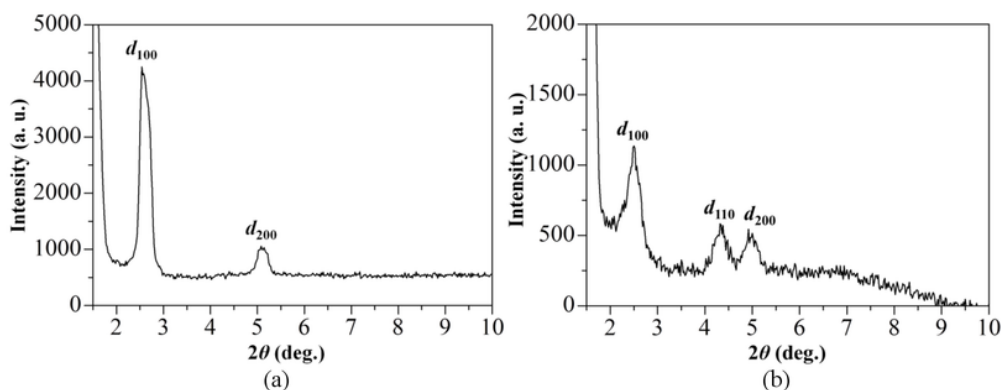


Figure 4. Diffraction peaks for TP_{C10TEG}/silica_{hex} after (a) spin-coated on a quartz substrate and (b) drop-casted inside AAO channels via gravitational filtration

3.2 Optical properties of TP_{C10TEG}/silica_{hex}

For identification of appearance of polycyclic aromatic hydrocarbons, a study on the absorption of organic chromophores can be evaluated using UV-vis spectroscopy [29]. Figure 2 shows the UV-vis spectrum of bulk TP_{C10TEG} (blue line) and TP_{C10TEG}/silica_{hex} that was spin-coated on a quartz substrate (black line), where this film was used as a reference throughout this study. In this figure, bulk TP_{C10TEG} showed a broad peak from 200 to 270 nm that maybe due to a π - π stacking between triphenylene molecules. Interestingly, TP_{C10TEG}/silica_{hex} showed a strong absorption peak of 264 nm that may due to the ordered and long-range π - π stacking of disc-like aromatic triphenylene core from their self-assembly in the silicate nanochannels, along with a shoulder at 320 nm for the charge-transfer of the organic molecules. Figure 3 (a) shows the photograph images of a sol-gel solution of TP_{C10TEG}/silica_{hex} that has a yellow-brownish color. After the sol-gel solution was spin-coated by a quartz substrate, it gave a colorless thin film as shown in Figure 3 (b). Moreover, the sol-gel solution was also fabricated onto the blank AAO membrane as in Figure 3 (c) to produce a light brownish hybrid AAO membrane as observed in Figure 3 (d). Although, it is really difficult to check the absorption peak of TP_{C10TEG}/silica_{hex} after being fabricated inside the AAO channels due to the opaqueness of the hybrid AAO nanocomposites, the images in Figure 3 (d) and its UV-vis spectrum provided a strong confirmation for the formation of columnar assembly with well self-organization inside the nanostructured materials from oligomerization of silica source.

3.3 Structural Analysis of TP_{C10TEG}/silica_{hex}

The structural analysis was done on fabricated TP_{C10TEG}/silica_{hex} nanocomposites and AAO substrate using XRD [15]. After fabrication onto a quartz substrate with a slow oligomerization of TBOS as a silica source, diffraction peaks due to the formation of a hexagonal structure for mesoporous silica at $2\theta = 2.59^\circ$ (d_{100}) and 5.08° (d_{200}) were observed as shown in Figure 4 (a). This diffractogram gave d -spacing value of 3.4 nm which was almost closed to the reported one [15]. After the fabrication of TP_{C10TEG}/silica_{hex} inside the AAO membrane (Figure 4 (b)), an additional d_{110} diffraction peak at $2\theta = 4.40^\circ$ was observed and this peak was not present in the fabricated one using quartz. The absence of the d_{110} was usually observed from the c -axis of a hexagonal unit cell that was oriented in parallel to the quartz [8]. Interestingly, although the intensity of the diffraction peaks after fabricated on the AAO membrane was slightly lower compared to the fabricated one onto a quartz substrate, the diffraction patterns for the typical hexagonal structure of mesoporous silica TP_{C10TEG}/silica_{hex} were still preserved. Moreover, it was suggested that the lower intensity might be highly possible for the formation of most hexagonal channels of mesoporous silica that were already inside the AAO pores.

3.4 Morphology Studies of $TP_{C10TEG}/silica_{hex}$

The XRD results were also supported by morphology studies using the TEM images in Figure 5 (a) and (b). The TEM images showed the presence of mesoporous silica materials with a hexagonal structure containing pore sizes ranging from 2.0 to 2.5 nm after fabricated on both a quartz substrate and an AAO membrane.

