



Review

Luminescent Periodic Mesoporous Organosilicas

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■ABSTRACT■ Progress in the development of periodic mesoporous organosilicas (PMOs) bearing luminescent organic groups is reviewed and directions for future research are highlighted. The inclusion of luminescent organic groups in the PMO framework allows for the preparation of materials with dissimilar luminescent groups in two spatially separated regions; the framework and the mesopore channels. Mesoporous silicas, in contrast, only bear luminescent dyes in the channels of the material structure. In the multi-dye system, the transfer of excitation energy from the framework donors to acceptors in the mesochannels is observed. Certain PMOs, such as biphenyl PMO, exhibit efficient light absorption due to the dense packing of chromophores in the framework, and high luminescence quantum yields comparable, demonstrating the potential of luminescent PMOs as a lighting technology. Fluorescence studies have revealed that the interaction among bridging organic groups in PMOs differs between crystal-like and amorphous frameworks. These recent developments highlight the potential of luminescent PMOs as a new technology, which should be supported by further investigation of the optical properties and functionalization of PMOs.

■KEYWORDS■ Periodic mesoporous organosilicas, Organic chromophores, Optical properties, Photoluminescence, Fluorescence quantum yield, Molecular design

1. Introduction

Ordered mesoporous silicas are unique materials with high surface area and uniform pore size, and have attracted much attention for potential applications in catalysts and as adsorbents. Since the pioneering works of Beck et al.⁽¹⁾ and Inagaki et al.,⁽²⁾ many studies have been conducted in attempts to functionalize these materials by incorporating functional materials such as clusters, nanowires, and metal complexes into the mesochannels, by grafting organic functional groups onto the frameworks and by co-condensation with monosilylated functional molecules.⁽³⁾ Periodic mesoporous organosilicas (PMOs), synthesized from 100% or less organic-bridged alkoxysilane precursors ($R-[Si(OR')_3]_n$; $n \geq 2$, R: bridging organic groups, R': CH_3 , C_2H_5 etc.),⁽⁴⁾ are a new class of functional materials having organic-inorganic hybrid frameworks and ordered mesopores.^(3b,5-8) A broad spectrum of functionalities can be introduced into these frameworks by designing organosilane precursors with specific bridging organic groups (R). A large variety of PMOs bearing organic groups (e.g., ethane,⁽⁵⁾ benzene,⁽⁹⁾

biphenyl,⁽¹⁰⁾ thiophene,⁽¹¹⁾ divinylbenzene,⁽¹²⁾ diacetylene⁽¹³⁾ and carbazole⁽¹⁴⁾) have been reported. Some PMOs with interactive organic groups such as benzene,⁽⁹⁾ biphenyl,⁽¹⁰⁾ and divinylbenzene⁽¹²⁾ have been found to exhibit crystal-like molecular-scale periodicity of the bridging organic groups in the framework. There have also been many attempts at further chemical modification of the framework organic groups using functional organics such as sulfonic⁽¹⁵⁾ and amine⁽¹⁶⁾ groups as a means of introducing catalysis and/or adsorption sites. The dielectric⁽¹⁷⁾ and chromatographic⁽¹⁸⁾ properties of PMOs have also been investigated.

The incorporation of organic chromophores in mesostructured and mesoporous materials is an attractive approach to functionalization, as the chromophores can be dispersed at high concentrations in the high-surface-area mesochannels without aggregation.⁽¹⁹⁾ In surfactant-templated mesostructured silica films, for example, dye molecules can be dispersed at high concentrations in the hydrophobic core or at the hydrophilic interface of the surfactant micelles, resulting in strong luminescence with less concentration quenching. Yang et al. reported amplified spontaneous emission at a low pumping threshold for mesostructured silica waveguide arrays in which the mesochannels were doped with large amounts of laser

dyes.⁽²⁰⁾ Mesoporous silicas in which the frameworks bear covalently attached chromophores have been studied extensively, mainly in application to optical sensors, as these materials are not subject to chromophore leaching, allow efficient diffusion of target molecules or ions in the mesochannels, and provide sensitive quenching of luminescence from the chromophores.⁽¹⁹⁾

