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Study of the mechanical properties of a ceramics of orthopedic and dental use

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Abstract. The study of the preparation of ceramics is consisted in sintering a hardened cement, wich is composed of mixture of three calcium phosphates : tetracalcium phosphate ($Ca_4(PO_4)_2O$), α-tricalcium phosphate (α-$Ca_3(PO_4)_2$) and monocalcium phosphate ($Ca(H_2PO_4)_2.H_2O$). After wetting, the cement evolved towards an apatitic phase while hardening. To check the effect of certain factors on the resulting ceramics, a complete central composite design of four variables (liquid/solid ratio, stoichiometric coefficient of monocalcium phosphate, rate of increase in temperature and duration of landing of termpertaure) was set up. The equation of the model and optimal conditions were defined.

1. INTRODUCTION

Calcium phosphates are known since a long time as biocompatible materials. That is why they are used as bioceramics for prosthetic application [1]. They are based mainly on hydroxyapatite ($Ca_{10}PO_4)_3(OH)_2$) and β-tricalcium phosphate ($Ca_3(PO_4)_2$).

The hydroxyapatite presents a particular interest, because of its structure which is like that of a bone's mineral part [2]. It has thus been used, for a long time, with success in orthopaedic and dental surgery.

Recently, cements evolving towards a final apatitic phase, have, been a subject of considerable interest in orthopaedic and dental applications [3,4]. They lead to a material that forms solid hydroxyapatite-like calcium phosphate.

These cements are either used directly for the bone filling and indeed they harden at the application site [5], or ceramicised at high temperature to make pieces of different forms in medical field [6].

The intermediate stage of the cement parmits the mouling of the piece. After hardening, the piece can be easily handed and machined.

In this work we describe a new methode for the preparation of calcium phosphate ceramics and we optimize conditions in obtaining ceramics.

2. CERAMICS PREPARATION AND OPTIMIZATION

The cement powder was prepared through the mixture of three calcium phosphates: tetracalcium phosphate ($Ca_4(PO_4)_2O$), α-tricalcium phosphate ($Ca_3(PO_4)_2$) and monocalcium phosphate ($Ca(H_2PO_4)_2.H_2O$).

With a certain quantity of water, this mixture evolves towards a hydroxyapatite following the reaction below:

$$a\,Ca(H_2PO_4)_2.H_2O + b\,Ca_3(PO_4)_2 + c\,Ca_4(PO_4)_2O \rightarrow Ca_{10}(PO_4)_6(OH)_2$$

Once the cement setled and hardened, was set at a temperature of 1250°C. Preliminary study allowed to choose factors which have an influence over the ceramicization of the cement.

The relationship between the shrinking of the ceramics and four quantitative variables: liquid/solid ratio (x_1), stoichiometric coefficient of monocalcium phosphate (x_2) (that means the molary proportion of monocalcium phosphate in the initial mixture), rate of increase in temperature (x_3) and duration of landing of temperature (x_4) (that means the time during which the temperature remains unsistent), was dertemined by a polynomial of the second degree in a set of experiments according to a complete central composite design[7].

Table 1 represents the working ranges which are obtained by varying each factor individually while keeping the other factors unchanged.

Table 1. The working ranges.

natural variables x_j	coded variables X_1, X_2, X_3, X_4				
	-2	-1	0	1	2
x_1 = liquid/solid(cc/g)	0.40	0.45	0.50	0.55	0.60
x_2 = stoichiometric coefficient	0.01	0.173	0.336	0.499	0.662
x_3 = rate of increase in temperature (°C/mn)	1	8	15	22	29
x_4 = duration of landing of temperature (h)	2	4	6	8	10

Table 2 shows the complete central composite design presented according to the standard order and the experimental data for shrinking of the ceramics.

Table 2. The complete central composite design and experimental data.

experiments	coded units of variables				shrinking of the ceramics (%)
	X_1	X_2	X_3	X_4	
1	−1	−1	−1	−1	10.35
2	+1	−1	−1	−1	10.80
3	−1	+1	−1	−1	13.55
4	+1	+1	−1	−1	13.25
5	−1	−1	+1	−1	11.53
6	+1	−1	+1	−1	11.50
7	−1	+1	+1	−1	14.40
8	+1	+1	+1	−1	13.89
9	−1	−1	−1	+1	12.03
10	+1	−1	−1	+1	11.90
11	−1	+1	−1	+1	14.40
12	+1	+1	−1	+1	15.40
13	−1	−1	+1	+1	12.67
14	+1	−1	+1	+1	13.79
15	−1	+1	+1	+1	14.65
16	+1	+1	+1	+1	14.95
17	−2	0	0	−2	14.49
18	+2	0	0	+2	13.35
19	0	−2	0	0	11.53
20	0	+2	0	0	14.20
21	0	0	0	0	12.50
22	0	0	0	0	14.55
23	0	0	−2	0	12.85
24	0	0	+2	0	14.50
25	0	0	0	0	13.70
26	0	0	0	0	13.90
27	0	0	0	0	13.40
28	0	0	0	0	13.60
29	0	0	0	0	13.70
30	0	0	0	0	13.90
31	0	0	0	0	13.80

The 15 terms (b_0, b_i, b_{ij}) which represent the coefficients of the equation model, were calculated by substituing data values in the expressions for the least-squares estimates of the coefficient (table 3). The relation between the response and the different factors is so given:

$$S(\%) = 13.714 + 1.05X_2 + 0.41X_3 + 0.58X_4 - 0.28(X_2)^2$$

Table 3. Analysis of variables effect.

Coefficient		T (Student)	significance
b_0	13.71		
b_1	−0.02	−0.134	non-significant
b_2	1.05	8.922	**significant**
b_3	0.41	3.461	**significant**
b_4	0.58	4.881	**significant**
b_{11}	−0.02	−0.187	non-significant
b_{22}	−0.28	−2.628	**significant**
b_{33}	−0.12	−1.101	non-significant
b_{44}	−0.08	−0.754	non-significant
b_{12}	−0.06	−0.398	non-significant
b_{13}	−0.01	−0.061	non-significant
b_{14}	0.17	1.159	non-significant

3. DISCUSSION

The modelisation of the response "shrinking of the ceramics" shows that monocalcium phosphate stoichiometric coefficient has a positive effect on the shrinking response, whereas its square has a negative influence. The early described shows that in order to have a homogeneous ceramics and a convenient setting time, the monocalcium phosphate stoichiometric coefficient must be fixed to its minimal natural value (we have already fixed it to 0.06: X_2 = -1.69). The model equation is written as the following:

$$S(\%) = 11.14 + 0.41X_3 + 0.58X_4$$

The response "shrinking of th ceramics" is a lineal fonction of the factors "rate of increase in temperature (X_3)" and "duration of landing of temperature (X_1). In order to have a maximal shrinking, we must fix these factors to their maximal values. Table 4 summarizes the optimal conditions in according with the shrinking of the ceramics.

Table 4. Optimal field of the explicative variables.

Factors	optimal field
liquid/solid ratio	0.50 - 0.58 cc/g
stoichiometric coefficient of monocalcium phosphate	0.06
rate of increase in temperature	29°C/mn
duration of landing of tremperature	10 h

4. CONCLUSION

It is possible to prepare dense bioceramics for orthopaedic applications which possesse suitable structural, mechanical and biological properties. The preparation conditions of these ceramics have been studied using a plan of experiments. We have, then, been able to optimize these conditions using the model equation that links the response with four studied factors.

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