

Research Article

Effect of Test Circumstances on Compressive Strength of Porous Calcium Phosphate Ceramics for Establishment of Standard Measurement Condition

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Abstract In order to establish a reasonable biomimetic condition for compressive strength of bioactive ceramics, the compressive strengths of several commercial bioactive ceramics were measured under various conditions. All specimens except for one of Japanese ceramics, specimen A, showed no significant differences among the conditions, but showed a tendency of decreasing the compressive strength more than 10% in comparison to the dry condition that was conventionally described in product brochures. Further, the specimen A indicated a significant decrease of the compressive strength in 24 h soaking conditions. Accordingly, a recommended biomimetic condition would be that specimen would be soaked in PBS after deaeration at 25 °C for 24 h and measured in air after brief wiping surface liquid.

Keywords bioactive ceramics; porous body; test circumstance; compressive strength; standardization

1 Introduction

Porous bioactive ceramics are widely used as bone fillers due to their excellent osteoconductivity. Although these bioactive ceramics do not have enough mechanical strength against load in daily life, mechanical strength measurement could be important to understand the nature of the implant materials for both scientists and clinicians. Therefore, mechanical properties of commercial bioactive porous ceramics are generally described in their brochures. On the other hand, bioactive ceramics are usually used in body-fluid wet circumstances. In general, the mechanical strength of ceramics decreases by soaking [1]; thus the mechanical strength of bioactive ceramics under biomimetic condition could be much more important as a practical value. In addition, providers who prepare/sell the materials desire the simplest condition as possible for reducing

unnecessary costs. Therefore, establishment of a good standard biomimetic condition for the bioactive ceramics is desired to compare the mechanical properties not only in domestic products but also among international products. In the present study, compressive strengths of the bioactive ceramics from different companies were measured under various wet conditions, and compared to the measurement conditions to decide a fair standard method for compressive strength test.

2 Materials and methods

Five kinds of bioceramic specimens, A, B, D, E and F, with the same spec as the each commercial product were provided by Japanese cooperative companies. One overseas product, G, was also purchased. The specimens A, B, D, E and F were provided in an approximately the same dimension: $\phi 5 \times h 10 \text{ mm}^3$ in size. The specimen G was cut into approximately $10 \times 10 \times 10 \text{ mm}^3$ in size. All specimen sizes were precisely measured using a micrometer before soaking. All five specimens were soaked in PBS by following two methods:

- (1) specimens were soaked in the PBS and deaerated by a vacuum pump (SV);
- (2) specimens were placed in the beaker in the vacuum desiccators and deaerated, and PBS was added to the beaker under vacuum (VS).

Soaking times were 1 h and 24 h, and temperatures were 37 °C and 25 °C. Compressive strengths were measured with a universal testing machine, AGS-1kN (Shimadzu Co.), with a specially equipped liquid circulation apparatus (Shimadzu Co.) at a crosshead speed of 500 $\mu\text{m}/\text{min}$ by following two methods:

- (1) specimens were measured under a circulation of warm PBS;

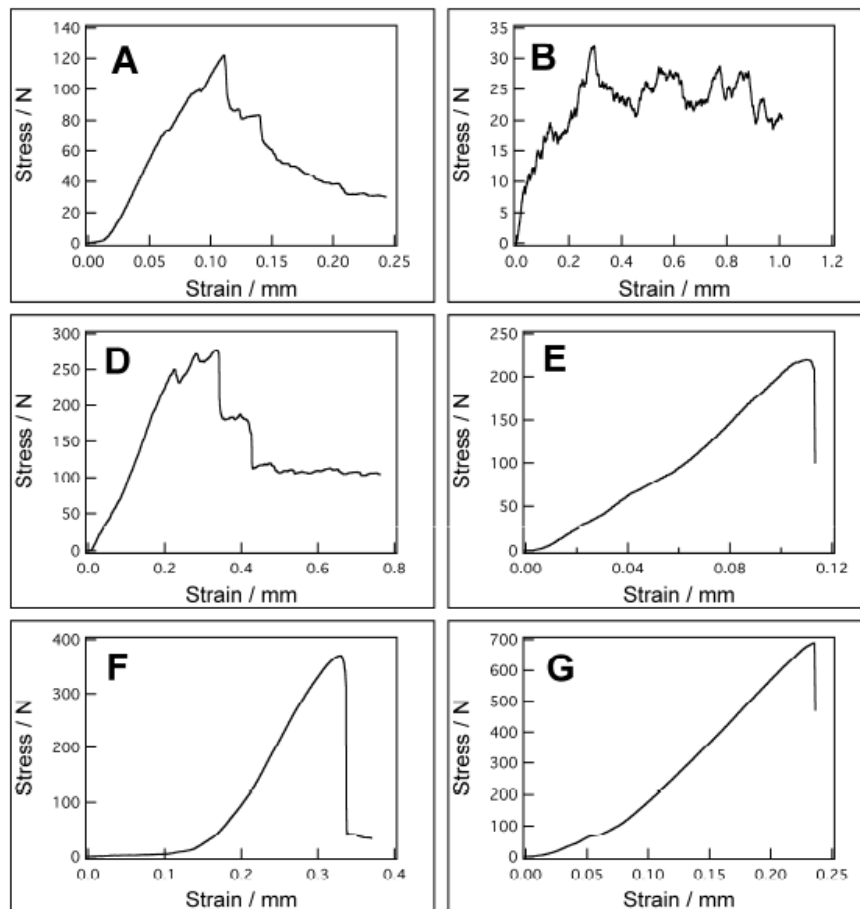


Figure 1: Typical stress-strain curves for specimens.

