

Preparation and evaluation of hydroxyapatite/silicate compositionally graded hollow spherical particles by spray-drying

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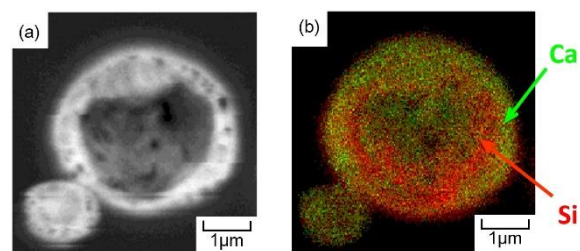
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In the spray-drying method, an aqueous solution containing dissolved materials is sprayed inside a furnace so that the materials undergo instant drying to form a powder. The powder thus formed consists of hollow spherical particles. When the sprayed droplets of the aqueous solution desiccate, the dissolved materials form particle wall, finally, we can obtain the hollow spherical particles.

I have previously reported the dissolution of hydroxyapatite ($\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$, HAp) by blowing CO_2 , and I am investigating the preparation of HAp hollow spherical particles by spray-drying a HAp solution. The obtained HAp hollow spherical particles are 2–5 μm in size and lightweight powder. However, the mechanical strength of these particles are very low. Therefore, I have also reported the preparation of compositionally graded particles by spray-drying. The preparation mechanism of compositionally graded particles is as follows. When two materials with different solubility exist in the solution, low solubility material is deposited by heating first, then, high solubility material is deposited continuously. With adapt this phenomenon in a droplet of spray-drying, it can be obtained compositionally graded hollow spherical particles such as the surface and internal composition with continuously changed. In the lecture, I will explain the preparation and evaluation of the mechanical strength of compositionally graded hollow spherical particles by spray-drying such that the resulting particles consist of HAp particles on the outside and silicate (potassium silicate) particles on the inside that assist mechanical strength.

A high-concentrated HAp solution (0.3 g/dm^3) was prepared to blow CO_2 gas into a HAp suspension. Then, a potassium silicate solution was added 0–0.3 mass% to a high-concentrated HAp solution. The resulting mixture solution was spray-dried at a drying temperature of 100°C , a pressure of 200 kPa. The obtained particles were 2–5 μm in size. The wall thickness of particles without the addition of potassium silicate was approximately 200 nm, and this value was increased to 300 nm by adding potassium silicate. **Fig.1** shows the elemental mapping of the internal structure of HAp/potassium silicate hollow spherical particles observed by electron probe microanalyzer (EPMA). The cross-section EPMA image of the particles indicated that Ca from HAp was distributed on the outer surface and the Si from potassium silicate was distributed in the inner surface of the hollow spherical particles.

The compressive strength of the obtained particles was evaluated by measuring the micro-compressive strength testing equipment of only one particle. The compressive strengths of hollow HAp particle and hollow HAp/potassium silicate particle were approximately 0.6 MPa and 17 MPa, respectively.



(a): SEM imaging, (b): EPMA mapping (green: Ca, red: Si)

Fig. 1 SEM and EPMA mapping of obtained hollow spherical particles.