Synthesis of spherical mesoporous organosilica by microwave heating

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INTRODUCTION

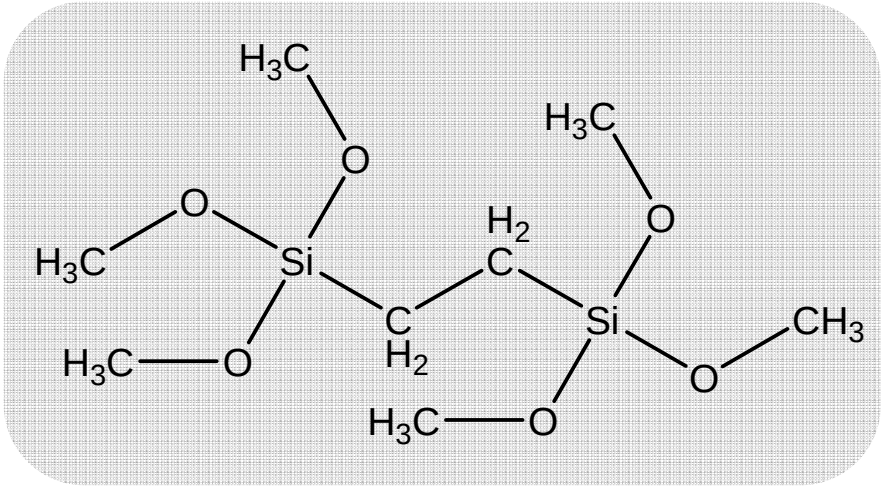
Organic-inorganic hybrid mesoporous material was synthesized using microwave heating method at various synthesis temperature and time condition. These materials exhibit good crystallinity which compared with the sample synthesized by hydrothermal method The decomposition of sample contained ethane group in their framework gradually appeared at temperature over 200 ℃ SEM images show that hybrid mesoporous materials synthesized by microwave heating have smaller particle size, spherical shape and well-defined morphology compared with them by hydrothermal method. Synthesis condition of hybrid mesoporous material is carried out in molar ratio of BTME : C_{16}TMA : NaOH : H_2O system. To control morphology of the hybrid mesoporous material, four factors were considered; synthesis time, synthesis temperature, synthesis process and microwave heating rate control at the first stage. By microwave heating system, synthesis time is remarkably reduced from 21 h to 0.5 h-6 h. Particle size of hybrid mesoporous material is enlarged by controlling the heating rate of microwave equipment at the first step. For HPLC column test, the hybrid mesoporous material synthesized by microwave heating at 95˚C for 4 h shows separation ability as good as the commercial silica.

EXPERIMENTAL

◆ Substrate molar composition

BTME	CTMACl	NaOH	H_2O
1	0.91	2.28	336

◆BTME (1,2-Bis(trimethoxysilyl)ethane)