The placement of chromophores within the PMO framework could be further advantageous in that such “luminescent PMOs” would allow chromophores to be simultaneously located in two spatially separated regions (the frameworks and mesochannels), potentially promoting excitation energy transfer from the framework donors to acceptors in the mesochannels.⁽²¹⁾ Such a scheme has great potential for the enhancement of luminescence from chromophores in the mesochannels. For application in optical sensors, target molecules or ions can be diffused more efficiently in PMOs than in chromophore-attached mesoporous silicas due to relatively low steric hindrance to the diffusion of targets in the mesochannels, which results in fast sensing response. In addition, chromophores can be packed densely in the PMO framework, enhancing light absorption and thus luminescence intensity.⁽²²⁾ Hence, it is desirable to synthesize PMOs having framework-contained chromophores and to understand the optical properties of these materials.

In this highlight, the advances in two approaches to the synthesis of luminescent PMOs are reviewed. The two approaches are preparation by co-condensation of chromophores having alkoxy-silyl groups and a large amount of the framework precursor (e.g., tetraethoxysilane); and preparation from 100% chromophore-bridged organosilane precursors (**Fig. 1**). The unique fluorescence properties of naphthalene-bridged PMOs and material designs for efficiently visible-light luminescent PMOs are also demonstrated.

2. Luminescent PMOs prepared by co-condensation

The preparation of luminescent PMOs by co-condensation aims to introduce luminescent properties into the inorganic or organic-inorganic hybrid framework itself. Minoofar et al. synthesized PMO films by co-condensation of ruthenium and/or europium complexes attached to 6 alkoxy-silyl groups and tetraethoxysilane (TEOS; > 98 mol%). The PMO films were doped with various chromophores (e.g., pyrene and naphthoquinone), and the luminescence

properties of the metal complexes in the framework and the chromophores in the mesopores were evaluated.⁽²³⁾ Minoofar et al. also prepared PMO films bearing a terbium complex in the framework and rhodamine 6G dye in the mesochannels, and successfully observed excitation energy transfer from the terbium complex to the dye.⁽²¹⁾ PMO films synthesized by co-condensation of 9,10-diarylanthracene-bridged organosilane and TEOS (ca. 95 mol%) were found by Alvaro et al. to exhibit significantly enhanced electrochemiluminescence compared to the corresponding non-porous organosilica film due to efficient charge transport to the organic units in the mesostructured film.⁽²⁴⁾ Wahab et al. obtained luminescent PMO films by co-condensation of 1,4-bis(diphenylamino)biphenyl-bridged organosilane and 1,2-bis(triethoxysilyl)ethane (ca. 88 mol%), and found that aggregation of the chromophores in the PMO films was suppressed compared to the corresponding non-porous organosilica film.⁽²⁵⁾ There have also been reports on the synthesis of PMOs containing organic chromophores such as viologen,⁽²⁶⁾ bispyridylethylene,⁽²⁷⁾ triphenylpyrylium⁽²⁸⁾ and azobenzene⁽²⁹⁾ by co-condensation of the chromophore-bridged precursors and framework precursors, although no luminescence properties have been recorded. The co-condensation approach is advantageous for the formation of highly ordered mesostructures, and for the suppression of self-

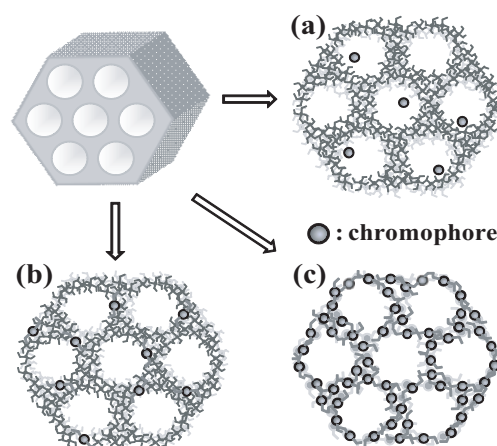


Fig. 1 Types of luminescent periodic mesoporous silicas and organosilicas. (a) Chromophore-doped silica (chromophores in mesopores). (b) PMO prepared by co-condensation (chromophores in framework). (c) PMO synthesized from 100% chromophore precursors (chromophores in framework).

quenching of chromophores due to high dispersion of these units in the PMO framework.

