

Rapid synthesis of MOF-5 via microwave radiation

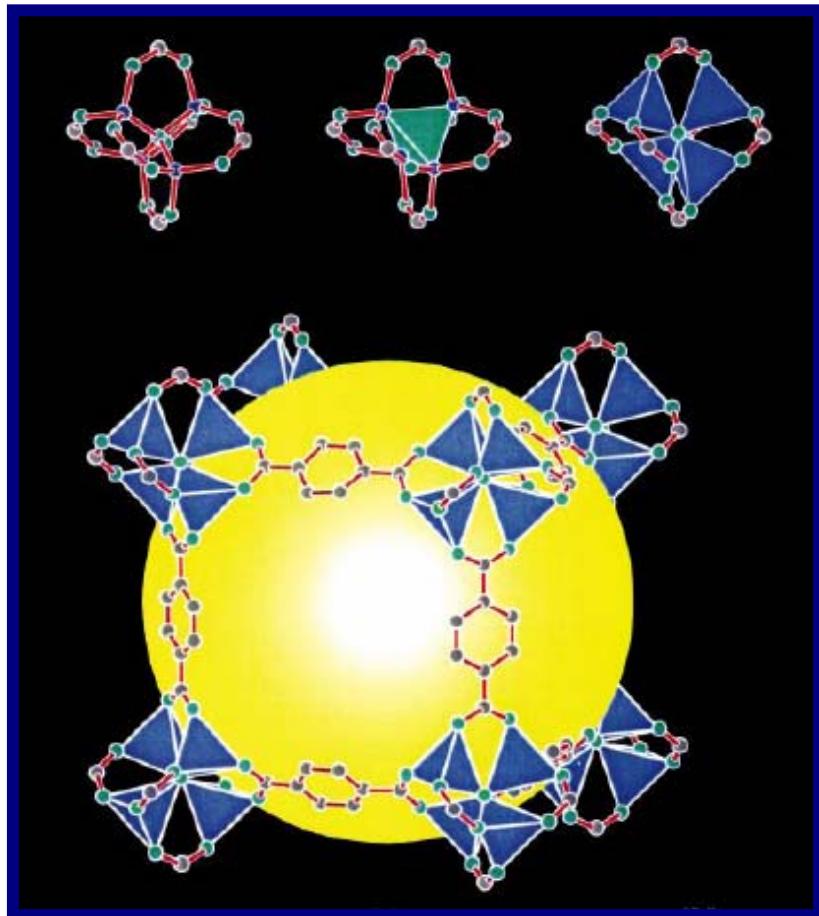


Jung-Sik Choi and Wha-Seung Ahn

Department of Chemical Engineering, Inha University, Korea

MOF(Metal Organic Framework)

● What is MOF(Metal Organic Framework)?



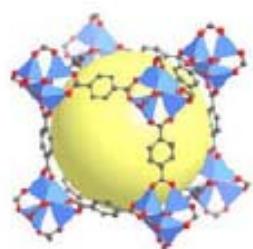
- 전이금속 이온이나 클러스터를 구조물의 꼭지로 하여 이들을 배위결합이 가능한 다양한 유기 리간드로 연결하여 높은 구조체.
- 다공성 화합물로 작용 가능. 현존하는 가장 비표면적이 큰 물질 중 하나.
($S_A > 5,000 \text{ m}^2/\text{g}$ 가능.)
- 다양한 유기 리간드와 금속 이온의 사용으로 기공의 구조와 화학적 물성 조절 가능.

대표적 MOF인 MOF-5 $[\text{Zn}_4(\text{O})\text{O}_{12}\text{C}_6]$

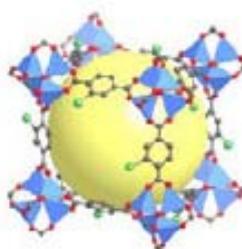
MOF (Metal Organic Framework)

● Isoreticular Metal-Organic Frameworks (IRMOFs)

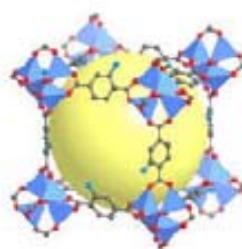
a series of materials having the same underlying topology and (typically) constructed from the same secondary building units