◆ Synthesis of PMO

C_{16}TMACl + NaOH + H_2O

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Adding BTME

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Stirring at 25 ℃ for 19h

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Microwave heating

95 ℃	115 ℃	135 ℃
	2h	
4h	4h	4h
	6h	

↓

Filtration

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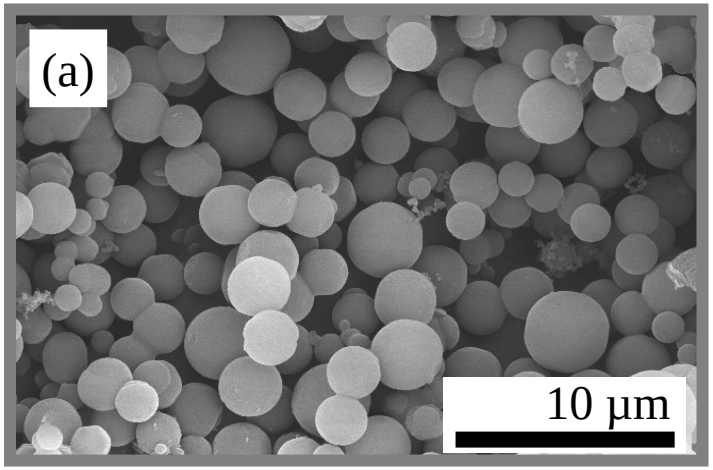
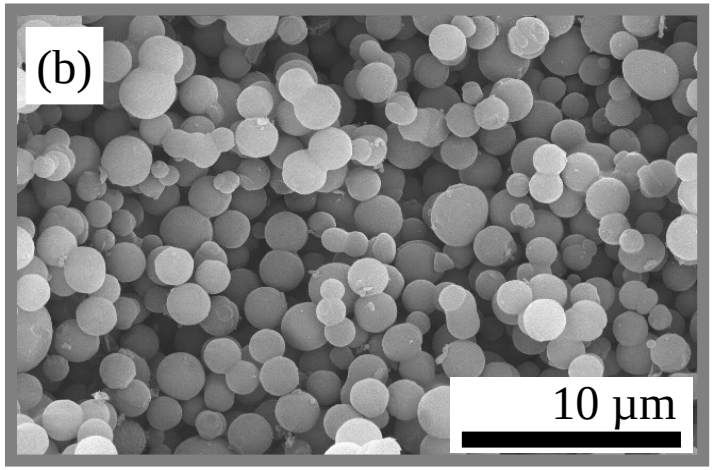
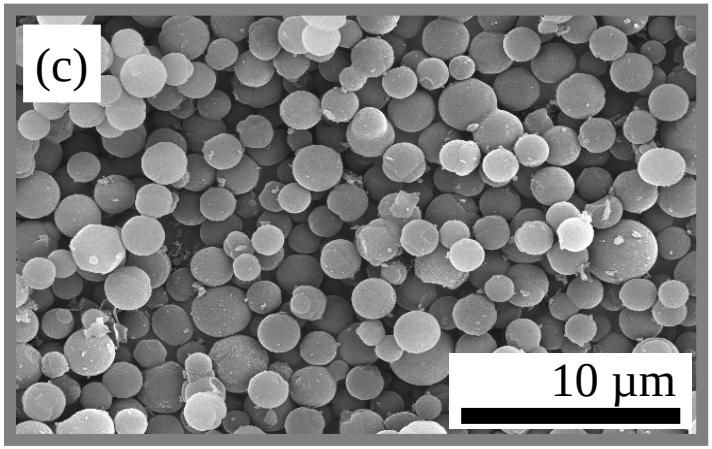
HCl / EtOH soln. extraction

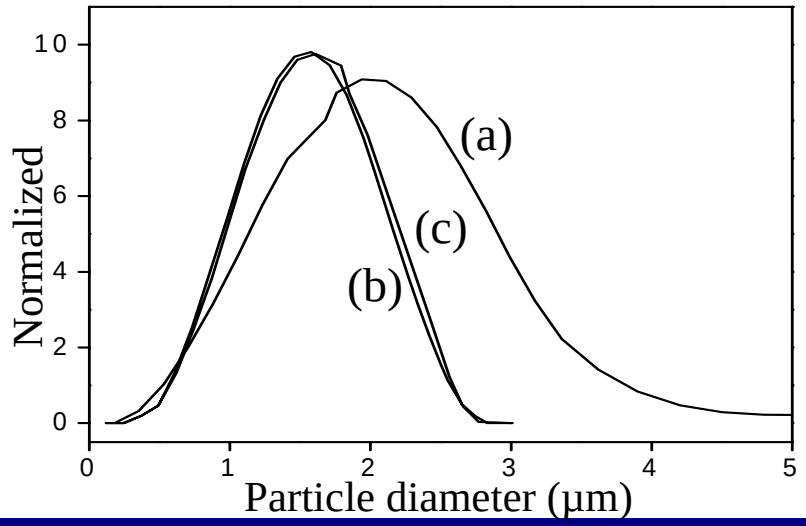
RESULTS & DISCUSSION

◆ **SEM images and particle size distributions of hybrid mesoporous materials synthesized at (a) 95 ℃, (b) 115 ℃ and (c) 135 ℃ for 4h.**