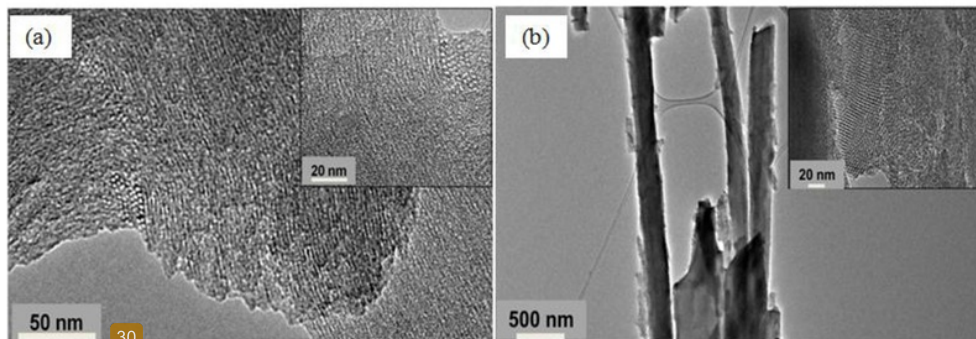


Figure 5. TEM images of (a) $TP_{C10TEG}/silica_{hex}$ and (b) $TP_{C10TEG}/silica_{hex}$ inside AAO channels. The inset Figures are to magnify the image with a scale bar of 20 nm

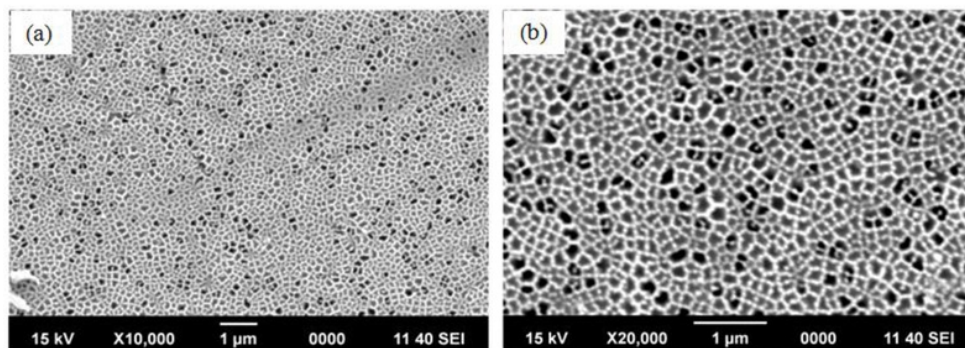


Figure 6. SEM images of $TP_{C10TEG}/silica_{hex}$ after fabricated inside AAO channels via gravitational filtration at (a) 10K and (b) 20K times magnification at 1 μ m scale

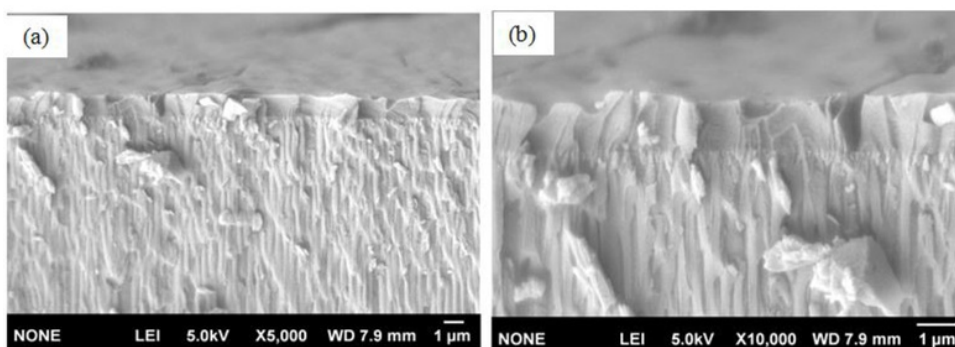


Figure 7. FE-SEM images of $TP_{C10TEG}/silica_{hex}$ after fabricated inside AAO channels via gravitational filtration at (a) 5K and (b) 10K times magnification at 1 μ m scale

