

# Nickel polyphosphate coacervates glass-ceramics: structural and Rietveld investigations

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Glass-ceramics based in nickel coacervates polyphosphates (NiPC) were obtained using the coacervation route as precursor. The coacervation process occurs in some cases through the addition of low molecular weight solvents to reduce the dielectric constant of the solution, leading it to colloidal vesicles formation in aqueous solution called “coacervates” [1]. In this work, we have initially studied the influence on the addition of different solvents, such as ethanol, dimethylketone, methanol, and monoethylene glycol in the preparation of NiPC. Subsequently, the Nickel polyphosphate glass-ceramics (NiP-GC) were prepared for each NiPC sample by the melt-quenching technique. The colloidal systems precursors were homogenized and melted in platinum crucibles at 1000 °C for 20 minutes. The thermal analysis, concerning the NiP-GC, showed the glass transition temperature ( $T_g$ ) for all samples and the glass-ceramic state of these samples was confirmed by diffraction peaks over the diffuse halo (amorphous phase). The identification and quantification (in % weight), of three polymorphs in all NiP-GC samples were performed through the Rietveld method. The nickel coacervates polyphosphates can crystallize as three polymorphs phases:  $Ni_2P_4O_{12}$ ,  $\beta-Ni_2P_2O_7$ , and  $\delta-Ni_2P_2O_7$ . All of these polymorphs could be easily differentiated by x-ray diffraction. Raman spectra of NiPC and NiP-GC samples indicated that the solvents addition to NiPC leads to hydrolysis. Therefore, the long polyphosphate chains breakdown generate vibrational modes to their pyrophosphates ( $Q^1$ ) and orthophosphates ( $Q^0$ ) species. The NiP-GC reflectance spectra showed that  $Ni^{2+}$  ions coexist in both octahedral and tetrahedral sites and the structural investigation results, by x-ray diffraction, showed the presence of two coordination sites.