Handling and mechanical properties of collagen -brushite composite Balamurugan Kumarasami <sup>(1),</sup> Charels Doillon<sup>(2)</sup>, Uwe Gbureck <sup>(3)</sup>, Jake Barralet <sup>(1)</sup>

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## Introduction:

Many studies have been conducted to improve and measure the cohesion of, mainly, apatite cement through addition of several additives and methods (1-2). In this study, we investigated the possibility of using collagen to improve the cohesion of brushite cement and quantitatively measured the amount of particles released into water at various powders to liquid (P/L) ratios. The results indicated that the addition of collagen (0.8 wt %) to brushite cement at powder to liquid ratios of 2.3, 3.5 g/ml resulted in 9 fold and almost 2 fold reductions in the % of particles released respectively as compared with brushite cement controls. Dry compressive strength (P/L – 3.5 g/ml) values were also found to be improved compared with collagen free controls.

## Materials and methods:

The cement consisted of an equimolar mixture of -  $\beta$  TCP and commercially available monocalcium phosphate hydrate (Baker, Germany). The  $\beta$ -TCP was synthesized by heating a mixture of monetite (DCPA; Mallinckrodt Baker, Griesham, Germany) and calcium carbonate (CC; Merck, Darmstadt, Germany) to 1050°C for 24 h followed by quenching to room temperature in a desiccator. The product consisted of phase pure and highly crystalline  $\beta$ -TCP as verified by X-ray diffraction. The sintered cake was crushed with pestle and mortar and passed through a 355µm sieve and milled for 1 h. The rat tail tendons were dissolved in acetic acid (0.02N) and freeze dried and re-dispersed in 20mM citric acid.

The collagen (0.8 wt %) was mixed with brushite cement, for 90 s, at a powder to liquid ratio of 3.5 g/ml, then immediately cast into a plastic mold (13 mm diameter with 4 mm height). The total citric acid content was kept at 0.8 M by adding extra citric acid powder to the (0.1M citric acid) collagen. After a total time of 2 minutes post mixing, the plastic mold was suspended in water at 37 °C for 30 minutes to allow the collection of released particles permitting the wt % of particles released to be calculated. For compressive strength measurement the samples were cast in a teflon mold (12mm height and 6 mm diameter). After 4 hr, the samples were removed and dried or stored in water at 37 °C for 20 hr, prior to the measurement of compressive strength using an Instron testing machine (5569) at a strain rate of 1 mm / minute.

**Results/Discussion:** Figure 1 shows the variation of weight % of particles released from brushite, collagen modified brushite cement, with respect to various powders to liquid ratios. For control group, the results indicated that the amount of particles released, decreased

as P/L increased, where as for collagen, irrespective of P/L ratio, only negligible quantities of particles were released at all P/L. After immersion less than 2 minutes after mixing, the control samples disintegrated whereas collagen containing formulations retained their shape and continued to set fully. Remarkable improvement in the dry compressive strength value was observed with the addition of collagen as indicated in Figure 2. This may be due to the collagen physically binding the calcium particles together. Reduction in wet strength values were observed for both control as well as collagen modified brushite groups and any benefit of collagen was lost. We are currently working on the mechanisms to retain the dry strength value of collagen modified brushite during in wet conditions.



Figure 1. Weight % of particles released from brushite (control) and collagen modified brushite.



Figure 2. Dry and wet Compressive strength values of brushite (control) and collagen modified brushite.

## **Conclusion:**

Addition of collagen to brushite cement has not only resulted in the improvement of dry compressive strength but also improved its cohesion property allowing settling to occur while submerged after as little as 2 minutes after mixing without disintegration.

## **References:**

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