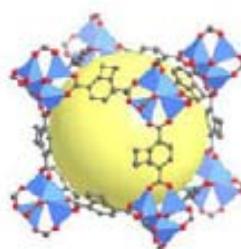
IRMOF-1



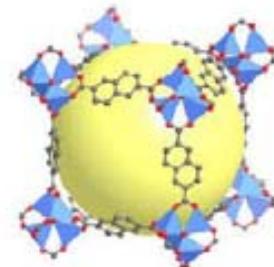
IRMOF-2



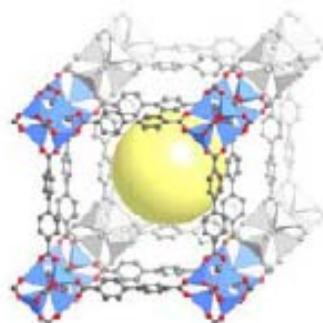
IRMOF-3



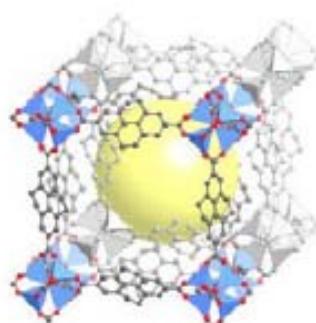
IRMOF-6



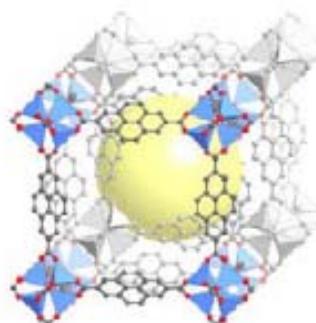
IRMOF-8



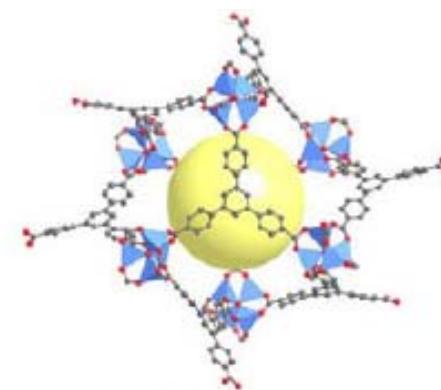
IRMOF-9



IRMOF-11



IRMOF-13



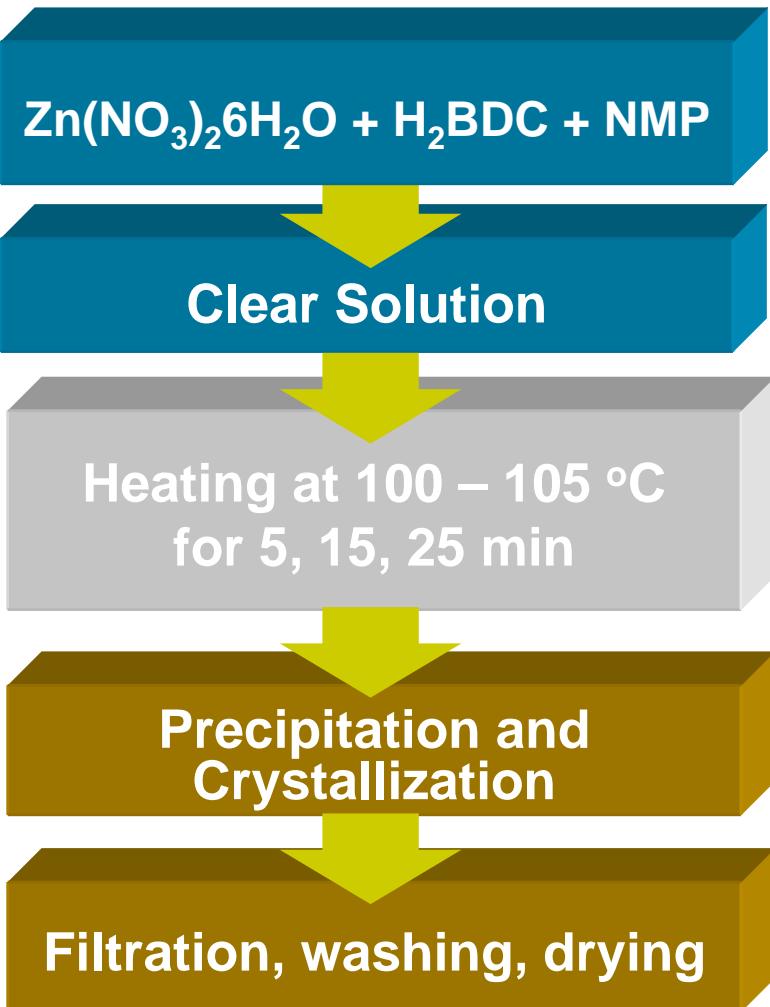
MOF-177

M. Eddaoudi, J. Kim, N. Rosi, D. Vodak, J. Wachter, M. O'Keeffe, O.M. Yaghi, *Science*, **2002**, 295, 469.

H. K. Chae, D. Y. Siberio-Perez, J. Kim, Y-B. Go, M. Eddaoudi, A. J. Matzger, M. O'Keeffe, O.M. Yaghi, *Nature*, **2004**, 427, 523.

