Ex-situ versus in-situ synthesis of NZFO/f-MWCNTs nanocomposites

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Nowadays, spinel ferrites magnetic nanoparticles (SF-MNPs) incorporated into 1D or/and 2D carbon-based matrix are intensively studied due to their outstanding promising application properties e.g. drug delivery systems for magnetic hyperthermia or microwave-absorbing materials [1-3]. A lot of attention has been recently paid to Ni-Zn-based ferrites with non–collinear spin structure [4] and microstructure which can be controlled by applying various synthesis methods. Our research is focused on the synthesis and complex characterization of $Ni_{0.5}Zn_{0.5}Fe_2O_4$ nanoparticles with 5 wt.% of (NZFO) incorporated into functionalized carbon nanotubes (f-MWCNTs). The synthesis of NZFO/f-MWCNTs nanocomposites was carried out by so-called *in-situ* and *ex-situ* approaches followed by the calcination. The XRD patterns and TEM images confirm the successful formation of nanocomposites. The crystallite size of NZFO particles significantly depends on the synthesis route, as evidenced by multi-technique characterization. The comparison of photoemission XPS spectra of studied nanohybrids reveals the domination of f-MWCNTs contribution as expected. The iron redistribution/separation confirmed by variation within deconvoluted Fe2p spectra is dependent on synthesis routes and the calcination process. The XAS and ResPES study confirms the evident difference in *in-situ* and *ex-situ italic text*synthesis. The magnetic properties are slightly affected by Fe-based catalyst residuals as evidenced for f-MWCNTs, but the magnetic state originating from NZFO is dependent on nanoparticles size.

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