

# Spectroscopic and microscopic investigations of the graphene oxide influence on hybrid powder products based on LDH structures

I Brezestean<sup>1,2</sup>, D Marconi<sup>1</sup>, A Colniță<sup>1</sup>, A. Ciorăță<sup>1</sup>, M. C. Corobea<sup>3</sup>, A. E. Stamate<sup>4</sup>, O. D. Pavel<sup>4</sup>, R Zăvoianu<sup>4</sup> and I Turcu<sup>1</sup>

<sup>1</sup>National Institute for R&D of Isotopic and Molecular Technologies, 67-103 Donat, 400293 Cluj-Napoca, Romania

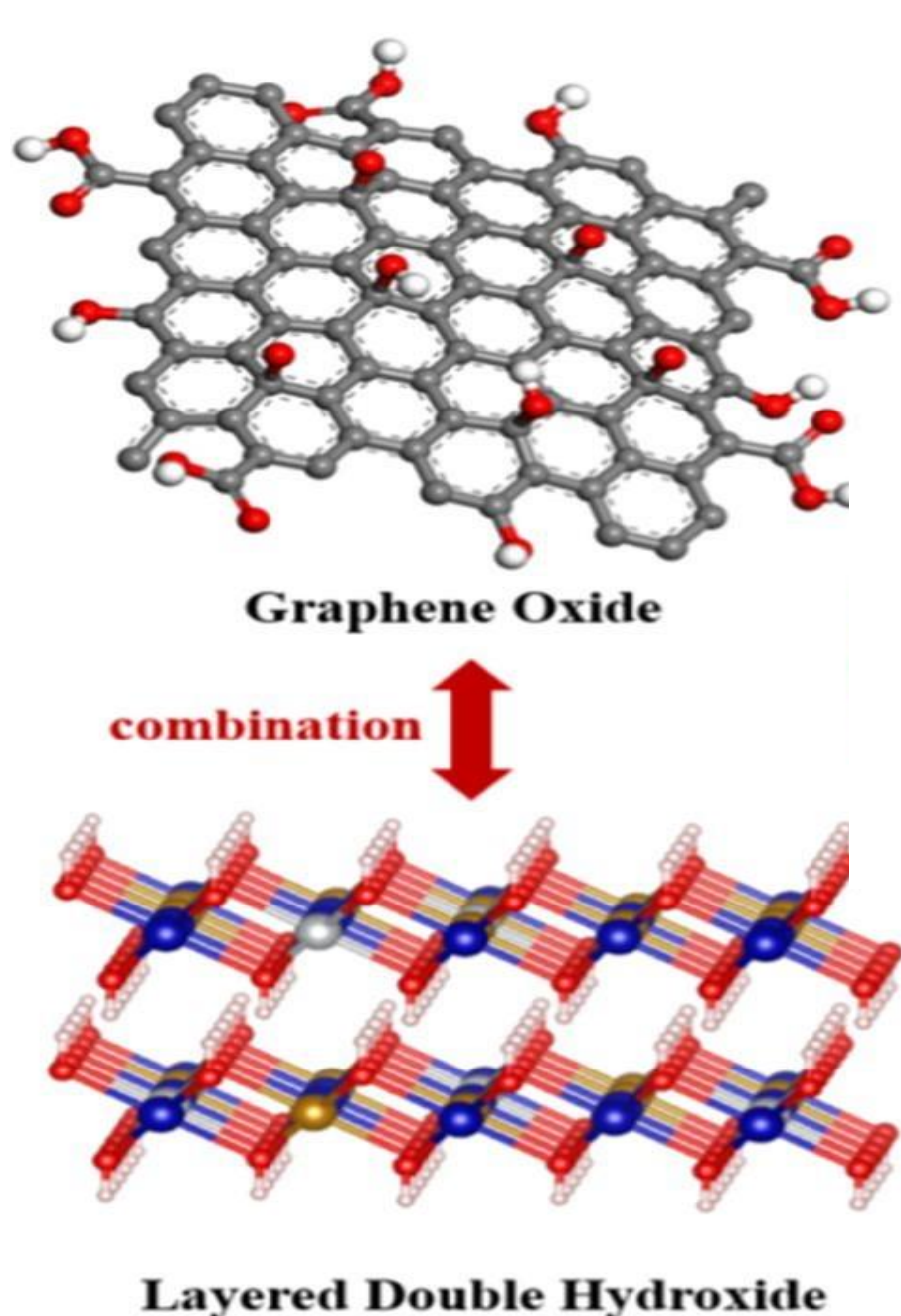
<sup>2</sup>Babes-Bolyai University, Faculty of Physics, Biomolecular Physics Department, Cluj-Napoca, Romania