EXPERIMENTAL

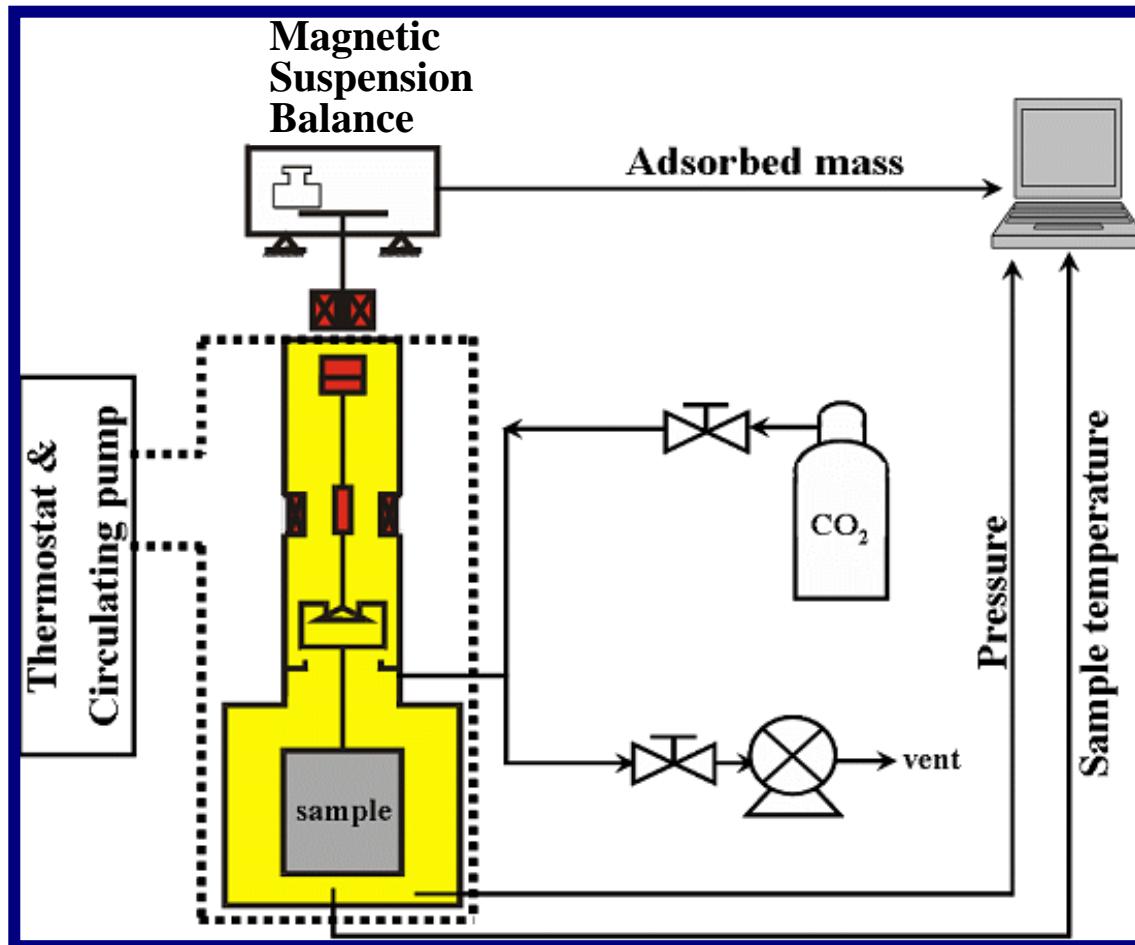
● Synthesis flow sheet



Microwave synthetic system

Equipment

● CO₂ 흡착/저장 성능의 정량화 : gravimetric analysis



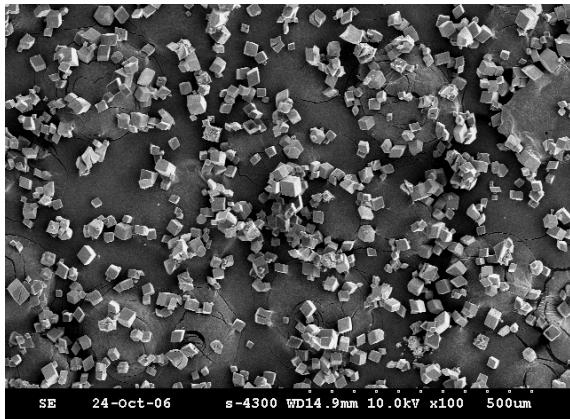
- 흡착등온선 (1-40 기압, 상온)
- 흡탈착 재생성 (Pressure Swing sorption).
- TGA 기능을 이용한 흡착제의 재생성 분석 (Temperature Swing Analysis).
- 흡/탈착 속도 측정.

Rubotherm
Magnetic Suspension Balance
- Resolution : 10^{-5} g
- Max. Weight : 80 g

