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SYNTHESIS OF CALCIUM PHOSPHATES BY GAS-SOLID REACTION OF $\text{CaO-P}_2\text{O}_5$

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Calcium phosphates are useful materials in fertilizer, dentifrice, bio-materials, etc., and have been popularly synthesized by a wet method, a hydrothermal method, a flux method, and a solid-state reaction method. However, there has been no report on synthesis by chemical vapor deposition (CVD) method or gas-phase reaction.

In the present study, calcium phosphates were synthesized by gas-solid reaction of CaO solid and P_2O_5 gas. The $\text{CaO/P}_2\text{O}_5$ ratio of starting materials was changed from 5.06 to 0.64, and the reaction was carried out at 400–1100°C. These calcium phosphates were investigated using X-ray diffractometry (XRD) and scanning electron microscope (SEM). By gas-solid reaction of $\text{CaO-P}_2\text{O}_5$, β -MET only was synthesized at 400–500°C, β -MET, HA, and β -PYR at 600–700°C, and β -MET, HA, β -PYR, and β -TCP above 800°C. Synthesized calcium phosphates were independent on the $\text{CaO/P}_2\text{O}_5$ ratio of starting materials but dependent on the reaction temperature.

Keywords: Calcium phosphates; $\text{CaO-P}_2\text{O}_5$ system; gas-solid reaction

INTRODUCTION

Calcium phosphates are useful materials as fertilizer, dentifrice, biomaterials, etc., and have been popularly synthesized by a wet method, a hydrothermal method, a flux method, and a solid-state reaction method.^{1,2} However, there has been no report on the synthesis of calcium

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phosphates by a chemical vapor deposition (CVD) method or gas-phase reaction, though the CVD has been applied to fabricate thin film in the electric field.

In the present study, calcium phosphates were synthesized by reacting CaO solid with P_2O_5 gas, which is vaporized above 350°C .

MATERIALS AND METHODS

Materials

CaO (99.9%) and P_2O_5 (98.0%) powder were used as starting materials. A reaction tube made of quartz (ϕ 13 mm \times 100 mm) was used as a reaction chamber.

Methods

CaO and P_2O_5 powders with 5.06–0.64 of CaO/ P_2O_5 were separately put into the reaction tube, as seen in Figure 1. The tube was heated at 400 – 1100°C in air for 1 hour.

After the reaction, all products were investigated using X-ray diffraction (XRD, Rigaku Corp., RINT2200H), and scanning electron microscopy (SEM, JEOL, JSM-5310LVB).

RESULTS AND DISCUSSION

Figure 2 shows a SEM photo after the reaction by CaO/ P_2O_5 with 0.64 at 900°C . In this photo, natural shape crystal cannot be seen.

Table I shows synthesized calcium phosphates by gas-solid reaction. β -calcium metaphosphate (β -MET) only was synthesized at 400 – 500°C , β -MET, hydroxyapatite (HA) and β -calcium pyrophosphate (β -PYR) at 600 – 700°C , and β -MET, HA, β -PYR, and β -tricalcium phosphate

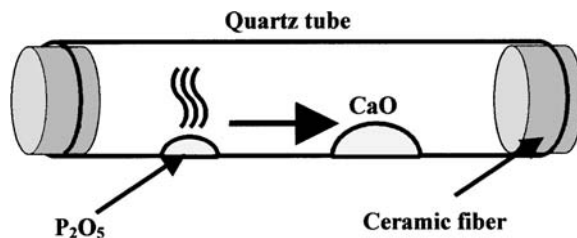


FIGURE 1 Scheme of reaction tube.

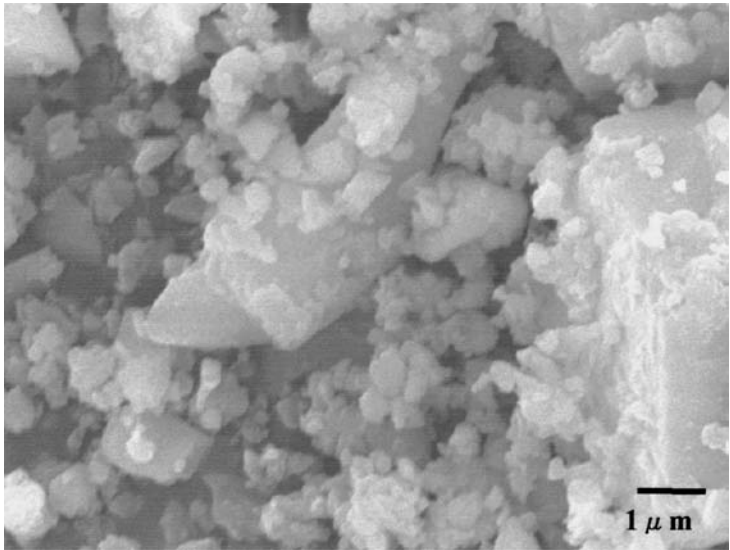
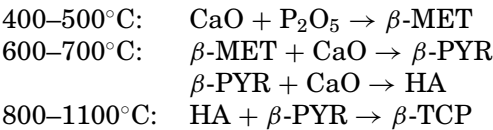


FIGURE 2 SEM photo of powder by gas-solid reaction.

(β -TCP) above 800°C. The calcium phosphates were independent on $\text{CaO}/\text{P}_2\text{O}_5$ of starting material. From these results, these reactions indicate as follows.



By these reactions, multilayers of calcium phosphates were formed above 800°C.

TABLE I Calcium Phosphates by Gas-Solid Reaction

	CaO/P ₂ O ₅				
	5.06	2.54	1.26	0.84	0.64
400–500°C	●	●	●	●	●
600–700°C	●■▽	●■▽	●■▽	●■▽	●■▽
800°C	○▽	●■○▽	●■○▽	●■○▽	●■○▽
900°C	●■○▽	●■○▽	●■▽	●■○▽	●■○▽
1000°C	■○▽	●■○▽	●■○▽	●■○▽	■○▽
1100°C	■○▽	■○▽	■○▽	■○▽	■○▽

●: β -MET, ■: β -PYR, ○: β -TCP, ▽: HA.

CONCLUSIONS

- By gas-solid reaction of $\text{CaO-P}_2\text{O}_5$, β -MET only was synthesized at 400–500°C, β -MET, HA, and β -PYR at 600–700°C, and β -MET, HA, β -PYR, and β -TCP above 800°C.
- Calcium phosphates synthesized by gas-solid reaction were independent on $\text{CaO/P}_2\text{O}_5$ ratio of starting materials but dependent on reaction temperature.

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