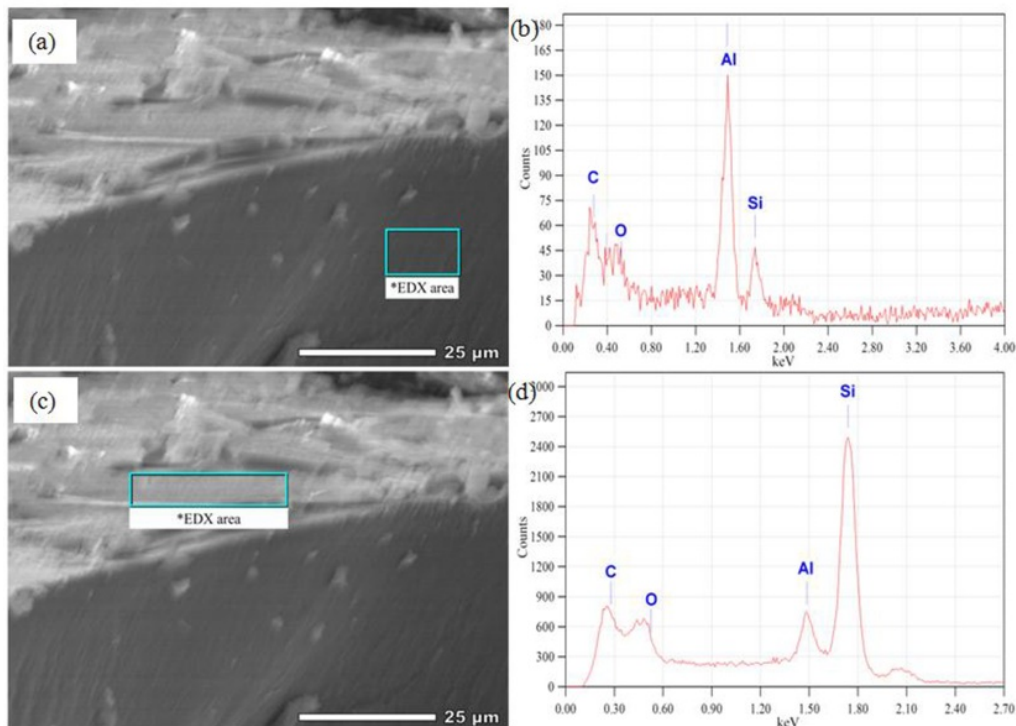


Figure 8. FE-SEM images of TP_{C10TEG}/silica_{hex} after fabricated inside AAO channels showing the selected area indicated by * for EDX analysis in (a) the middle of the membrane with (b) its respective EDX and (c) the top area of the membrane with (d) its corresponding EDX

Based on SEM images in Figure 6 (a) and (b), it showed the successful filling of most AAO channels with the TP_{C10TEG}/silica_{hex} composites, with the diameter of AAO channels ranging from 200 to 300 nm. These images also suggest that many TP_{C10TEG}/silica_{hex} composites have been successfully penetrated to the AAO channels without a structure collapse. Such filling of the template for mesoporous silica was in good agreement with their X-ray diffractogram and TEM images. Hence, the 1D arrangement of TP_{C10TEG} silica nanocomposites in the AAO membrane film was achieved via a simple template sol-gel synthesis and a gravitational filtration.

By characterization of the mesoporous silica/alumina hybrid membrane film using the cross-section of FE-SEM as shown in Figure 7 (a) and (b), it was confirmed that mesoporous silica not only formed inside the AAO channels but also on top of AAO membrane. Energy-dispersive X-ray (EDX) analysis suggests the existence of Al, Si, C, and O elements inside AAO of the TP_{C10TEG}/silica_{hex}. Upon characterizing the area of the nanocomposites from Figure 8 (a), the Si element can be found along with Al as shown in Figure 8 (b), indicating that mesoporous silica nanocomposites were present inside the AAO pores. Further EDX analysis on the top layer as shown in Figure 8 (c) and (d), Si ratio was found to be higher than Al. This result indicated that the formation of mesoporous silica would cover the pores on top of the AAO membrane. Therefore, in order to prevent any formation of mesoporous silica on top of the membrane, Teramae *et al.* [17] proposed the usage of aspiration technique while doing the filtration other than using gravity. However, this method might be useful for silica source with faster oligomerization rate such as tetramethyl orthosilicate (TMOS).

4. Conclusion

Triphenylene bearing a C₁₀TEG as the amphiphilic chains (TP_{C₁₀TEG}) in the form of light yellow oily liquid was successfully prepared in 30 % yield (298.0 mg, 0.139 mmol). The resulting TP_{C₁₀TEG} was used in the sol-gel synthesis as a template to form TP_{C₁₀TEG}/silica_{hex} that later was fabricated on a quartz substrate and inside the AAO membrane. After the fabrication of TP_{C₁₀TEG}/silica_{hex} onto a quartz substrate, the diffraction peaks due to a hexagonal structure of mesoporous silica nanocomposites were observed and it was in good agreement with their TEM images. Upon characterization of TP_{C₁₀TEG}/silica_{hex} inserted into the AAO channels using XRD, it was confirmed that the diffraction patterns for hexagonal channels of mesoporous silica were still preserved, accompanied by the presence of additional diffraction peak of d_{110} at $2\theta = 4.40^\circ$. Based on the SEM, FE-SEM and EDX analysis, it was confirmed that 1D arrangement of mesoporous silica nanocomposites were achieved as the TP_{C₁₀TEG}/silica_{hex} grew one-dimensionally inside the porous AAO with a perpendicular orientation to the AAO substrate.

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9

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