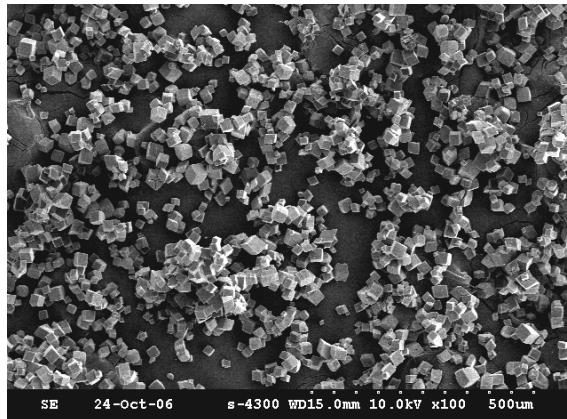
RESULTS & DISCUSSION

● Scanning Electron Microscope image

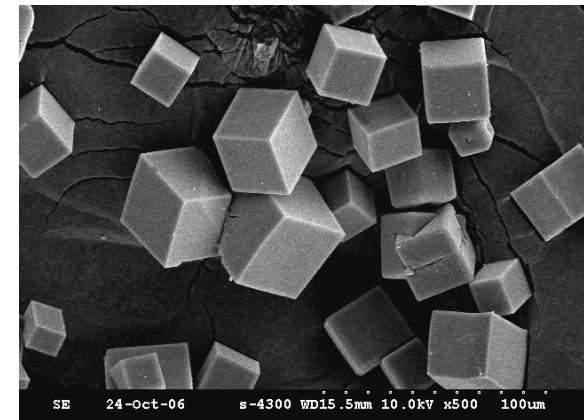
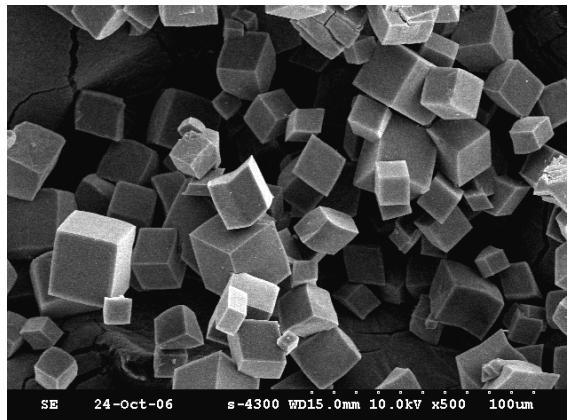
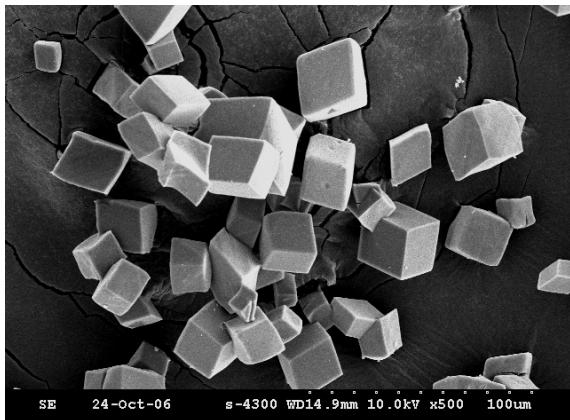
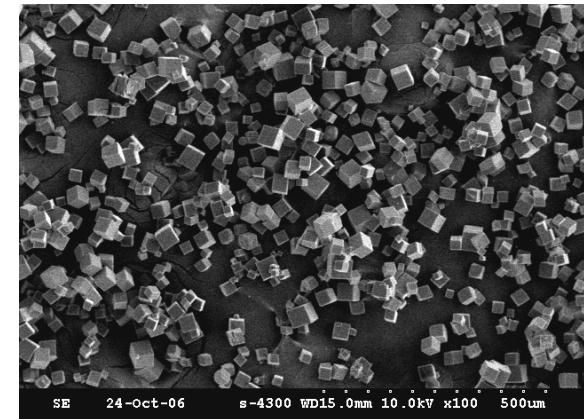
Power 25%, 5 min