3. Luminescent PMOs synthesized from 100% chromophore precursors

Most organic molecules undergo a decrease in luminescence quantum yield upon solidification due to the strength of the interaction among molecules in the excited state. This behavior, known as solid-state quenching, may be one of reasons why the synthesis of PMOs from 100% chromophore precursors has not been studied to the same extent as the co-condensation approach. Recently, Chandra et al. reported the synthesis of a luminescent PMO film by condensation of 100% bis(propyliminomethyl)phenol-bridged organosilane.⁽³⁰⁾ The PMO film thus prepared was found to display affinity for metal cations (e.g., Fe³⁺ and Zn²⁺), and the luminescence spectrum was found to vary depending on the species of the exogenous metal cation, a function that may be utilized for metal ion chemosensors. The success of Chandra et al. appears to be the first report demonstrating luminescence from PMOs synthesized from 100% chromophore precursors.

More recently, Goto et al. investigated the fluorescence properties of benzene (Ph), biphenyl (Bp), naphthalene (Nph), and anthracene (Ant) bridged PMO films prepared from 100% bridged organosilane precursors.⁽²²⁾ The absorption spectra of the products were found to be similar to those of the precursors, suggesting very weak interaction among organic

groups in the framework in the ground state. The fluorescence spectra, on the other hand, were significantly red-shifted from those of the precursors (Fig. 2), suggesting strong interaction in the excited state. However, while the fluorescence quantum yields of the Ph-, Nph- and Ant-PMO films were markedly reduced from those of the precursors due to solid-state quenching, the Bp-PMO film exhibited an exceptional increase in fluorescence quantum yield (Table 1). This unusual increase in fluorescence quantum yield was also observed for a Bp-PMO powder (0.35 to 0.38).⁽³¹⁾ Such enhancement of fluorescence by solidification has been observed in various organic molecules,⁽³²⁾ polymers⁽³³⁾ and nanoparticles,⁽³⁴⁾ but had yet to be reported for organic-inorganic hybrid materials including PMOs. Although the mechanism of fluorescence enhancement for Bp-PMO remains unclear, it is likely to be attributable to a restriction of the intramolecular rotational vibration of Bp groups due to the dense packing structure. Another unique feature of the Bp-PMO film is strong absorption, again due to the dense packing of organic groups in the framework.⁽²²⁾ The absorption coefficient for this material is 87000 cm⁻¹ (Table 1), which is considerably higher than for dye-doped mesoporous silicas. The efficient light absorption and high fluorescence quantum yield of the Bp-PMO film make this technology a strong candidate as a lighting material.

4. Luminescence properties of PMOs with crystal-like and amorphous frameworks

One of the unique features of PMOs prepared from 100% organosilane precursors is the molecular-scale periodicity of bridging organic groups in the

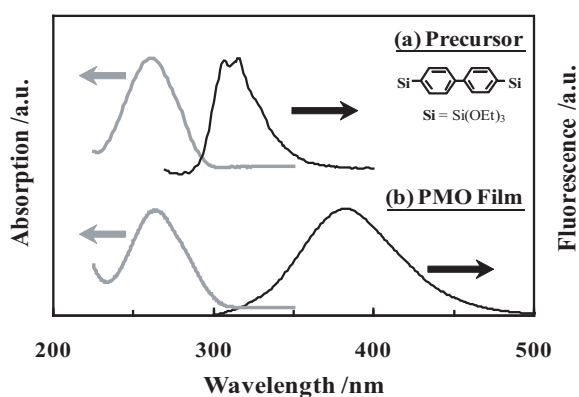


Fig. 2 Absorption (gray) and fluorescence (black) spectra for (a) the precursor (in 2-propanol) and (b) the corresponding Bp-PMO film (excitation wavelength, 280 nm).