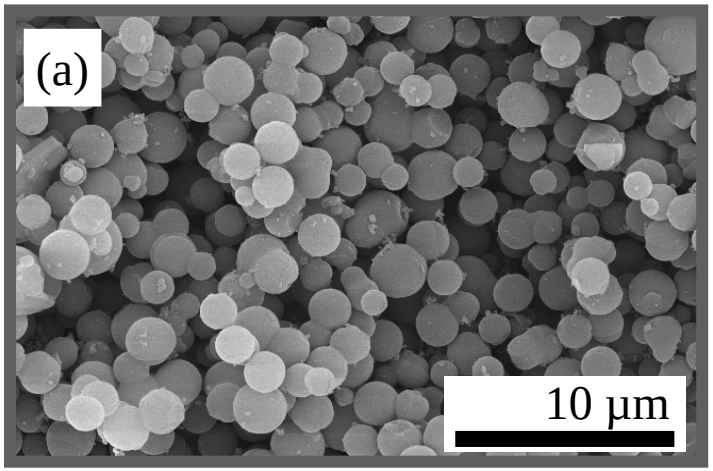


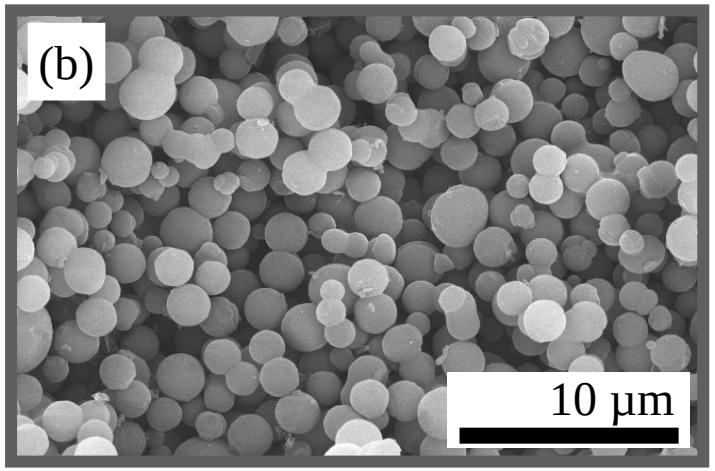


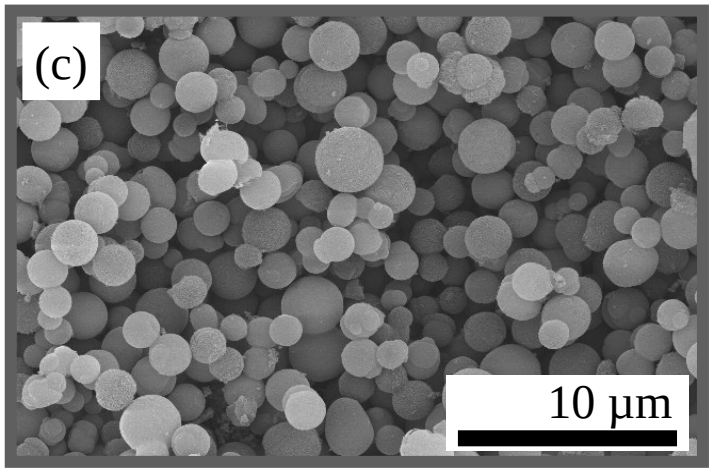


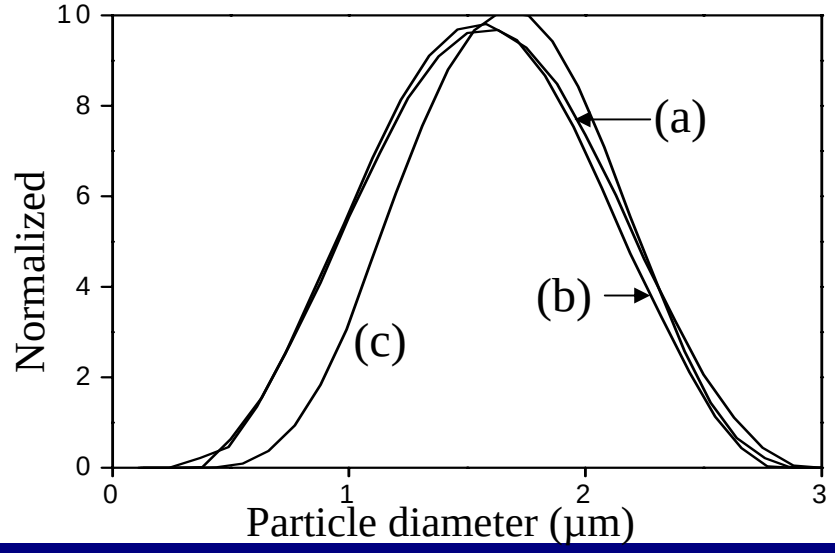
RESULTS & DISCUSSION

◆ **SEM images and particle size distributions of hybrid mesoporous materials synthesized at 115 ℃ for (a) 2 h, (b) 4 h and (c) 6 h.**