Power 25%, 15 min



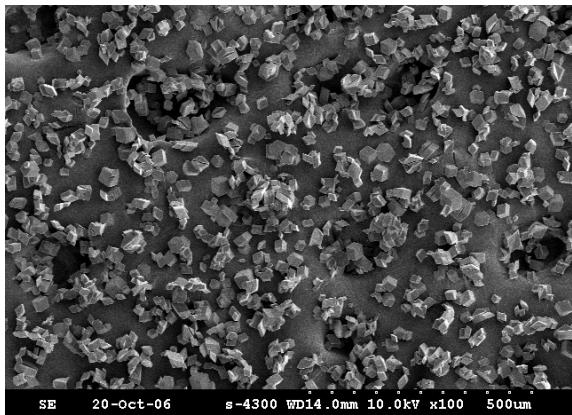
Power 25%, 25 min



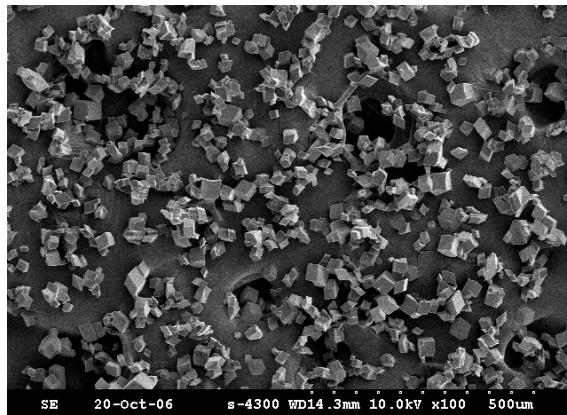
RESULTS & DISCUSSION

● Scanning Electron Microscope image

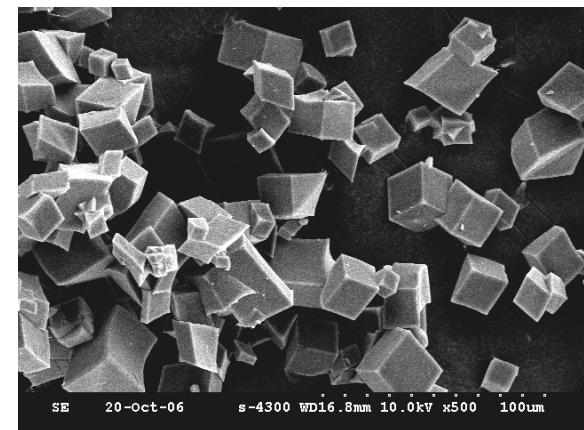
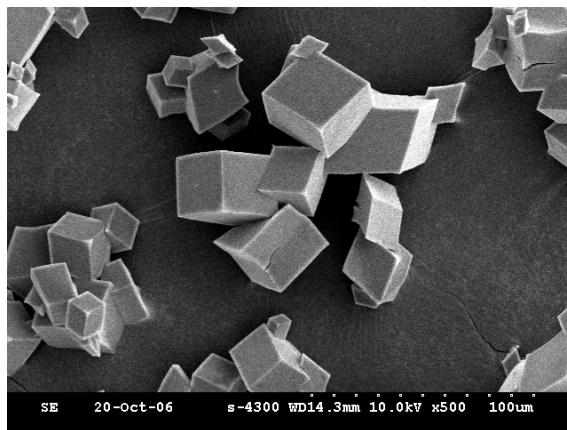
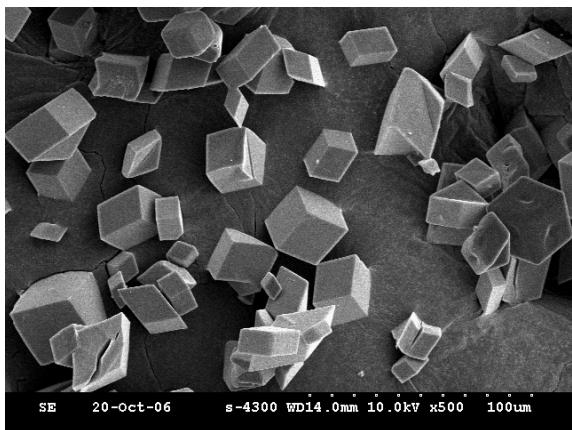
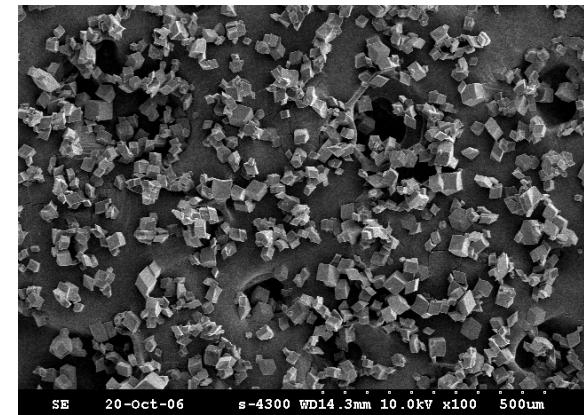
Power 75%, 5 min