Table 1 Absorption coefficients (α) and fluorescence quantum yields of PMO films and molar extinction coefficients (ϵ) and fluorescence quantum yields of the corresponding organic group-bridged organosilane precursors in 2-propanol.

	$\alpha / 10^4 \text{ cm}^{-1}$	$\epsilon / 10^4 \text{ M}^{-1} \text{ cm}^{-1}$	Fluorescence quantum yield	
	(at λ /nm)	(at λ /nm)	(excitation at λ /nm)	
	Film	Precursor	Film	Precursor
Ph-PMO	0.57 (270)	0.092 (270)	0.03 (266)	0.07 (266)
Bp-PMO	8.7 (263)	5.7 (260)	0.45 (266)	0.35 (270)
Nph-PMO	2.4 (282)	0.38 (278)	0.09 (266)	0.33 (280)
Ant-PMO	1.4 (382)	2.6 (380)	0.07 (380)	0.92 (360)

framework (**Fig. 3**). Such crystal-like frameworks have been observed for Ph,⁽⁹⁾ Bp⁽¹⁰⁾ and divinylbenzene⁽¹²⁾ bridged PMO powders. It is of interest to compare the luminescence properties of these crystal-like PMOs with PMOs having amorphous frameworks in order to better understand the interactions among organic groups in the frameworks.

Mizoshita et al. successfully prepared Nph-bridged PMOs with both crystal-like and amorphous frameworks, and found that Bp-PMO displayed excimer fluorescence in both the amorphous and crystal-like forms, while the amorphous Nph-PMO exhibited excimer fluorescence and the crystal-like Nph-PMO displayed monomeric fluorescence (**Fig. 4**).⁽³¹⁾ The monomeric fluorescence was confirmed by fluorescence measurements at -196°C , indicating the presence of a distinct monomer band emission with vibronic structures. Excimers in Bp-PMO are likely to be formed by rotation motion of the benzene rings around the Si–C bond axis, even in the crystal-like arrangement of Bp groups in the framework. In contrast, rotation motion of the naphthalene ring around the Si–C bond axis is inhibited in Nph-PMO, isolating the Nph groups in the crystal-like arrangement and giving rise to monomeric fluorescence.

Fluorescence studies revealed that the interaction among bridging organic groups in PMOs differs between the crystal-like and amorphous frameworks. This finding suggests that various functionalities can be designed by appropriate control of the molecular-scale arrangement of the organic groups in the framework.

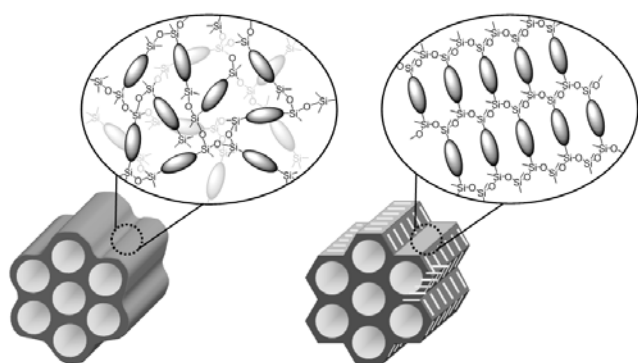


Fig. 3 Structural models of (left) randomly and (right) periodically arranged organic groups in the PMO framework.

