## Abstract

Chitosan (Cs) and hydroxyapatite (Ha) were analysed by X-ray photoelectron spectroscopy (XPS). Phosphorylated Cs microparticles and hybrid Cs/Ha microparticles were prepared and analysed by XPS before and after immersion in a solution 1.5 times more concentrated than a simulated body fluid (SBF). The accuracy of spectrum recording, peak decomposition and peak component assignment was insured by a post-control of charge stabilization, and by the examination of correlations between spectral data guided by stoichiometry and charge balance. The concentration of organic oxygen was determined from the concentrations of the oxidized forms of carbon, allowing a sharper insight into speciation and O 1s peak shape. This indicated that the hydroxide ion of Ha, and hydrogenophosphate if present, give a contribution near 532.4 eV, which overlaps with organic oxygen. As a result of immersion in the 1.5*SBF solution, the formation of $CaCO_3$ and of Ha material occurred. A quantification could be made for the constituents of biomaterial interest, contaminating salts and paraffin oil residues from the microparticle manufacturing process. The uncertainties regarding the nature of the model calcium phosphate used and the best marker for calcium carbonate were addressed by comparing the possible effect on the output, which was facilitated by using ternary composition diagrams. Whatever their formulation, the native microparticles were found to be coated by a thick layer of paraffin oil. The induction of calcium carbonate and phosphate precipitation or the retention of precipitates by the microparticles was favored by the presence of phosphate in the initial formulation either by phosphorylation or by incorporation of Ha. Copyright