Power 75%, 15 min

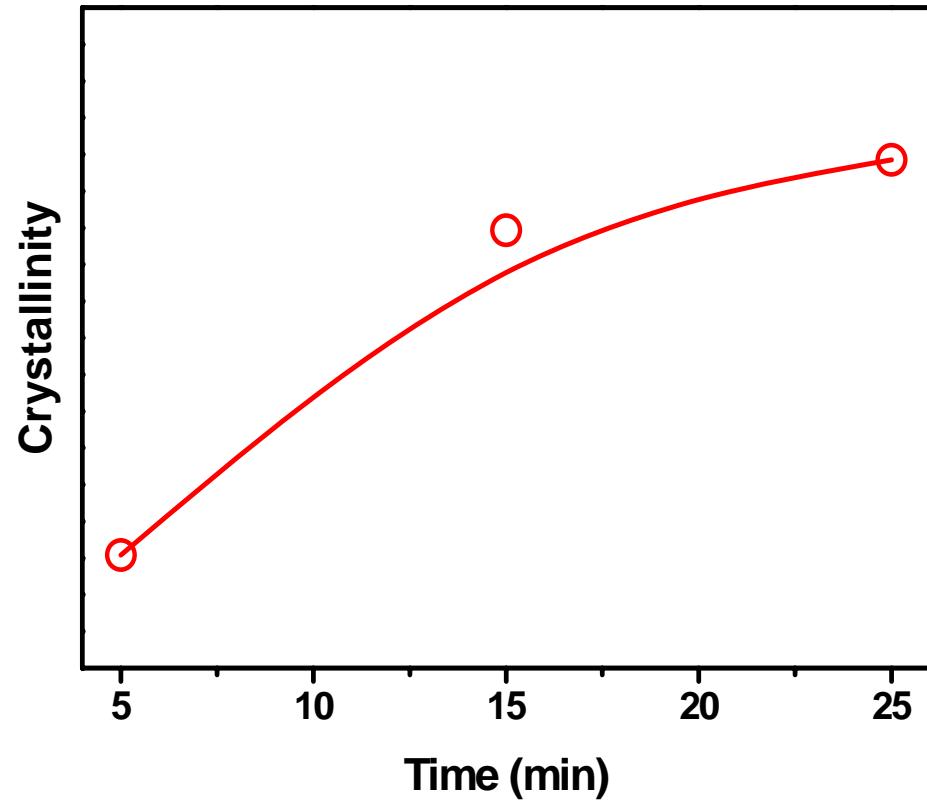
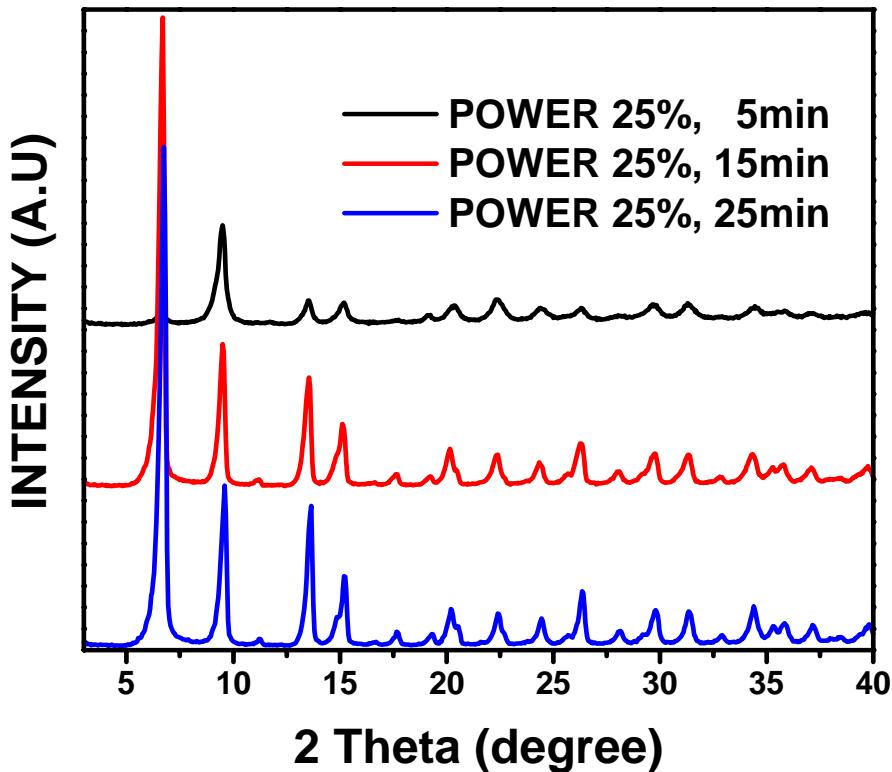