5. Visible-light luminescent PMOs

Although Bp- and Nph-PMOs are known to exhibit fluorescence, the absorption and fluorescence bands are restricted to the ultraviolet region, and the fluorescence quantum yields are low compared with those for commercial metal-oxide phosphors (e.g., >0.6).⁽³⁵⁾ Efficient visible-light luminescent PMOs are required for applications such as lighting materials. We designed organosilane precursors to expand π -conjugated systems for red-shift of absorption and fluorescence bands of PMOs. Expansion of π -conjugated systems in PMOs has been also investigated by Sayari et al.^(12a) and Fröba et al.^(12b,36)

Anthracene is one of the organic groups that may be suitable for preparing visible-light luminescent PMOs. 2,6- and 9,10-Ant-bridged precursors have been used to prepare Ant-PMO films (**Fig. 5(a)**),^(22,37) and both films exhibit absorption in the visible region (ca. 400 nm) and blue fluorescence (450–500 nm) as expected (**Fig. 6**). However, the fluorescence quantum yields of these materials remain very low (0.13 and 0.07, respectively). The higher fluorescence quantum yield for the 2,6-Ant-PMO film is considered to be attributable to more severe restriction of the rotation motion of 2,6-Ant groups.

With the aim of fabricating highly luminescent PMOs, oligo(phenylenevinylene) (OPV) bridged precursors with different lateral substituents have been used to prepare OPV-PMO films (**Fig. 5(b)**).⁽³⁸⁾ All of

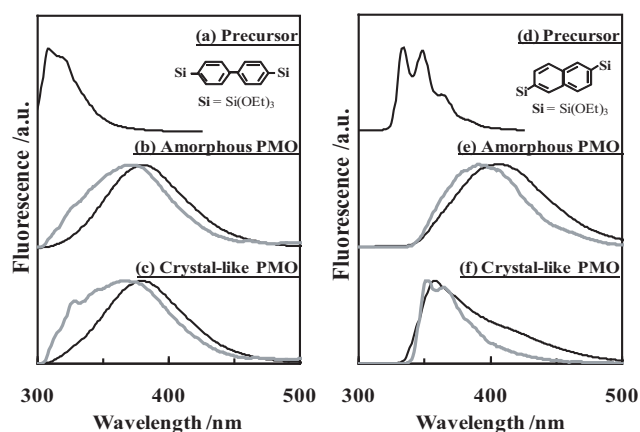


Fig. 4 Fluorescence spectra (black lines) for amorphous and crystal-like (a-c) Bp-PMO and (d-f) Nph-PMO and the corresponding precursors in 2-propanol. Gray lines denote fluorescence measurement at -196°C . Excitation wavelength, 280 nm.

the OPV-PMO films thus produced were prepared to have periodic mesostructures by the addition of TEOS at a rate of 1:2 by weight the precursor. Periodic mesostructured films were not obtained from the 100% OPV-bridged precursors. For OPV-H-PMO film without lateral substituents, the red-shift of the absorption and fluorescence bands was relatively small, and the fluorescence quantum yield remained low (0.25) due to the formation of H-aggregates among OPV groups in the framework (Fig. 7). The introduction of lateral substituents (hexyloxy or 2-ethylhexyloxy), however, resulted in red-shift of absorption to the visible region (ca. 400 nm) and blue fluorescence (450-500 nm) (Fig. 7) with fluorescence

quantum yields of 0.61 (hexyloxy) and 0.66 (2-ethylhexyloxy), comparable to those of commercial metal-oxide phosphors. The lateral substituents suppress aggregation of the OPV groups in the framework and thus inhibit solid-state quenching. The appropriate design of bridging organic chromophores has thus been demonstrated to allow for the preparation of PMOs with efficient emission of visible-light fluorescence.