<sup>3</sup>INCDP-ICECHIM, Polymer Dept., Splaiul Independentei No. 202, Bucharest, 060021, Romania

<sup>4</sup>University of Bucharest, Faculty of Chemistry, Bd. Regina Elisabeta No. 4-12, Bucharest, Romania

ioana.brezestean@itim-cj.ro

This work investigates a complete morpho-structural characterization of new composites based on  $Mg_3Al_{0.75}Ce_{0.25}$  (Ce-doped LDH) and graphene oxide (GO) in various concentrations using Raman spectroscopy, an important tool able to assess defects in hybrid powders and display the presence of GO in their composition. Scanning electron microscopy (SEM) was employed for the structural characterization of the new compounds and the identification of the GO crystallization process and their interaction with Cerium modified LDH composites. Energy-dispersive X-ray (EDX) spectra also validated the Raman results. The similar morpho-structural defects regardless of GO concentration in the doped hybrid powders indicates that the amount of GO used in our samples can be successfully integrated into novel composites with enhanced mechanical properties



## SAMPLE PREPARATION AND EXPERIMENTAL SET-UP



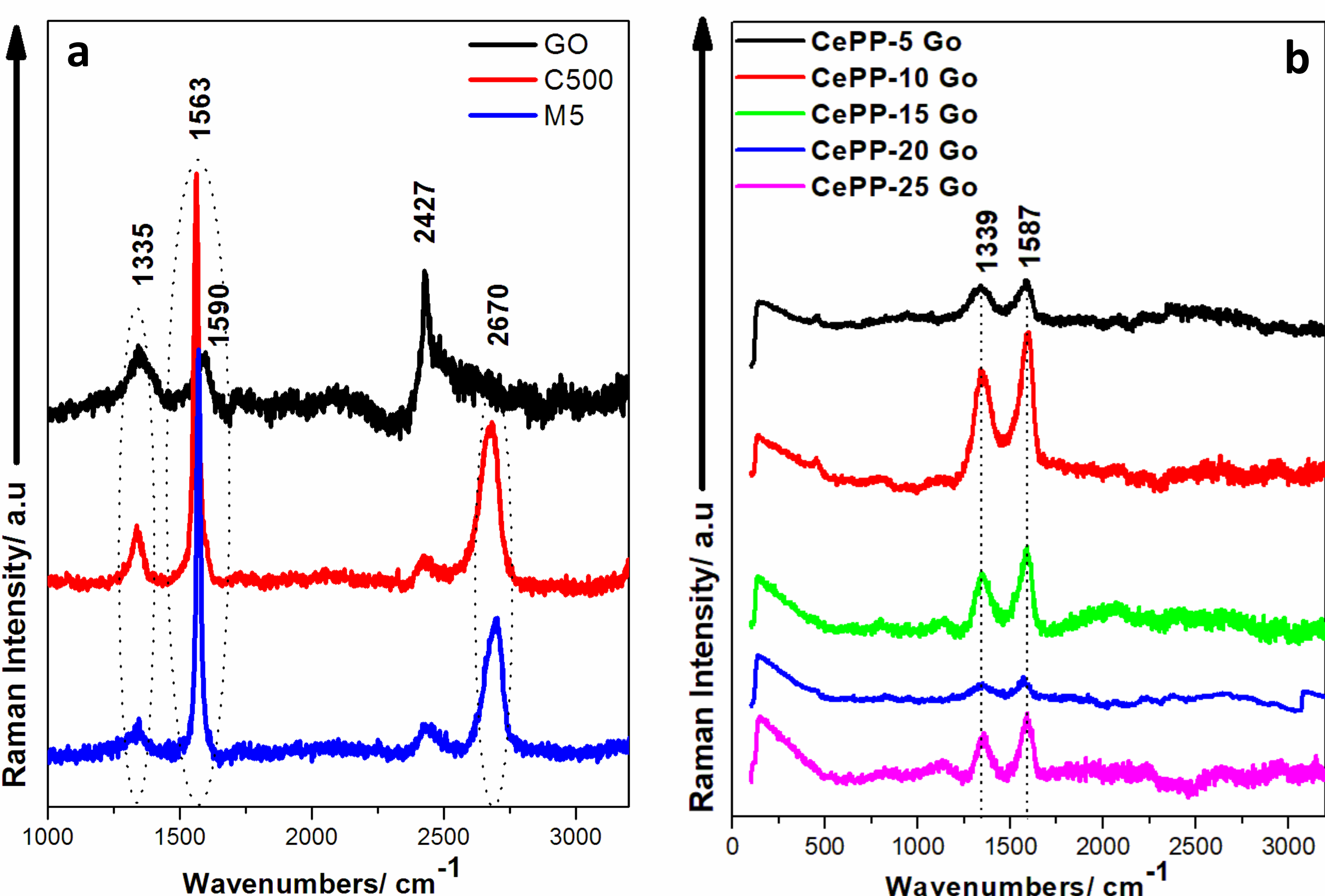
(a) Confocal Raman microscope Renishaw InVia Reflex, Diffraction gratings: 1200, 1800 lines/mm, (b) Lasers lines: Cobolt DPSS 532 nm (200 mW), Diode 785 nm (300 mW)



