Wet spinning and Characterization of Collagen Fibers Incorporating Hydroxyapatite in Mixed and Coated Form

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Statement of Purpose: The composition of natural bone comprises of organic collagen (Cg) and inorganic calcium phosphate particularly hydroxyapatite (HA), acting as reinforcement. Mineral HA is an osteoconductive material that provides a temporary scaffold for bone growth. The osteoconductive nature of HA coatings results in the formation of strong bonds with bone.

The present study was focused on incorporating mineral HA in collagen. Wet-spinning technique was applied to form Cg fibers with 2.5% dispersion and Cg/HA composite fibers. The osteointegration of a biomaterial depends not only on the properties of the material but also on the surface conditions. Collagen has a function of regulating the distribution of HA by preventing the aggregation of small HA particles Coating of HA crystals on pure Cg fibers was achieved by suspension of HA particles in water.

The fibers were characterized using Thermogravimetric Analyzer (TGA) and Thermomechanical Analyzer (TMA) to find the weight retention, dimension changes and tensile modulus of the fibers. Surface morphology was observed from the EPI-DIC images and X-ray diffraction was performed to visualize the presence of HA coating on Cg fibers. Low load bearing and high melting point properties of HA were evident from the study.

Methods: 25 gms of dry type I Cg obtained from the bovine flexor tendon was mixed with 1liter of water along with 2ml of lactic acid to obtain 2.5% dispersion. Cg dispersion, extruded out of a hollow needle was passed through a coagulation bath consisting of acetone and ammonium hydroxide to form a continuous fiber. The fibers were then air-dried.

Cg/HA fibers were obtained by mixing HA powder with 2.5% Cg dispersion in weight ratio of 5/95 to form a mixture. This dispersion was used to spin the fibers. HA coated Cg fibers were obtained by immersing pure Cg fibers for a period of 24 hrs in a HA suspension obtained by suspending 2 mg of HA powder in 30 ml of water.

The fibers were characterized for their tensile modulus at isothermal temperature with a constant force rate of 0.02 N/min and temperature dependent dimension changes at a constant force of 1N by using TMA. Temperature dependent weight retention was measured by TGA. X-ray diffraction was performed on HA coated Cg fibers with the pure HA spectrum as a reference. Surface morphology of the fibers was observed by using optical microscope.

Results / Discussion: With TMA, the highest breaking point temperature was obtained for the Cg/HA fibers and the least for pure collagen fibers. However, tensile modulus had the opposite trend. TGA showed least weight retention with pure Cg.

Table: 1 Characterization of fibers using TGA and TMA

Type of fiber	Breaking pt temp	Tensile modulus	Weight retention
Cg	252.78^0C	$1141.8e10^6$ N/m^2	46.21%
Cg/HA	267.4^0C	$198.2e10^6$ N/m^2	80.17%
HA coated Cg	257.09^0C	$490.5e10^6$ N/m^2	60.94%

Difference in the surface morphology of the fibers was clearly noted in the EPI-DIC images. Pure Cg fiber had some striations. Dense lumps of HA were seen in Cg/HA composite fiber. The coating of pure HA crystals of size 50-90 microns was seen on the HA coated Cg fiber and non-homogeneity of the mixture was observed in the Cg/HA composite fiber.

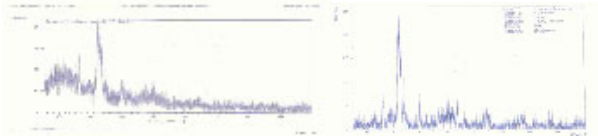

Fig:1 X-Ray Diffraction of (1)HA coated Cg fiber and (2)Pure HA powder.

The spectrum obtained with pure HA matched with the spectrum obtained from the HA coated collagen fibers.

Conclusions: Thermal analysis proved that HA had a poor mechanical loading property. However, since HA has high melting temperature, the fibers incorporated with HA could withstand higher temperatures at a constant load of 1N before breaking and had high weight retention at the temperature of 350^0C.

These results indicate that the amount of HA deposited on the coated fibers was less than the HA present in the composite fibers. This study showed a new technique for coating HA crystals on Cg fibers. Wet spinning for the composite fibers was employed for the first time. A good experiment will be to test the osteoconduction and osteoinduction properties on these fibers. Also, it is necessary to increase the strength of the fibers by crosslinking them with different agents without decreasing the percentage of HA in the fibers.

HA Coated Cg fibers with biomimetic methods such as simulated body fluid and the coated fibers obtained from the present study need to be compared. Methods for the uniform dispersion of HA in Cg fibers are to be investigated.

References: 1.A.S Posner, R.A Beebe Semin Arthritis Rheum. 1975; 4:267-291.
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