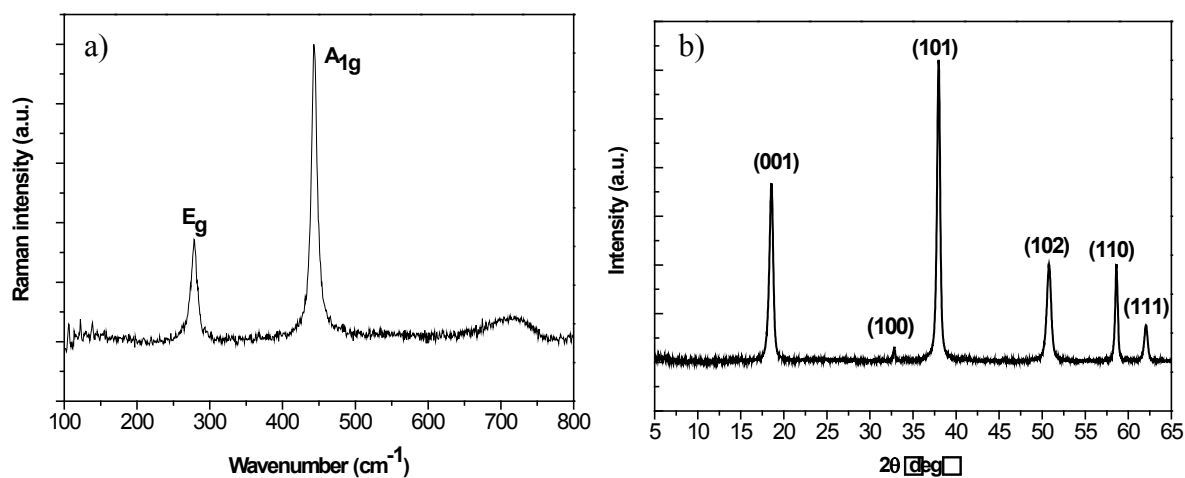# Supporting information



**Figure S1 : Raman spectra (a) and XRD patterns (b) of the solid phase obtained at the end of the Mg$^{2+}$ solution titration**
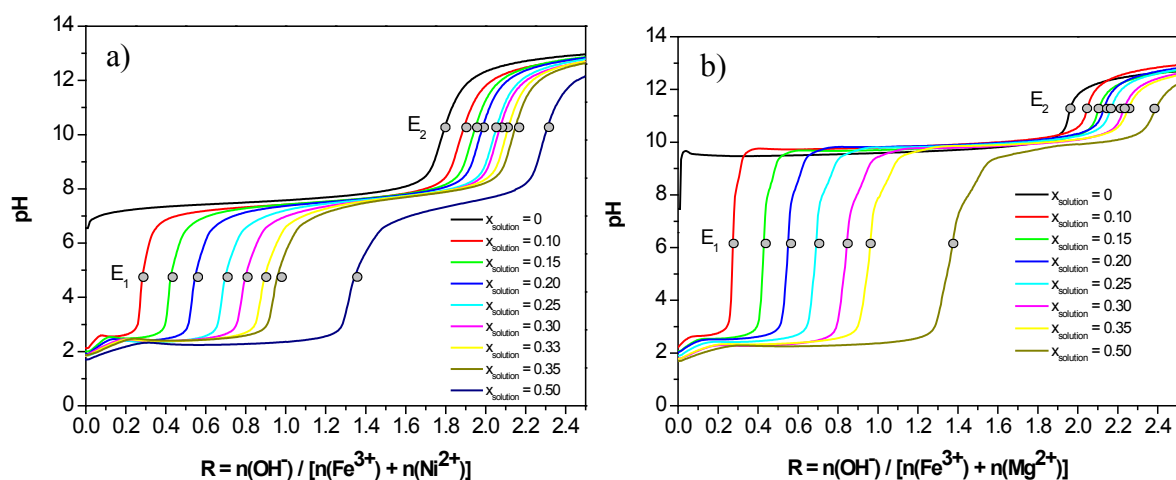


**Figure S2 : Titration curves for different $x_{solution}$ for (a) Ni$^{2+}$-Fe$^{3+}$ mixed cation solutions and (b) Mg$^{2+}$-Fe$^{3+}$ mixed cation solutions**
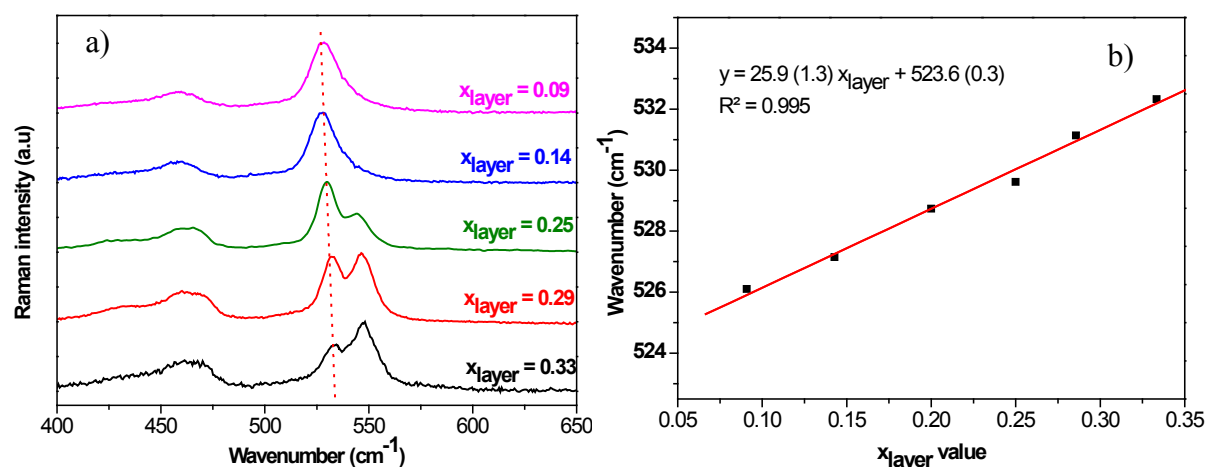