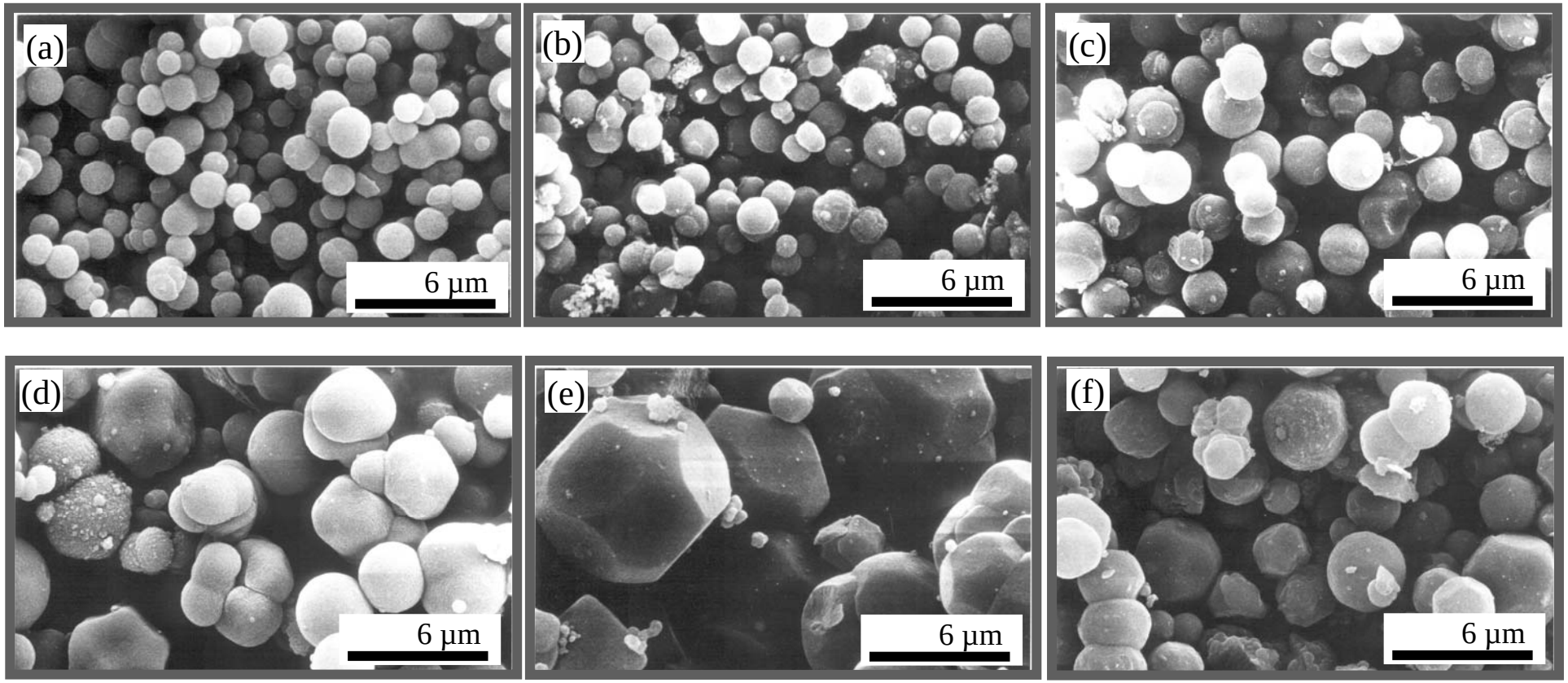






RESULTS & DISCUSSION

◆ **SEM images of hybrid mesoporous materials synthesized using different heating rate by the control of heating power (maximum 950W at 2,450MHz) at 115 ℃ for 4h; (a) 100% (b) 50% (c) 25% (d) 15% (e) 5% of maximum power (f) 100%, addition of a swelling agent (mesitylene : surfactant = 2)**



CONCLUSIONS

- Organic-inorganic hybrid mesoporous materials were synthesized using microwave heating system.
- The hybrid mesoporous material with uniformly particle and smaller particle size was synthesized by microwave heating because rapid heating rate promotes nucleation process and simultaneously reduces reaction time in the crystallization stage.
- Particle size of the hybrid mesoporous materials in diameter is controlled between 1.5 μm and 2.2 μm.
- By microwave heating method, the synthesis time of these materials is largely reduced.