INFLUENCE OF THE COMPOSITION ON SETTING TIME AND POROSITY IN HYDROXYAPATITE CEMENTS WITH ALGINATE AND CHITOSAN

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***Abstract*— Twenty formulations of synthetic hydroxyapatite cements (HAC) of two different types are presented. The evaluation of the setting time and the porosity has been done through an experimental design. Type A cements consist in a mixture of hydroxyapatite (HA) and gypsum (GS) as a solid part (P), and a potassium phosphate solution (KP) as a liquid part (L). The sodium alginate (SA) powder was dissolved or in the solid part or in the liquid one. The setting times of the Type A cements vary from 8 to 16 min, which are similar values to report in the literature. The porosity values found (41-43 %) were not influenced by the changes evaluated. The best of type A cements was that prepared with 0.5 wt % KP and dissolving the SA in the solid part. The solid part of type Q cements consist in a mixture of HA - non sintered (ns-HA) or sintered (s-HA)- , a calcium generator -calcium oxide (CaO) or ß-tricalcium phosphate (ß-TCP)-, and an inorganic material which acts as agglutinant - zinc oxide (ZnO) or magnesium oxide (MgO)-. The liquid phase of type Q cements is formed by an acid solution of chitosan, in malic (MH) or succinic (SH) acid. The setting times observed for these cements are ranged between 2 and 24 min. The use of MgO instead of ZnO as agglutinant decreases the setting time of the cements. The same effect was observed when the succinic acid was substituted by malic acid as dissolution medium of the chitosan. The porosity values for type Q cements vary between 50 and 70 %.**

***Keywords*— hydroxyapatite cements, sodium alginate, chitosan, setting time, porosity, polymers.**

I. INTRODUCTION

Calcium hydroxiapatite [$Ca_{10}(PO_4)_6(OH)_2$] (HA) is well known as the primary constituent of bone and teeth of animal organisms (Jarcho *et al*., 1977; Katz and Harper, 1986). Many papers have been published about the use of materials based on HA as bone substitutes in medical and dental treatments. Specially, clinical dental applications include the maintenance of periodontal defects (Ogilvie *et al.*, 1987 and Cranin *et al*., 1987a), the implantation into tooth extraction sockets to conserve alveolar ridge height (Denissen and de Groot, 1979; Scheer and Boyne, 1987), and the augmentation of a deficient alveolar ridge to improve denture support and stability (Cranin *et al.*, 1987b; Kent *et al.*, 1986; Larsen *et al*., 1987 and Rothstein *et al*., 1984). However, when the implantation of HA in the animal organism is performed, the particles are loose and can migrate beyond the intended regions. Moreover, the stabilization of the material seems to require at least one month. The setting occurs in some extent during this time, with the consequent reduction in the implant size (Kawakami *et al*., 1992).

The hydraulic cements consist of a powdered mixture able to form a plastic paste upon mixing with water or acid solution, commonly phosphoric acid, and hardening with time as a result of the hydration reactions, giving a new form of calcium phosphate (Lemaitre *et al*., 1992). The results of these reactions are in some cases calcium deficient hydroxyapatite or dehydrated dicalcium phosphate (Mirtchi *et al*., 1989). However, the exposure of this paste to blood just after mixing results in the decay of the cement, the situation being similar to that observed for the gypsum. The gypsum sets when is mixed with water; however, the gypsum paste also decays gradually when immersed in water just after mixing. The decay is not a problem when the cement paste is used in areas where there is no liquid in contact with the cement, such as in root filling materials. The problem of decay of the cement limits in any case the potential wide applications of this bioactive material (Ishikawa and Asaoka, 1995).

In order to increase the application of this type of materials new formulations have been recently developed, in which different synthesis parameters have been modified. Thus, the addition of sodium alginate to the liquid part in the preparation of a non-decay calcium phosphate cement of fast setting produces a decrease of the setting time value in a factor of eight (Ishikawa *et al*., 1995). In other way, the solid to liquid ratio and the pressure applied to prepare the cement disc exert an inverse influence on the porosity, *i.e.*, an increase of the pressure produces a decrease of the diametral tensile strength, caused by a low number of pores (Lemaitre *et al.*, 1992). Other factors, such as the solid to liquid ratio and the zinc oxide concentration exert a direct influence upon the setting time of cements containing chitosan.