(2) specimens were measured in air after brief wiping surface liquid out by Kim Wipe.

Compressive strengths obtained were statically analyzed by one-way ANOVA using KaleidaGraph 3.6J for Mac.

3 Results and discussion

Typical stress-strain (S-S) curves for all specimens are shown in Figure 1. Specimens A, B and D indicated a stepwise collapse due to the stepwise collapse of the weaker part in each pore wall. Specimens E, F and G revealed a dense body-like S-S curve, i.e. their pore wall would have a similar structure to dense ceramics. Accordingly, specimens after the compressive test showed different shapes as shown in Figure 2. The results of the compressive strength were summarized in Figure 3. Variances of the compressive strengths of specimens were comparatively large and no significant differences among measurements were detected except for several conditions of specimen A. Even with the naked-eye observation, specimen G was not homogeneously prepared, e.g. very large pores were observed in some

specimens but other specimens showed a very few pores; thus, no trend was detected. Specimen E also indicated no trend, even the E had a homogenous structure. This could depend on its chemical composition: Si-base glass ceramics instead of calcium phosphate. Except E and G, all specimens decreased their compressive strength in trend more than 10% by soaking in PBS. Significant differences between dry and 24 h soaking conditions were detected for the specimen A, even the variance was as large as other specimens. The specimen A was composed of β -tricalcium phosphate and higher dissolution rate in comparison to hydroxyapatite. It could be the reason why the specimen A only showed the significant decrease of strength. However, no significant differences were found even for the specimen A when the specimens were soaked in distilled water, which was reported in the annual meeting of the "Standardization for Characterization of Bioactive Ceramics" Project. These results suggested that reasonable conditions for compressive strength of bioactive ceramics in initial stage of implantation could be in air with brief removing of surface liquid after soaking in PBS for 24 h at 25 °C by addition of PBS after deaeration of the specimens.

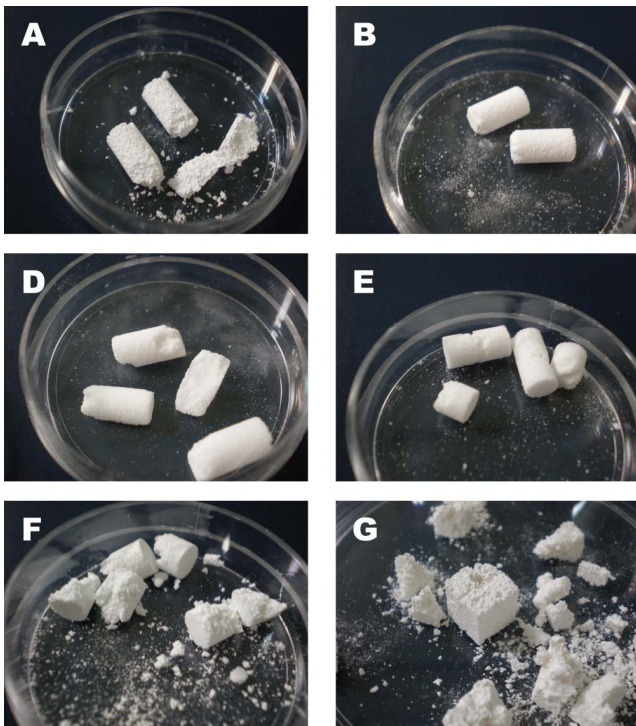


Figure 2: Typical shapes of specimens after compressive test.

in PBS could be informative for surgeons. Further, the reasonable biomimetic measurement conditions would be that the specimen would be soaked in PBS after deaeration followed by 24 h soaking at 25 °C and measured in air after brief wiping surface liquid out.

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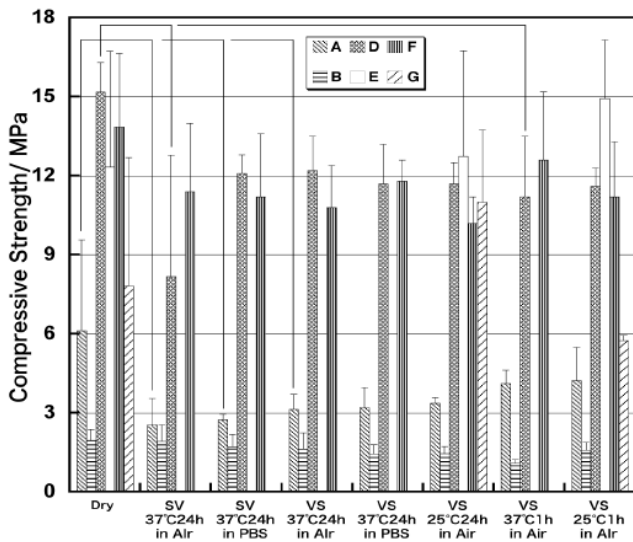


Figure 3: Results of compressive strength tests under various conditions.

4 Conclusions

Bioactive calcium phosphate ceramics decreased their mechanical strength more than 10% when the surgeon placed them on implant site. Thus, changing the behavior of mechanical properties of bioactive ceramics after soaking