MICRO-CT TECHONOLOGY IN DEVELOPING A CONTROLLABLE MODIFICATION OF SMART MICRO-PORE ON THE SURFACE OF MESO-PORE MATERIALS

Tzu-Hung Lin[*1], Fan-Chin Chang[†1] & Hsin-Ming Cheng[1]

[1]Material and Chemical Research Laboratories, Industrial Technology Research Institute, Taiwan (ROC)

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Summary: In order to enhance the water adsorption efficacy, high resolution micro-CT was used to analyse the structure and pore distribution in SGHF structure for the design of pore modification.

1. INTRODUCTION

Recently, government raises awareness of the recycling and zero emission of wastewater. Dealing with the high solid content slurry with low environmental humidity is the key step of this procedure. However, thermal drying which is the most common method requires huge power. Power efficient and low cost technique for water recycling is needed and adsorption desalination is one of the candidates. Based on this technology, we modified a micro/meso pore structure to enhance the water adsorption efficacy.
The technology of X-ray micro-CT was used in this project. Micro-CT technology can provide high resolution and detailed 3D image for us to investigate the 3D information inside our sample without destruction. At first, we used X-ray micro-CT (XCT) to analyse the inner structure and pore distribution inside silica gel hollow fiber (SGHF) and we further designed the micro pore material, metal organic frameworks (MOF, Uio66-NH2), which was then covered to the interface of SGHF fingerprint structure. According to the micro-CT results, the appropriate structure of MOF was selected and we further choose several kinds of methods to modify MOF on silica gel hollow fiber. Micro-CT was used to examine the distribution of MOF by different kinds of modify methods and we further analyze the correlation between MOF distribution and water adsorption efficacy.

2. EXPERIMENTAL METHOD

2.1 Microcomputed Tomography (micro-CT).
The samples were cut into appropriate size and then scanned by micro-CT. Samples were subjected to the X-ray microtomography apparatus by using Skyscan 1272 (Bruker Skyscan, Kontich, Belgium). Scanning was done at 50 kVp and 200μA. For structure analysis, the images were collected at a resolution of 550 nm/pixel (1x1 binning). For MOF distribution analysis, the images were collected at the resolution of 4.6 μm/pixel (2x2 binning). Reconstruction of sections was carried out with GPU-based scanner software (GPU-NRecon). Quantification of morphometric indices and porosity was performed by scanner software (CTAn, Version 1.16.1.0). Orthogonal images were obtained using Dataviewer (Version 1.5.2.4) and 3D visualization images were obtained with CTVox software (Version 3.1).

2.2 SEM/XRD
Followed by previous investigation, nano-MOF was synthesized, washed and re-dispersed in DI water for SGHF modification and further characterization. The morphologies and size distribution of nano-MOF were characterized by scanning electron microscope using a JEOL 7610 operated at 5 to 15 kV equipped with energy dispersive spectrometer analyses (EDS). Crystal phase of nano-MOF was identified by wide-angle powder X-ray diffraction patterns obtained with Bede D1 with monochromated Cu Kα radiation (35 kV, 35 mA).

* e-mail: itri453401@itri.org.tw
† e-mail: KarenChang@itri.org.tw

2.3 Water adsorption test

Water uptake ability of SGHF with MOF modification in different weight percent was tested by Discovery TGA (TA Instruments, Delaware, USA). MOF-modified SGHF was divided into an appropriate size to place on the TGA sample pen. Dried nitrogen gas (6N, high purity grade) was utilized as a carrier gas bring water vapour to sample pan placed quartz liner by passing through an Erlenmeyer flask contained DI water. Before purging water vapour, samples were dried under 150 ^{o}C for 30 minutes then cooled down to 35 ^{o}C under pure N2 atmosphere. The changed weight during these five dry/wet cycles was recorded for further analysis.

3. RESULTS

The SGHF structure was scanned by using very high resolution (550 nm/pixel) to investigate the pore distribution inside this 3D structure. Micro-CT results showed that there were two major layers of the structure (Fig.1a), the high dense layer and low dense layer were both constructed by the same material, one of them is water absorption material. We then isolated two area (300 um * 300 um * 180 um) for further analysis (Fig. 1b). The morphometric indices of the structure, porosity and pore size distribution were analysed (3D visualized in Fig. 1c). The major pore size of high dense layer and low dense layer is 2-3 um and 5-15 um, respectively. We then choose the appropriate size of MOFs to modify the SGHF structure with several kinds of modification methods.

Micro-CT results demonstrated that thermal self-assembling technique provides nice MOF distribution. In addition, appropriate MOF distribution contributed better water adsorption. This new modified micro/meso pore structure could be developed as effective solution for water recycling.

- Results of high resolution micro-CT was used for the design of pore modification of SGHF structure
- MOF distribution can be analysed using micro-CT and is positively correlated with water adsorption efficacy

References

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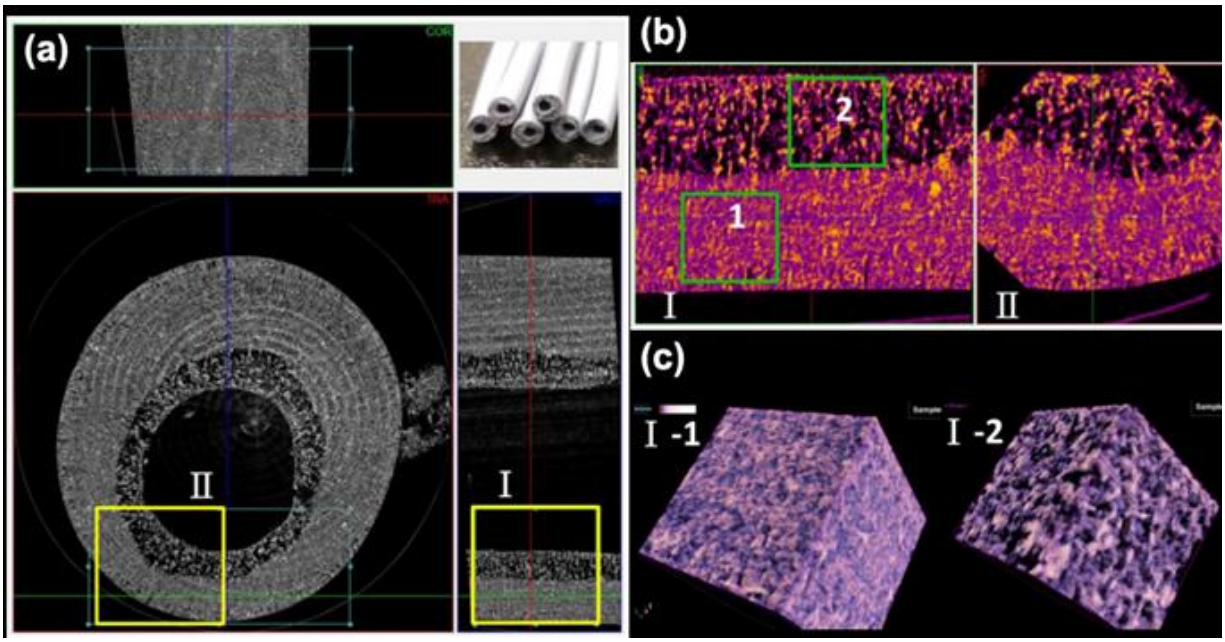

Figure 1: SGHF was scanned by micro-CT using sub-micron resolution (550 nm/pixel). The orthogonal view of SGHF (a). Two areas were isolated for analyzation and visualization (b and c).