6. Summary

Developments in luminescent periodic mesoporous organosilicas have proceeded rapidly in recent years. PMO preparation by co-condensation has been shown to be advantageous for the formation of highly ordered mesostructures and for suppression of chromophore self-quenching. PMOs synthesized by this approach have also been found to exhibit unique electrochemiluminescence and excitation energy transfer from donors in the framework to acceptors in the mesochannels. The synthesis of PMOs from 100% chromophore precursors is a relatively new route to these materials, and some of the products have been demonstrated to display fluorescence as a potential lighting technology. Bp-PMO in particular exhibits efficient absorption and high fluorescence quantum yield. Fluorescence studies have revealed that the interaction among bridging organic groups differs between crystal-like and amorphous frameworks, suggesting that it may be possible to design various

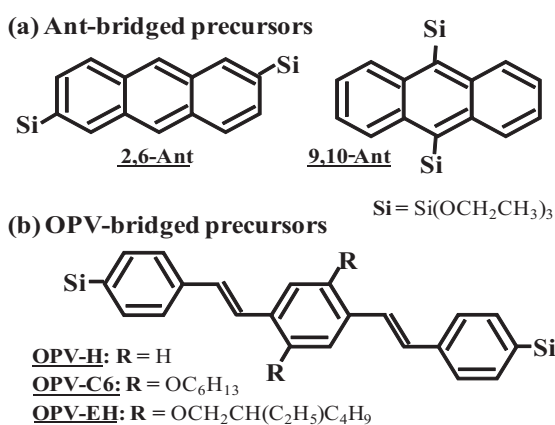


Fig. 5 Designed organosilane precursors for visible-light fluorescent PMOs. (a) 2,6- and 9,10-Ant-bridged organosilanes. (b) OPV-bridged organosilanes with and without lateral substituents.

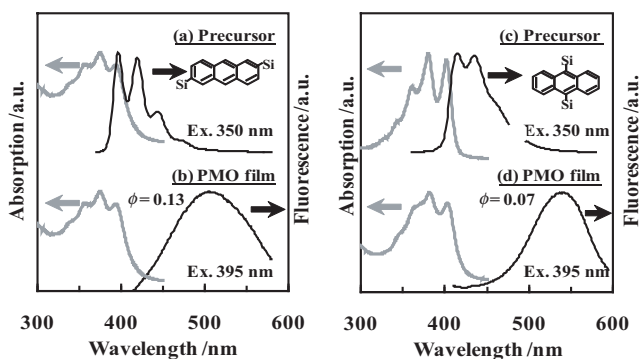


Fig. 6 Absorption (gray) and fluorescence (black) spectra for (a,b) 2,6-Ant-PMO and (c,d) 9,10-Ant-PMO films and the corresponding precursors in 2-propanol.

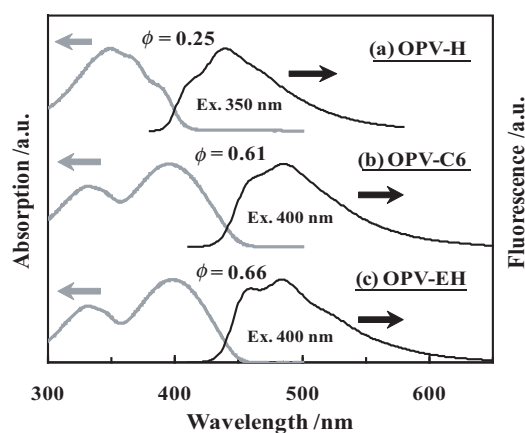


Fig. 7 Absorption (gray) and fluorescence (black) spectra for OPV-PMO films with and without lateral substituents: (a) no substituent, (b) hexyloxy (C6), and (c) 2-ethylhexyloxy (EH).

functionalities by appropriate control of the molecular-scale arrangement of the organic groups in the PMO framework. Similarly, efficient visible-light fluorescent PMOs could be obtained by molecular design of the bridging organic chromophores in the framework. These recent developments highlight the potential of luminescent PMOs as a new technology, which should be supported by further investigation of the optical properties and functionalization of PMOs.

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- The Society of Polymer Science, Japan
- International Mesoporous Materials Association
- International Zeolite Association

Awards :

- The Award of Chemistry on Catalyst Preparation, 1994
- The Promotion Award of the Japan Society on Adsorption, 2001
- The Chemical Society of Japan Award for Creative Work for 2004
- The Minister Award of Education, Culture, Sport, Science and Technology, 2005
- The Japan Society on Adsorption Award, 2008

