



Synthesis of ordered macro-mesoporous Mg–Al composite oxides with high thermal stability and mechanical strength



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ABSTRACT

Ordered macro-mesoporous Mg–Al composite oxides (denoted as OMM-MA-x) with tunable macropore size and highly homogenous distribution of Mg have been synthesized via a confined evaporation induced self-assembly method, in which triblock copolymer F127 and polystyrene colloidal crystals are utilized as templates for ordered mesoporous and macroporous structures, respectively. The resultant OMM-MA-x materials exhibit a much more ordered 2D hexagonal mesostructure constituting wall skeleton of ordered macropores and more excellent textural properties than those of ordered macro-mesoporous alumina materials. More importantly, both ordered mesostructure and macroporous structure of OMM-MA-x can be well maintained even after thermal treatment at 900 °C or pressure treatment at 15 MPa.

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1. Introduction

Compared to conventional alumina used extensively in catalysis and adsorption [1,2], ordered macro-mesoporous alumina (OMM-A) materials should demonstrate significantly boosted performance, due to their advantages in efficient mass transportation through macropores [3,4] and high surface areas contributed by mesopores [1]. Via a dual-templating approach, OMM-A materials can be easily obtained [5]; while, it is difficult to prepare OMM-A with high thermal stability, since the phase transitions usually leads to the thermal collapse of ordered structure [1]. Moreover, the low mechanical strength is still another important factor seriously impeding the practical applications of OMM-A materials. It is challenging to prepare OMM-A materials with ordered bimodal porous structures, excellent textural properties, and enhanced stability.

The recent reports have shown that via the sol-gel procedure, the incorporation of Mg into alumina matrix plays an important role in promoting the construction of ordered mesostructure with enhanced thermal stability [6,7]. Inspired from these previous works, herein, ordered macro-mesoporous Mg–Al composite oxides with tunable interconnected macroporosity and ordered 2D hexagonal mesostructure were successfully synthesized, and the roles of homogeneously incorporating Mg in improving the structural and textural properties as well as stability of OMM-A materials were investigated.

2. Experimental section

2.1. Synthesis procedure

In a typical synthesis, 3.2 g of triblock copolymer F127, 0.51 g of $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, and 0.4 g of citric acid were successively dissolved in 20 mL anhydrous ethanol containing 2.0 g of 37 wt% hydrochloric acid, followed by the slow addition of 3.26 g of aluminum isopropoxide. After being vigorously stirred at 30 °C for 24 h at a sealed condition, 0.7 g of monodisperse polystyrene (PS) spheres with a given diameter (adjusted from 200 to 400 nm), were added to the clear solution with continuous stirring for 15 min. The resultant reaction mixture was transferred into a petri dish for solvent evaporation at 45 °C for 24 h and then thermal treatment at 100 °C for 24 h. The final products were calcined at 500 °C for 5 h to remove the templates, and named as OMM-MA-x, where x stands for the diameter of used PS spheres.

For comparison, ordered macro-mesoporous alumina (denoted OMM-A-300, using PS spheres with a diameter of 300 nm as the hard-template) and ordered mesoporous Mg–Al composite oxide (named as OM-MA) were also prepared according to aforementioned synthesis method (without the addition of Mg precursor and PS spheres, respectively).

2.2. Characterization

The microscopic features of the samples were obtained by a transmission electron microscope (TEM, JEOL 2011) and a