Power 75%, 25 min



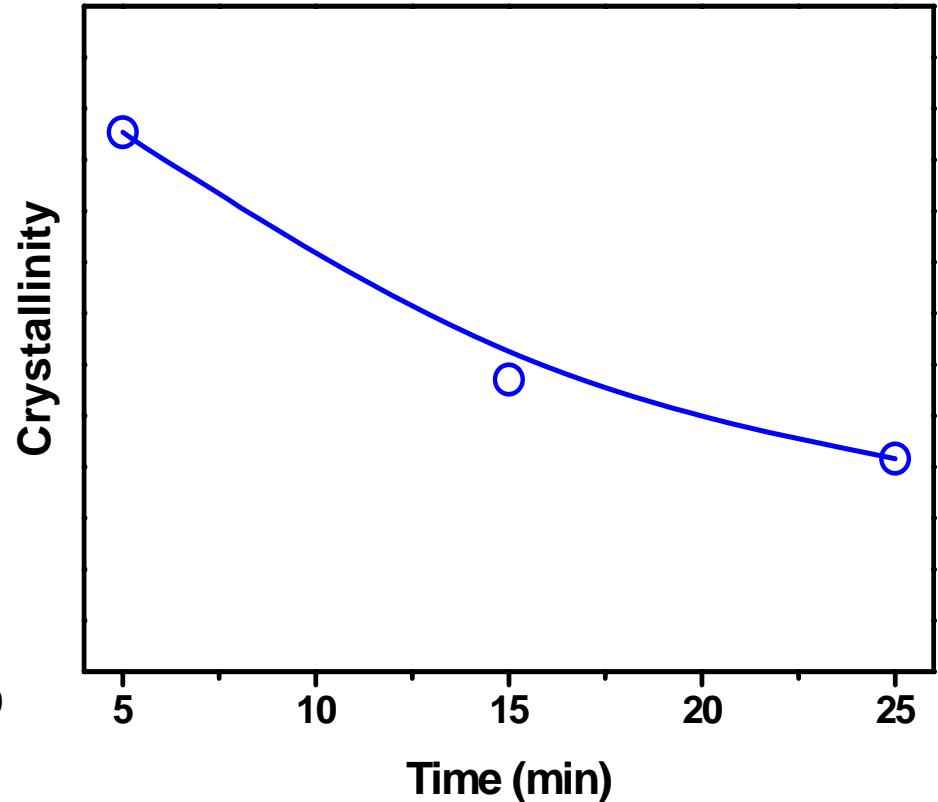
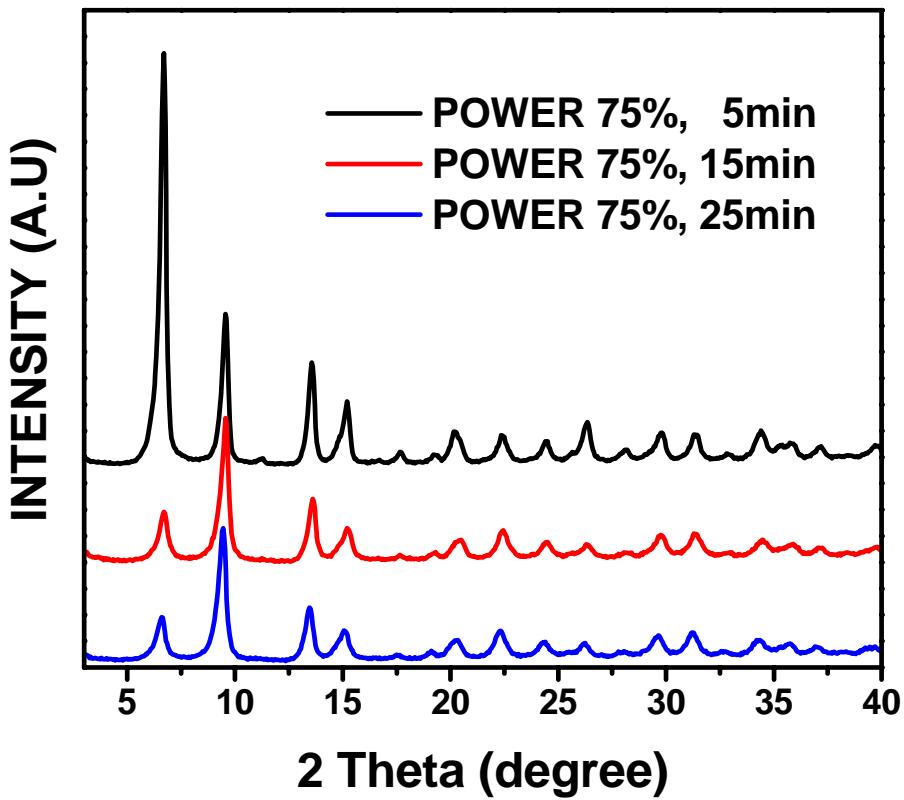
RESULTS & DISCUSSION

● X-Ray diffraction patterns



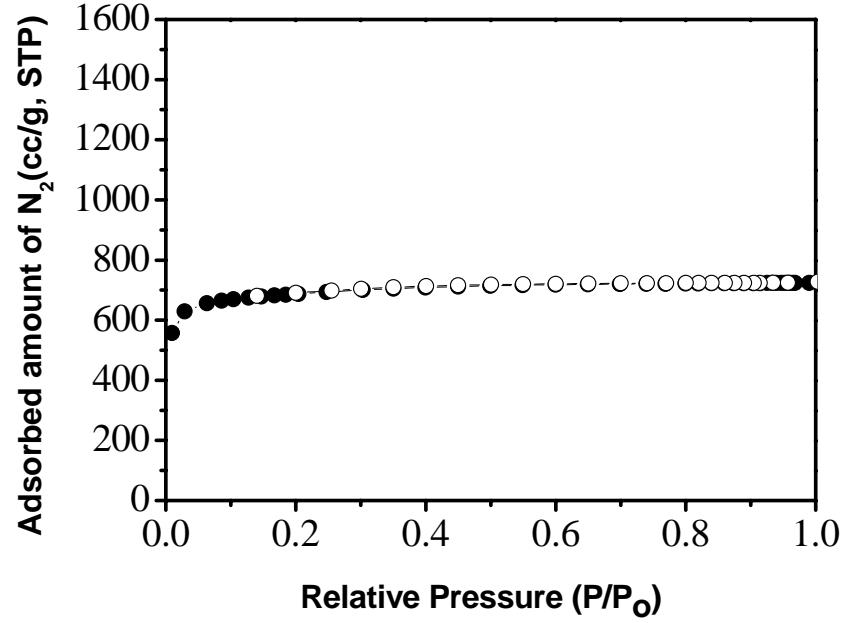
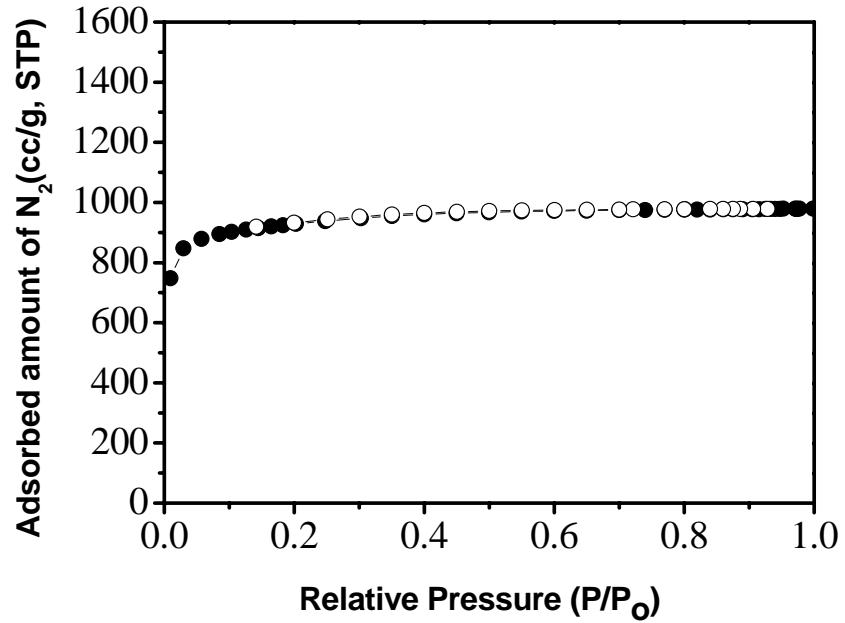
RESULTS & DISCUSSION

