

Bioactive calcium-containing gel and ceramic systems

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Calcium silicate ceramics are intended to apply as implant materials, usually for bone substitution. The aim of the present work was to study the relationship between synthesis conditions (chemical compositions; the type and concentration of catalysts; the temperature and time of aging and heat treatment; etc.) and the structure of calcium silicate and calcium silicate phosphate materials. The ceramics were produced by a low energy-consuming sol-gel method. Without any catalyst, optically clear but strongly fragile gel samples can be obtained, the gelation process demands a long aging-drying time. The application of acetic acid leads to formation of transparent, homogeneous gel systems. Due to the fast hydrolysis in acidic medium loose, randomly branched structures can be obtained. In the presence of ammonia catalyst, a basic calcium salt containing sol forms. Base-catalyzed reactions yield more dense materials due to the longer hydrolysis time, which can provide the possibility for the aggregation of particles into the most thermodynamically stable arrangement. In order to increase the bioactivity phosphate content were built up into the system. In the case of phosphoric acid also a calcium phosphate precipitate forms.

The gel and sol products derived from sol-gel process must be dried (at 80 °C) and heated (at 700 °C). The decomposition processes finish between 415 and 665 °C; the calcium silicate bond system evolves above 500 °C; the temporary phase of calcium carbonate disappears until 500 °C; and the mechanical strength increase considerably above 500 °C. The Ca ions do not bond only to the silicate matrix or silicate anions in the crystalline phases, but also to the carbonate as well as OH ions in the range of 160 – 500 °C. Independently from the type and volume of the catalyst, the crystalline phase is always β -dicalcium silicate in the range of 700-800 °C.

Ammonia catalysis (1 and 10 mole ratio) resulted the best bulk ceramics with good mechanical strength and low solubility.

In the present work we determined the FTIR position of Ca-O-Si bonds in various systems. The Si-O-Ca vibration appears at 965 cm^{-1} in the IR spectrum of dicalcium silicate hydrate; at 930 cm^{-1} in that of β -dicalcium silicate; at 890 cm^{-1} in that of mono-calcium silicate and 920-930 cm^{-1} in the amorphous calcium silicate samples. Comparing the ^{31}P MAS NMR and XRD data we can identify that the peak at -9 ppm can be attributed to β - $\text{Ca}_2\text{P}_2\text{O}_7$ and the peak at -11 ppm to the γ form; the wide peak at -25,5 ppm to δ - $\text{Ca}(\text{PO}_3)_2$ and at -33 ppm to Si-O-P in $2\text{Ca}_2\text{SiO}_4 \cdot \text{Ca}_3(\text{PO}_4)_2$ phase.