

# FTIR and XRD Characterization of Heat Effect on Nanostructured Synthetic Hydroxyapatite and Dental Enamel

Rabelo, J. S.<sup>1</sup>; Ana, P.A.<sup>2</sup>, Mazzocchi, V. L.<sup>1</sup>; Valério, M. E. G.<sup>3</sup>; Zzell, D.M.<sup>1\*</sup>  
 1-IPEN – CNEN/SP, Sao Paulo-Brazil. 2- UFABC, Sao Bernandodo Campo, Brazil. 3- UFS, Sergipe-Brazil  
 zzell@usp.br

**Abstract:** Nanometer sized HAP and dental enamel powders were evaluated regarding changes in their degree of crystallinity and composition after oven or Er,Cr:YSGG laser heating. Decrease of the Ca/P ratio to levels close to the theoretical ones for both synthetic nano-HAP and for the enamel was measured. Laser irradiation caused considerable decrease in the amount of carbonate in the materials heated to high temperatures, but without changes in crystallographic phase.

## 1. Introduction

The study evaluate the physical changes and/or chemical that occurs in synthetic nano-structured hydroxyapatite (HAP) and in enamel under action of thermal heating in oven or laser irradiation of Er,Cr:YSGG that may cause changes in its structure to make them more resistant to demineralization aiming the formation of dental caries.

The synthetic nano-structured HAP was produced and enamel powder was collected from the bovine teeth. Samples of powder enamel and synthetic HAP were subjected to thermal heating in oven at temperatures of 200 °C, 400 °C, 600 °C, 800 °C and 1000 °C. For the laser irradiation of materials, were made with 5,79 J/cm<sup>2</sup> of irradiation, 7,65 J/cm<sup>2</sup>, 10,55 J/cm<sup>2</sup> and 13,84 J/cm<sup>2</sup> for synthetic HAP and 7,53 J/cm<sup>2</sup>, 10,95 J/cm<sup>2</sup>, and 13,74 J/cm<sup>2</sup> for the enamel. The samples were evaluated by X-ray diffraction (XRD) for analysis of crystallographic phases and analysis by the Rietveld method, to determine their respective proportions in the material, as well as results of changes of the lattice unit cell parameters (axis-a, axis-c and volume), crystallites sizes and the occupation rate of sites of Ca and P atoms. The samples were analyzed by Fourier transform infrared spectroscopy (FTIR) regarding changes in the material composition.

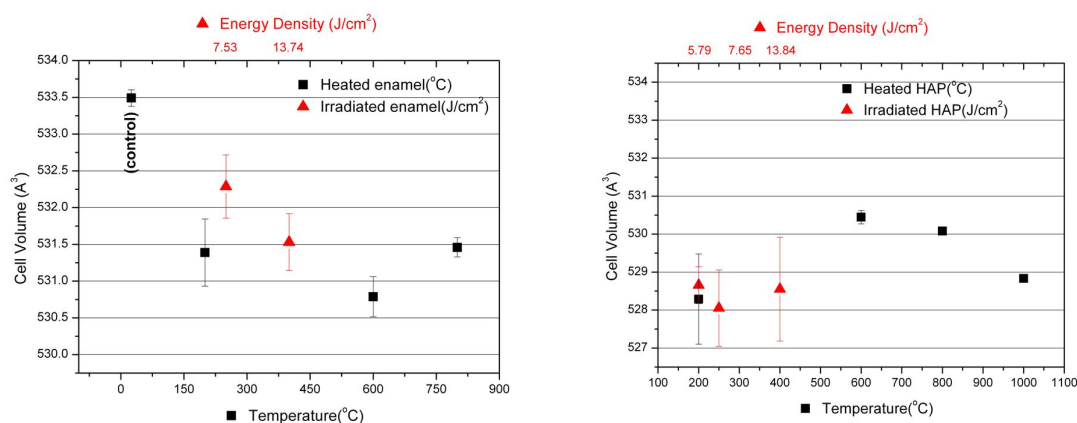


Fig. 1. Variation in cell volume of cell unit in enamel(left) and nano-HAP (right) heated by oven or Er,Cr:YSGG laser irradiated.

Besides the major hydroxyapatite crystallographic phases, there was formations of octacalcium phosphate (OCP) and phase  $\beta$  of tricalcium phosphate ( $\beta$ -TCP) in enamel heated at 800 °C. There was reduction of the axis-a, volume and size of crystallites to the temperatures between 400 °C and 600 °C and also on laser irradiated samples. Above the temperature of 600 °C it is observed the effect in the lattice parameters. The Ca/P relation in all the samples decreased. The enamel samples irradiated by 7,53 J/cm<sup>2</sup> showed Ca/P equal to 1,6817 and by 13,74 J/cm<sup>2</sup> Ca/P was 1,6831. The effects of heating in oven and laser irradiation causes reduction of carbonate content, and this effect was more evident in laser irradiated samples, there was also decreases in water and adsorbed hydroxyl contents. All these changes alter the properties of the material, as its solubility and therefore affect the demineralization process and can be useful for caries prevention as well as dental erosion prevention.

Acknowledgements: CNPq (INCT 465763/2014-6 and PQ 312397/2013-5) and CAPES (PROCAD 88881.068505/2014-01).