**Fiber Reinforced Calcium Silicate Phosphate Bone Cements**

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**Statement of Purpose:** Tricalcium silicate (C3S) based cements have higher strength than calcium phosphate cements and better biological properties that PMMA based bone cements. C3S cement is self-setting, hydraulic, bioactive and osteoconductive [1,2]. It hydrates to calcium silicate hydrate (CSH) gel and calcium hydroxide (CH). In vivo study has shown its ability to regenerate bone; ~50% resorption of the cement was measured after 12 months [3]. However, setting C3S has high pH (~11), long setting time (~3 h) and relatively low fracture toughness (~0.1 MPa m1/2). Addition of monocalcium phosphate monohydrate (MCPM) decreases pH and enhances bioactivity of the cement [4], by reacting with CH to precipitate amorphous and crystalline phosphates. We hypothesize that cement toughness can be increased by micro-fiber reinforcement. Due to the slow rate of cement resorption, it is anticipated that the fibers will be embedded within the newly grown bone.

**Methods:** Tetraethyl orthosilicate and calcium nitrate tetra hydrate aqueous solution were used in a sol-gel synthesis followed by firing at high temperature to obtain C3S powder. All chemicals were reagent grade. Four groups were prepared: (C) is C3S; (C-10M) is C3S with 10 wt% MCPM; (C-3CF) is C3S with 3 wt% of 7 μm diameter/3 mm long carbon fibers; and (C-10M-3CF) is C3S with both 10 wt% MCPM and 3 wt% of the same carbon fibers. After mixing (at water/powder ratio ~0.4), the cements set for 10 days at 37°C. Fracture toughness (KIC) was measured using the notchless triangular prism (NTP) specimen test [5]. Compressive strength was determined on 12mm tall by 6mm diameter cylindrical samples. At least three samples were tested to determine the average strength and toughness. The samples were further characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), and densitometry.

**Results:** The presence of un-reacted C3S was determined in all samples by XRD phase analysis. Portlandite (CH) represents the degree of the hydration reaction of calcium silicate, as CSH gel is amorphous and thus does not have distinctive XRD pattern. In set C-10M samples, the relative intensity of the C3S to CH peaks was lower than in pure C3S, which could be attributed to the reaction of CH with MCPM. There were also monetite peaks in C-10M pattern, which could be an intermediate phase before formation of hydroxyapatite, the most thermodynamically stable phase in this system. Table-1 shows that significant improvement in the compressive strength and KIC was obtained after addition of carbon fibers to both C and C-10M samples. Addition of CF has not considerably affected the porosity of C3S, but MCPM has increased the porosity in C3S. Reaction of the CH and MCPM results in formation of water [4], which may lead to increased porosity.

<table>
<thead>
<tr>
<th>Sample Name</th>
<th>Strength (MPa)</th>
<th>KIC (MPa m1/2)</th>
<th>Porosity (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>54.8±0.60</td>
<td>0.120±0.017</td>
<td>37.7±0.1</td>
</tr>
<tr>
<td>C-10M</td>
<td>50.9±4.63</td>
<td>0.197±0.020</td>
<td>45.4±0.6</td>
</tr>
<tr>
<td>C-3CF</td>
<td>88.3±5.90</td>
<td>0.487±0.025</td>
<td>37.3±0.9</td>
</tr>
<tr>
<td>C-10M-3CF</td>
<td>80.2±6.28</td>
<td>0.398±0.054</td>
<td>41.8±0.4</td>
</tr>
</tbody>
</table>

**Conclusions:** Carbon fiber reinforced calcium silicate phosphate cements were processed and evaluated. Compressive strength increased by factor 1.5-1.7 and KIC increased by factor 2.0-2.5 upon addition of 3 wt% of the fibers. The dominating toughening mechanism appears to be fiber pullout, in which the energy is dissipated by the frictional work done in the fiber-matrix interface during the pull-out [6]. To our knowledge, this is the first time that the fiber reinforced calcium silicate phosphate composite cement have been processed and characterized. We believe that the C-10M-3CF cements, with relatively good mechanical properties (~80 MPa compressive strength and KIC<0.4 MPa m1/2) could be applied as bone cements. The follow-up of this research will include biocompatibility and osteoconductivity evaluations by in vitro tests, resorption rate determination in vitro and in vivo, and the use of accelerators to control the cements’ setting time.

**References:**


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