Setting and Strength of Brushite Cements with Varying Reactant Particles

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Objectives:

Brushite cements have potential for both bone filling and dentine re-mineralisation as they can convert into hydroxyapatite in vivo. The aims of this study were to quantify effects of varying cement reactant particle source on setting kinetics and strength and assess if these can be explained by changes in particle size and structure.

Methods:

The size and morphology of particles of monocalcium phosphate monohydrate (MCPM) and 6 different β-tricalcium phosphates (TCP) were assessed using scanning electron microscopy (SEM). Equimolar TCP and MCPM were pre-mixed (for 6 or 11 days) or freshly mixed prior to reaction with 800 mM aqueous citric acid at a powder: liquid ratio of 3.3:1. Subsequent brushite formation rate was assessed through ATR-FTIR, final brushite crystal structure by SEM and flexure strength at 24 hours using a ball-on-ring test method.

Results:

SEM indicated TCP particles ranged from 4 to 30 microns and had varying porosity. Those of MCPM were ~ 30 micron irregular dense chips. All formulations gave a delay period before reaction providing working time followed by rapid conversion and set. Delayed setting and slower reaction was observed if powders were not freshly-mixed (see Figure 1). SEM illustrated this was due to dry powder reaction between MCPM and TCP leading to formation of undesirable anhydrous di-calcium phosphate rather than brushite. Larger and / or denser TCP particles reacted more slowly as might be expected with slower dissolution but did not affect final brushite crystal structure. Flexural strength ranged between 20 and 40 MPa and was higher with larger TCP size.

Conclusions:

Pre-mixing particles detrimentally affects potential shelf-life of brushite cements. Reactant particle size and structure must be carefully controlled in order to maintain optimal handling, setting and mechanical properties.