● X-Ray diffraction patterns



RESULTS & DISCUSSION

● Nitrogen Ad/Desorption analysis



Power 25%

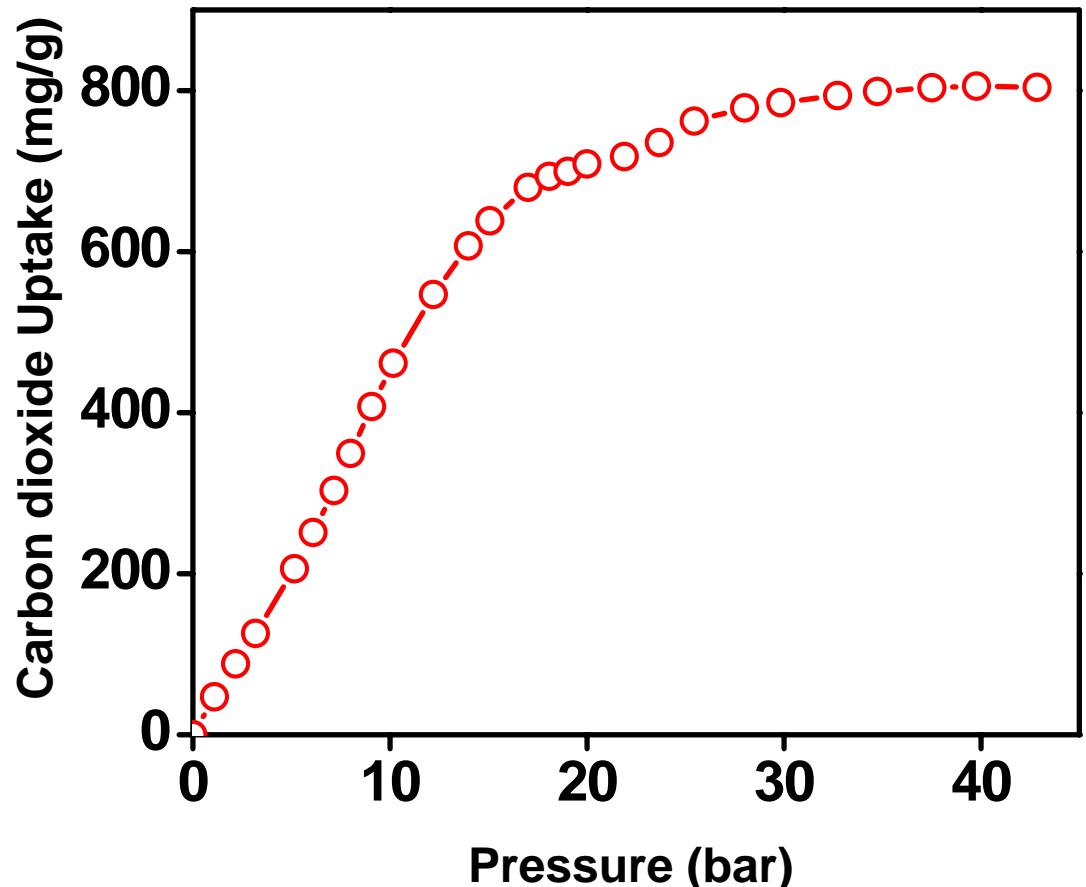
Langmuir Area (m ² /g)	3200–4000
Micropore Area (m ² /g)	2400–3000
Pore volume (cc/g)	1.20–1.60
Micropore Volume (cc/g)	1.13–1.45

Power 75%

Langmuir Area (m ² /g)	2700–3000
Micropore Area (m ² /g)	2000–2200
Pore volume (cc/g)	1.00–1.12
Micropore Volume (cc/g)	0.95–1.07

RESULTS & DISCUSSION

● CO₂ adsorption capacity of microwave synthesized MOF-5



Conclusions

- MOF-5 was successfully synthesized by microwave heating with ca. 3000-4000 m²/g surface area.
- Synthesis period of MOF-5 was reduced from 2 days to 5-25 min.
- CO₂ adsorption capacity of MOF-5 synthesized by microwave was 805 mg/g at 40 bar.