

## Preparation of magnetic $\beta$ -glucan microspheres for MRI-detectable embolic materials

Jun Hee Cho<sup>(1)</sup>, Sang Gil Ko<sup>(2)</sup>, Yangkyu Ahn<sup>(3)\*</sup>, Eun Jung Choi<sup>(4)</sup>

- (1) Quantum Optics Division, Korea Atomic Energy Research Institute, Deajeon, Korea, 305-353  
 (2) Environment & Resources Research Center, Korea Research Institute of Chemical Technology, Deajeon, Korea, 305-600  
 (3) Department of Chemistry, Konyang University, Nonsan, Chungnam, Korea 320-711  
 (4) Department of Ophthalmic Optics Konyang University, Daejeon, Korea 302-718  
 \* Corresponding author.

**Abstract** – Magnetite nanoparticles were encapsulated with a biopolymer, such as  $\beta$ -glucan, to improve their biocompatibility [1]. We have synthesized magnetite ( $\text{Fe}_3\text{O}_4$ ) nanoparticles by using a sonochemical method with oleic acid as a surfactant. Powder X-ray diffraction measurements showed the spinel structure for the magnetite nanoparticles. The synthesized magnetite nanoparticles were spherical with an average diameter of about 9 nm. These nanoparticles were embedded in a  $\beta$ -glucan by sonochemical method, which were formed ferrofluid. This ferrofluid was sprayed on the surface of an acid solution to make microspheres that are profitable shape and size for embolic materials. The T1- and T2-weighted MR images of these magnetic  $\beta$ -glucan microspheres were obtained with a 4.7 T magnetic resonance imaging system (Bruker, BioSpec 47/40). These microspheres were exhibited the enhancement of MRI contrasts *in vitro*.

A mixed solution of 0.15 M  $\text{FeCl}_2$  (30 ml, 7.5 mM) and 0.30 M  $\text{FeCl}_3$  (30 ml, 15.0 mM) was prepared for synthesis of uniform magnetite nanoparticles. After adding oleic acid to the solution, ultrasonic waves were immediately irradiated to the mixture with the power of 250 W. TMAOH (72.0 mM) solution was rapidly added to the mixture at 80 °C to obtain black nanoparticles as a magnetite ( $\text{Fe}_3\text{O}_4$ ). It was washed at least three times by using ultracentrifugation. It was obtained a spherical shape with an average diameter of about 9 nm. The magnetite nanoparticles were dispersed into 5%  $\beta$ -glucan-sodium hydroxide solution while giving ultrasonic irradiation for 15 min. Iron (Fe) concentration of this stock solution was 0.02 M and the stock solution was diluted to 0.2 mM. The magnetic  $\beta$ -glucan solution was sprayed by a nozzle on the surface of the acid solution to prepare embolic materials in the form of microspheres. The magnetic  $\beta$ -glucan microspheres were thus required to narrow down the size distribution of the sprayed microspheres so that microspheres of 100–150  $\mu\text{m}$  in diameter were sifted out. Sieved microspheres were nearly spherical and maintained their shape in water for more than 60 days.

The precipitated fine particles were characterized by XRD for structural determination. Fig. 1 shows X-ray diffraction patterns for the sample, which proved this sample was highly crystalline spinel ferrite crystal structure. There are no detectable traces of extra crystalline. Each peak in the pattern can be indexed on a spinel crystal structure.

T1- and T2-weighted MR images of the magnetic  $\beta$ -glucan microspheres was obtained in various Fe concentrations, such as 0.02 M, 0.002 M, 0.2 mM and 0.02 mM (Fig. 2). The MRI of 0.002 M Fe solution was enhanced images. Therefore, the magnetic  $\beta$ -glucan microspheres have a possibility to be used as MRI-detectable embolic materials.

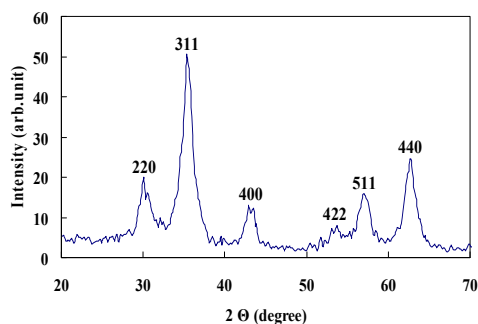


Fig. 1. XRD result of magnetite nanoparticles

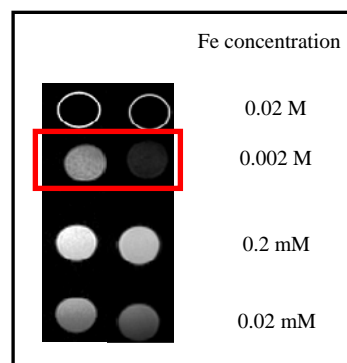


Fig. 2. T1- (left) and T2-weighted image (right) of Magnetic  $\beta$ -glucan microspheres

### References

- [1] Masashige shinkai, J. biosci. bioeng. 94 (2002), 606-613.