**Figure S3 : (a) Raman spectra of Ni$^{II}$-Fe$^{III}$ LDH with different $x_{layer}$ values and (b) correlation between the wavenumber of the LDH Raman band and the $x_{layer}$ values. The numbers in brackets refer to the standard deviation for each parameter of the linear regression.**

**Figure S4 : (a) Raman spectra of Mg$^{II}$-Fe$^{III}$ LDH with different $x_{layer}$ values and (b) correlation between the wavenumber of the LDH Raman band and the $x_{layer}$ values. The numbers in brackets refer to the standard deviation for each parameter of the linear regression.**
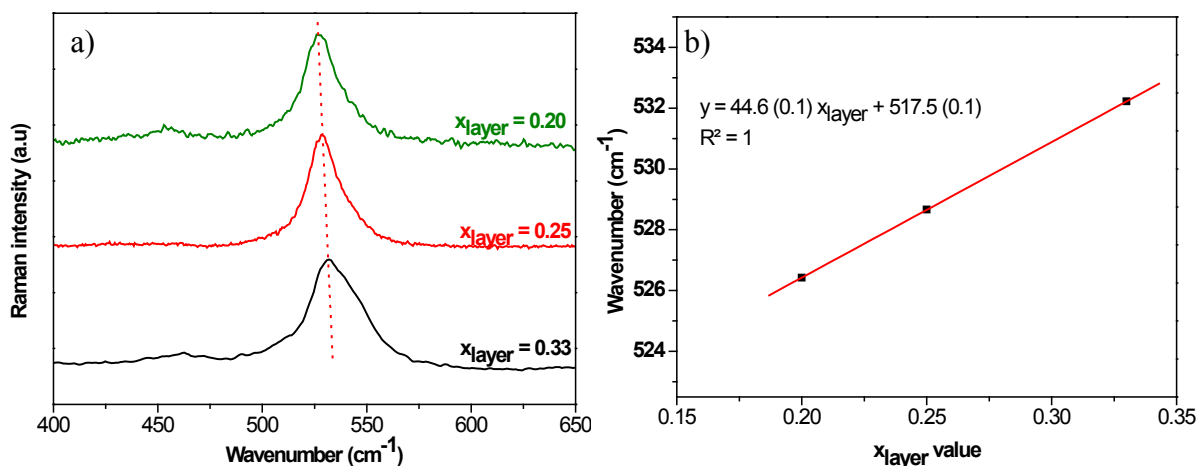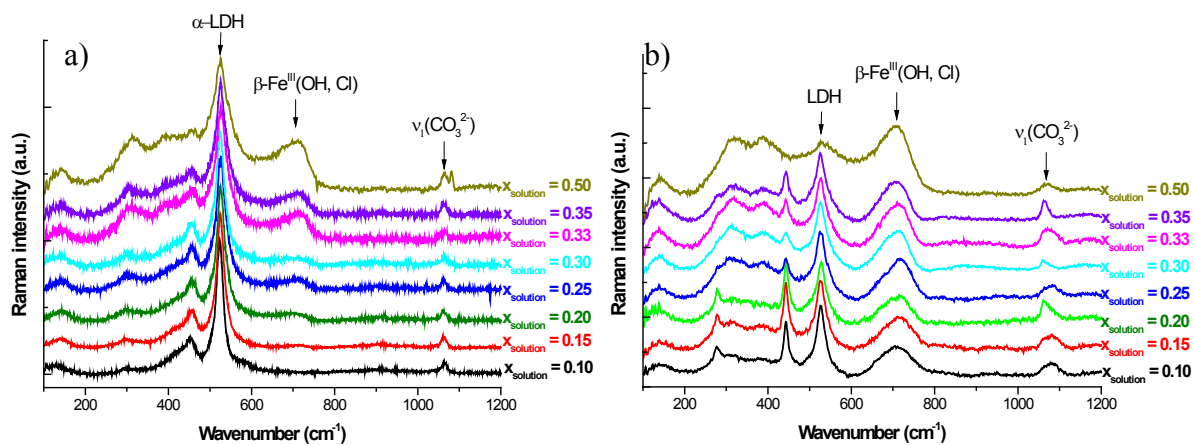


**Figure S5 : Raman spectra in the spectral range 100-1200 cm$^{-1}$ (a) same as Figure 8a and (b) same as Figure 12a**