Scanning electron microscope (SEM) Hitachi SU 8230 Cold Field Emission, coupled with EDX analysis (Oxford Instruments, AZtec Software)

## RESULTS AND DISCUSSIONS

### I.1 Raman Characterization

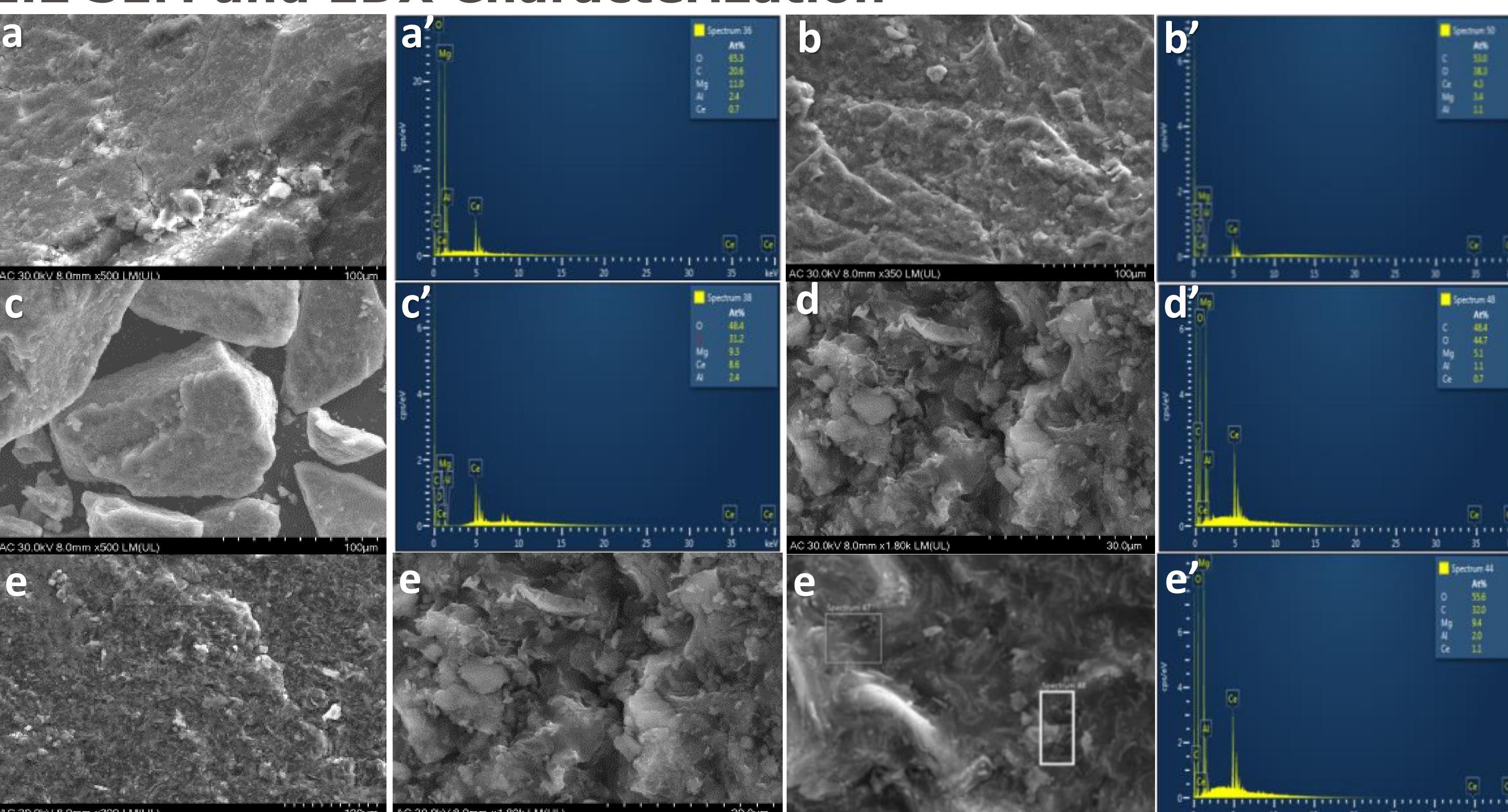


Samples	$I_D/I_G$
CePP-5Go	0.878
CePP-10Go	0.88
CePP-15Go	0.89
CePP-20Go	0.878
CePP-25Go	0.88
GO	1.06

Summaries of the ID / IG ratio of CePP hybrid powder samples with different GO concentrations

In the Raman spectra of the 5 hybrid powder samples CePP-5Go, CePP-10Go, CePP-15Go, CePP-20Go, CePP-25Go the signal from the GO component screens all the bands characteristic of pure LDH and Ce components in the region 100-1100  $cm^{-1}$ , even if it was in a very high concentration compared to that of the compound GO. The characteristic bands of Go graphene oxide at 1336  $cm^{-1}$  and 1572  $cm^{-1}$  can be observed.

### I.2 SEM and EDX Characterization



SEM characterizations obtained for CePP compounds for different GO concentrations: (a), (a') CePP - 5GO, (b), (b') CePP-10 GO, (c), (c') CePP-15 GO, (d), (d') CePP-20GO and (e), (e') CePP 25GO, with selected EDX spectrum for each concentration

## Conclusions

- For CePP-GO samples (0.5-0.1), the defect ratio remains constant in almost all cases, the defects following the calculation of the ratio varying very little by 0.01 for some samples but remaining around 0.88-0.89. From the spectra presented above, it appears that GO can be used for the synthesis of hybrid powders and the amount used in the synthesis **can be successfully integrated** in the compound in its use in the ranges presented concentration.
- Based on the above presented results, it can be concluded that the co-precipitation of  $Mg_3Al_{0.75}Ce_{0.5-0.1}$  LDH in the presence of GO suspension leads to HT3Ce-xGO composites with increased crystallinity of the LDH.
- The presence of GO nanosheets with an increased number of defect sites on the surface, as it was indicated by the ID/IG ratio of 1.06 determined by Raman spectroscopy, can both promote **the in-situ formation and immobilization of LDH on GO**, and **inhibit the agglomeration of LDH nanosheets** during the growth process while orientating the crystallization of the LDH phase.