EVALUATION OF THE PHYSICO-CHEMICAL PROPERTIES OF HYDRAULIC CALCIUM SILICATE-BASED SEALER MODIFIED WITH CALCIUM HYDROXYAPATITE, FLUOROHYDROXYAPATITE, AND MAGNESIUM WHITLOCKITE

Paulina Mozūraitė[1], Goda Bilvinaitė[1,2], Saulius Drukteinis[1], Simas Šakirzanovas[2]

[1]Vilnius University, Faculty of Medicine, Institute of Dentistry, Lithuania
[2]Vilnius University, Faculty of Chemistry and Geosciences, Institute of Chemistry, Lithuania
paulina.mozuraite@mf.stud.vu.lt

Hydraulic calcium silicate–based sealers (HCSS) are widely used materials in modern endodontics due to their advantageous handling properties and clinical performance, mainly attributed to favorable biocompatibility and bioactivity. However, their relatively long setting time and insufficient chemical stability often lead to increased solubility and altered setting reactions, representing key limitations of HCSS. Further, the hydration products of HCSS differ from mineral phases naturally present in dental tissues, limiting their biomimetic properties. Although several attempts have been made to modify HCSS with conventional calcium hydroxyapatite (Ca-HAp, $Ca_{10}(PO_4)_6(OH)_2$), showing improved physico-chemical properties of HCSS, the influence of chemically distinct calcium phosphate phases – such as fluorohydroxyapatite (F-HAp, $Ca_{10}(PO_4)_6(OH)_{2-x}F_x$) and magnesium whitlockite (Mg-WH, $Ca_{18}Mg_2(HPO_4)_2(PO_4)_{12}$) – on HCSS modification remains unexplored. Nevertheless, both Mg-WH and F-HAp possess intrinsic physicochemical and biological properties that make them promising HCSS modifiers. Therefore, the present study systematically investigated and compared the physico-chemical properties of HCSS modified with Ca-HAp, F-HAp, and Mg-WH.

Ca-HAp, F-HAp, and Mg-WH were synthesized using chemical precipitation, chemical precipitation combined with pH cycling, and a low-temperature dissolution–precipitation method, respectively. Phase crystallinity and purity were analyzed by X-ray diffraction (XRD), while functional groups were identified using Fourier-transform infrared spectroscopy (FT-IR). Particle morphology was examined using scanning electron microscopy (SEM). The commercial and clinically used HCSS BioRoot RCS (Septodont, France) was then modified with Ca-HAp, F-HAp or Mg-WH. The chemical properties and ISO 6876:2012 standard parameters of unmodified and modified HCSS were evaluated. Setting time and flowability were determined following ISO 6876:2012 guidelines. Surface morphology and elemental composition were analyzed using scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM–EDX). Solubility was assessed by mass loss measurements, while ion release was quantified using inductively coupled plasma optical emission spectrometry (ICP-OES). Mechanical hardness was evaluated by compression testing, thermal stability by thermogravimetric analysis (TGA), and porosity by micro–computed tomography (micro-CT). Surface morphology, solubility, pH, ion release, thermal stability, and porosity were evaluated after 24 h and 7 d of incubation in Hank's balanced salt solution at 37 °C.

The applied experimental approach enabled a comprehensive evaluation of original and modified HCSS, allowing a comparative assessment of the effects of Ca-HAp, F-HAp, and Mg-WH on clinically relevant physico-chemical properties.

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