Structure and Morphology of Calcium Phosphates Obtained From A Flowable Dental Composite Stored in Simulated Body Fluid and Artificial Saliva

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Scope: Bioactive materials have become increasingly relevant in dentistry and one mechanism often associated with bioactivity is apatite formation. The apatite-forming ability of flowable dental composite (FDC) was tested here with two storage solutions - simulated body fluid (SBF per ISO 23317:2014) and artificial saliva (SAGF). Characterization was performed using SEM (Scanning Electron Microscopy), EDS (Electron Diffraction Spectroscopy), XRD (X-Ray Diffraction), and Fourier-Transform Infrared Spectroscopy (FTIR) investigation. The study was done to better understand the impact of the different storage solutions on the determination of structures obtained on the surface of the flowable composite.

Methods: The dental composite evaluated contains nanocrystalline phosphates including: nanohydroxyapatite (nHA), nanofluorapatite (nFA), nano β-tricalcium phosphate (nβ-TCP). Composites were stored for at least 28 days in SBF, SAGF, and deionized water at 37 ºC. The SEM/EDS (JEOL JSM-6010PLUS/LA), FTIR (Nicolet iS50) and XRD Analysis (Bruker D8 Advance) were used for determination of morphology and composition of structures obtained.

Results: A variety of structures were observed from SEM and EDS imaging. The presence of other ions including Mg^{2+}, Na^{+}, Si^{4+}, Cl^{-} were detected as well (Fig1). Finally, the most effective factor was type of storage solution. FTIR analysis showed peaks related to PO_4^{3-} group vibrations characteristics for HA. However, in some cases peaks related to HPO_4^{2-}, PO_2^{-} as well as CO_3^{2-} and SiO_4^{4-} groups vibrations are observed. From XRD, some patterns confirm the presence of hydroxyapatite, including characteristics lines of other calcium phosphates (Fig.2).

Conclusions: Presented results confirm apatite forming ability (in vitro) of the dental composite. However, based on the results, it is critical to consider if differences in SAGF or SBF ultimately led to differences in the calcium phosphate structures and morphologies observed. From both SEM and XRD analysis, additionally OCP (octacalcium phosphate) and potentially other calcium phosphate structures were observed. EDS analysis indicates the presence of Mg^{2+}, Na^{+}, Si^{4+}, Cl^{-} ions in structure, which may substitute Ca^{2+} and P^{5+} ions in structure.

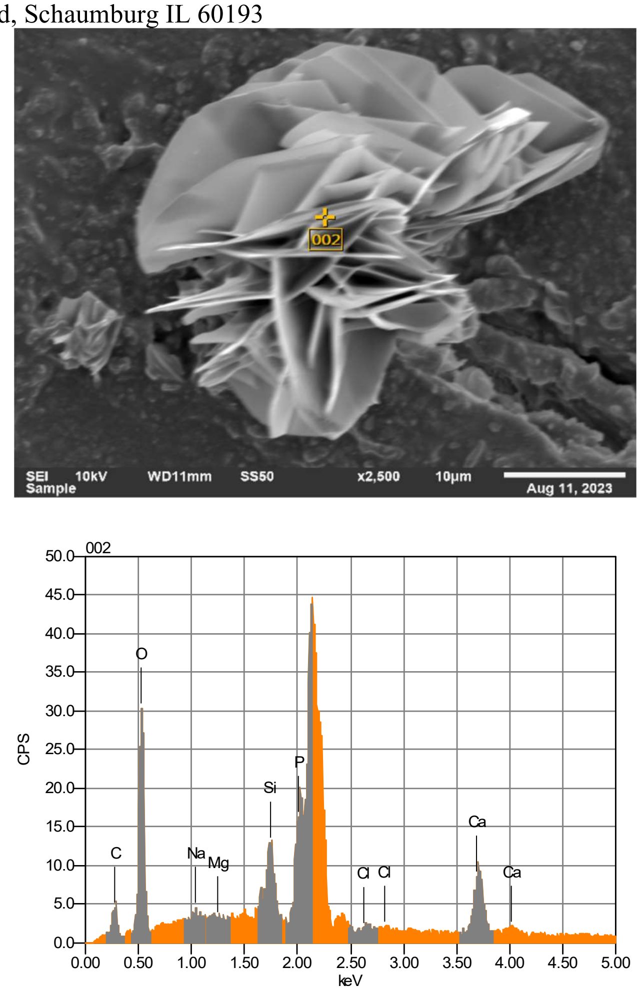

Fig. 1. SEM image and EDS spectra of 5 mass% containing nHA flowable dental composite sample surface stored 56 days in SAGF at 37 ºC.

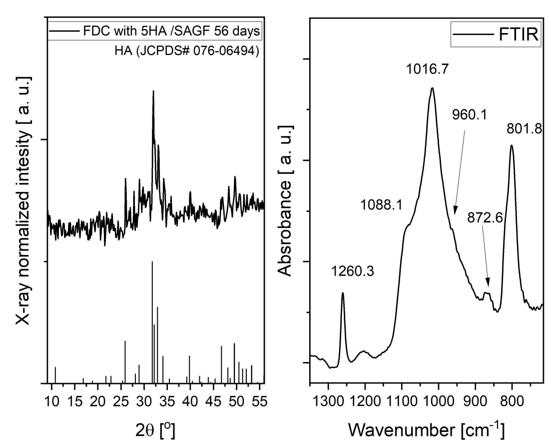

Fig. 2. XRD (left) and FTIR (right) of 5 mass% containing nHA FDC storage 56 days in SAGF at 37 ºC.