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TITLE: Effects of hydroxyapatite surface pretreated with tetraethyl orthosilicate

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ABSTRACT BODY:

Objectives: Biomaterial surfaces in the nano to submicron scale surface have been proved to facilitate bone cell growth. The silicon doped calcium phosphates have also been shown to stimulate the pre-osteoblast proliferation. The tetraethyl orthosilicate (TEOS) was used as an additive in the sol-gel processing, to pretreat hydroxyapatite (HA) before sintering at different temperatures. Effects of pretreatment on surface morphology, composition, and phase structure were evaluated in this study.

Methods: Four HA disks (12mm diameter×2mm thick) made of hydroxyapatite powder pressed under 2000 kgf were immersed in the TEOS solution (pH adjusted to 11 using NH₄OH to start the colloidal reaction) for 10 min under vacuum. After drying at 60°C for 120 min, the disks were sintered at 900°C, 1050°C, and 1200°C respectively. Four pure HA disks without TEOS pretreatment were set as control group.

Results: Under SEM, a silicon-rich layer (5.2 to 26.6 wt%) was observed on the HA surface with loose and discrete morphology after being sintered at 900°C and 1050°C. This layer turned melted-like in the nano to submicron scale grains after being sintered at 1200°C. X-ray diffraction patterns demonstrated that the phases of TEOS-immersed HA consisted of hydroxyapatite and β-TCP (β-tricalcium phosphate) structure essentially. The HA/β-TCP ratio was decreased with increasing sintering temperatures. The FTIR spectra showed that when the sintering temperature was higher than 1050°C, typical absorption bands of β-TCP at 1116, 972, 942 cm⁻¹ were found with the disappearance of hydroxyl group at 3572 cm⁻¹, confirming that phase transformation occurred in TEOS-immersed HA.

Conclusions: The TEOS-immersed HA changed its phase structure from pure HA to β-TCP with nano to submicron scale surface texture at high sintering temperature.

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