Supercritical CO_2 assisted production of porous TiO_2-HA composites

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The purpose of the present study is to obtain a new porous support for osteogenesis (bone formation) from titanium dioxide (T) and hydroxyapatite (HA) by a sol-gel process and supercritical-CO_2 drying. The main objective is to develop porous scaffolds of TiO_2 with different mass proportion of HA nanoparticles and to assess the effect of the studied compositions on the adhesion properties of cells MG-63.

Nanometric HA particles were synthesized by a hydrothermal method and subsequently applied during the sol-gel process to obtain TiO_2-HA composite gels from titanium butoxide ($Ti(OBu)_4$, acetic acid, isopropanol, distilled water and polyvinylpyrrolidone (PVP) as porogen agent. In the next step, the obtained TiO_2-HA gels of cylindrical morphology were dried using supercritical CO_2 at conditions varying between 60 and 90° C at pressure from 240 to 400 bar. Finally, the dried gels were calcined at 800°C in air by 1 h. Characterization was carried out by X-ray diffraction (XRD), Raman spectroscopy, Density/porosity measurement, Thermal analysis (TGA-TDA), Scanning electron microscopy (SEM) and *in vitro* cells MG-63 adhesion tests on dried and calcined samples.

As a result, TiO_2-HA porous composites were successfully obtained by integration of sol-gel and supercritical drying processes. XRD analysis showed that between 18,6 and 29,4 % m/m of HA can be included into a TiO_2 matrix, allowing to stabilize the anatase phase over the rutile phase. The anatase stability is increased by the increase of HA content through chemical interaction between phosphate groups in HA with hydroxyl groups in TiO_2 gels, by means of which Ti-O-P bonds are formed, proved by the peak at 796 cm^{-1} in the Raman spectra. Optimum conditions for drying the aerogels were found as 60 ° C and 250 bar, TiO_2-HA composites with open porosity of 70-80% were obtained with a uniform distribution of pores between 5-20 µm in size.