The effects of fiber length and loading on the handling and mechanical properties of a calcium phosphate material.

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Statement of Purpose: Incorporation of a bioabsorbable material (82:18 PLLA-PGA) into a ceramic matrix (CPC) was investigated to improve the toughness of the resultant biomaterial. This test was completed to determine the bending strength, flexural load at fracture, initial load at fracture, and work of fracture of samples reinforced with 1, 2, and 4 mm fibers. The test will be used to evaluate the effect of fiber reinforcement on the mechanical properties of calcium phosphate cements.

Methods: Calcium phosphate cement powders were a blend of α-tricalcium phosphate, calcium carbonate, and monocalcium phosphate monohydrate. Suture materials were cut to the various lengths using a fiber cutter designed internally for this function. Cut fibers were incorporated into the ceramic powders in the blends as shown in Table 1. The CPC powders and the various fiber loaded calcium phosphate cement powders (FCPC) were portioned into pouches. The solution component used to hydrate this mixture to yield a flowable paste is a dilute sodium hyaluronate solution that is manufactured aseptically. Mechanical testing included four-point bend testing[1], and handling testing included ability to be mixed and injected, as well as an assessment of cohesion when placed in a large volume of phosphate buffered saline.

Results / Discussion: Test results from this experiment are summarized below in Table 1. Beam strength and flexural load increase up to 4 times for FCPC samples compared to plain CPC. Formulations containing 1mm fibers were found to be significantly stronger than plain CPC, with a clear correlation between increased fiber loading and higher strength.

Work of fracture shows the greatest improvement from fiber loading, presumably because of the crack-bridging effect of the fibers (see figure 1). 1%, 2%, 3% and 4% fiber loadings give increases in work of fracture of about 25, 50, 80, and 100 times over plain CPC, respectively.

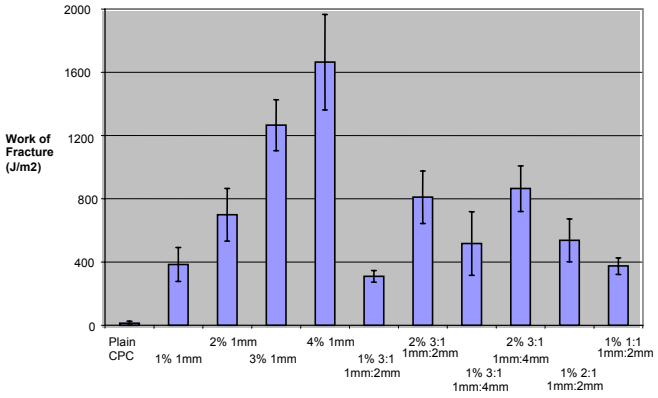
Blends of 1mm and longer fibers (2mm and 4mm) were compared to pure 1mm fibers for their effect on flexural properties. All of the blends have significantly higher strength and work of fracture than plain CPC. However, the blends do not appear to offer much advantage when compared to formulations with equal loadings of pure 1mm fibers. Only blends containing 25% 2mm and 4mm fibers show better strength than equal loadings of pure 1mm fibers. Blends with higher ratios (33% and 50%) unexpectedly did not show any improvement. Also unanticipated, adding longer fibers did not result in any improvement in work of facture.

Mixing of all compositions was possible but increased difficulty of homogenizing with increasing fiber length was observed. Assessment of washout in large volumes of 37ºC PBS suggested that fiber lengths greater than 1 mm led to breakdown in solution.

Table 1: 4-Point Bend Test Results

	Beam Strength	Flexural Load	Initial Peak	Energy	Work of Fracture
	(MPa)	(N)	(MPa)	(mJ)	(J/m^2)
Plain CPC	3.18	3.82	3.18	0.16	13.51
1% 1mm	3.64	4.36	3.25	4.62	385.19
2% 1mm	5.38	6.46	3.72	8.39	699.16
3% 1mm	8.74	10.48	6.00	15.18	1265.38
4% 1mm	11.32	13.58	5.74	19.97	1664.51
1% 3:1 1mm:2mm	5.73	6.88	5.30	3.72	310.25
2% 3:1 1mm:2mm	6.19	7.43	4.57	9.72	810.27
1% 3:1 1mm:4mm	6.11	7.33	5.48	6.21	517.75
2% 3:1 1mm:4mm	6.04	7.25	5.00	10.38	864.66
1% 2:1 1mm:2mm	5.37	6.44	5.01	6.45	537.58
1% 1:1 1mm:2mm	5.54	6.65	5.45	4.50	375.28

Figure 1: Work of Fracture of Various Mixtures



Conclusions: Beam strength, flexural load at fracture, initial load at fracture, and work of fracture were established for ten fiber loaded samples in addition to plain CPC. It was shown that loading CPC with short bioabsorbable fibers gives a significant increase in both bending strength and work of fracture. This testing also demonstrates a clear correlation between increasing fiber loading level and increasing strength and work of fracture up through 4% loading of 1mm fibers. Blending 1mm fibers with longer fibers (2mm and 4mm) was shown to give little or no improvement in strength and work of fracture over the use of 1mm fibers only.

References: 1. ASTM C1161-02c Flexural Strength of Advanced Ceramics at Ambient Temperature.