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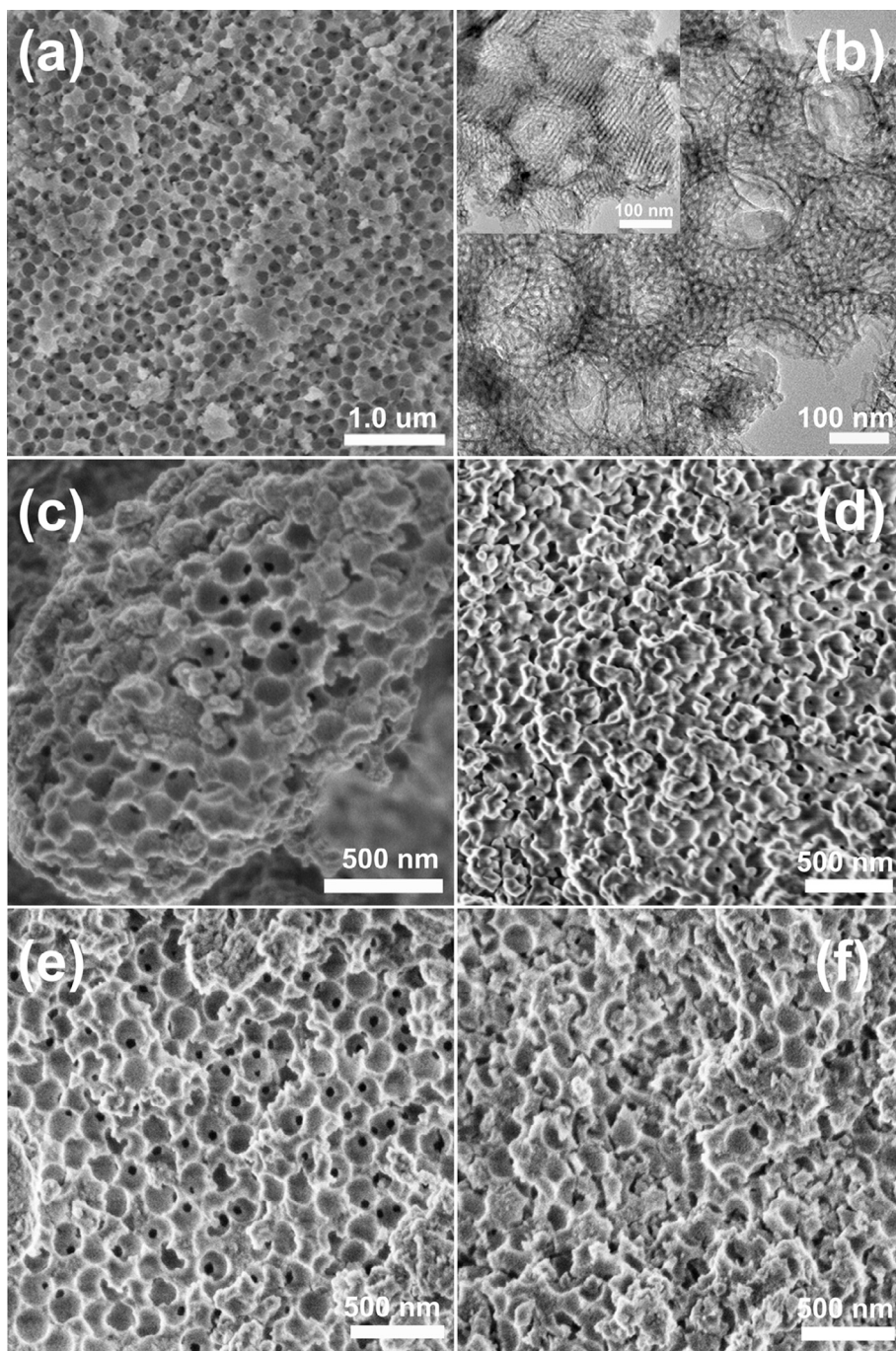


Fig. 1. SEM (a, c, d, e, and f) and TEM (b) images of OMM-MA-300 (a and b), OMM-MA-300(HC) (c), OMM-A-300(HC) (d), OMM-MA-300(HP) (e), and OMM-A-300(HP) (f). The TEM images are viewed along [100] (b) and [110] (the inset in b) orientations, respectively.

Table 1

The structure parameters of samples before and after different treatments.

Sample	a (nm)	S_{BET} (m^2/g)	V_{p} (cm^3/g)	D_{meso} (nm)	D_{macro} (nm)
OM-MA	11.58	260	0.57	9.9	–
OMM-MA-300	11.58	330	0.70	9.9	180
OMM-MA-300(HC)	10.40	262	0.55	8.6	160
OMM-MA-300(HP)	11.08	218	0.47	8.6	165
OMM-A-300	10.19	309	0.53	6.9	180
OMM-A-300(HC)	8.50	220	0.36	5.7	–
OMM-A-300(HP)	9.80	147	0.29	8.7	–

Note: a is the cell dimension, S_{BET} is BET surface area, V_{p} is the total pore volume, D_{meso} is the mesopore size calculated from the adsorption branch using the BJH method, and D_{macro} is the macroporous size calculated from the SEM and TEM observation.

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