Investigation of mechanism of glycine-nitrate reaction using electron microscopy

Dedkova, K.[1], Kuznikova, L.[2,1], Cvejn, D.[1,3] and Kukutschova, J.[1,4]

[1] Regional Materials Science and Technology Centre, VŠB - Technical University of Ostrava, 17. listopadu 15, 708 33 Ostrava, Czech Republic, [2] Nanotechnology Centre, VŠB – Technical University of Ostrava, 17. listopadu 15, 708 33 Ostrava-Poruba, Czech Republic, [3] ENET Centre, VŠB-Technical University of Ostrava, 17. Listopadu 15/2172, 708 33 Ostrava-Poruba, Czech Republic, [4] Center of Advanced and Innovation Technologies, VŠB-Technical University of Ostrava, 17. Listopadu 15/2172, 708 33 Ostrava-Poruba, Czech Republic

Although the glycine-nitrate reaction, as a source of oxide nanoparticles, has been known for more than 30 years, the mechanisms of the particular chemical transformations remain still unclear. According to our previous observations [1], thermal decomposition (usually occurring at 600°C) of transient complex formed *in situ* from $Lx(NO_3)_3.H_2O$ (where Lx stands for various lanthanides) and glycine is not straightforward combusted and contains several intermediates and *meso*-stages. The direct observation of the transient matters by scanning electron microscope (SEM) equipped with energy dispersive X-ray spectroscopy (EDS) appears to be a useful tool, at least for screening the transition stages worth to be further analyzed. We have observed the glycine-$Gd(NO_3)_3$ aqueous mixture treated at temperatures of 195 °C, 220° C, 240 °C, and 310 °C, respectively, to record possible intermediate steps. Throughout the four temperatures of the preparation of samples, there is a clear trend of a slow transformation of amorphous gel-like structure with sporadic surface ruptures at lower temperature treatments to the more organized net-like structure apparently sourcing from the initial ruptures at higher temperature treatments. Moreover, EDS analysis indicates a significant progressive loss of nitrogen in the samples clearly connected with the rising treatment temperature. These results suggest there is a chemical reaction taking place. This observation is a part of our broader undergoing investigations aimed to reveal and explain the mechanism(s) of formation lanthanide-oxide nanoparticles by glycine-nitrate processes.

[1] Dedkova, K., Kuznikova, L., Pavelek, L., Matejova, K., Kupkova, J., Cech Barabaszova, K., Vana, R., Burda, J., Vlcek, J., Cvejn, D., Kukutschova, J., *Materials Chemistry and Physics* 197 (2017) 226-235.

Acknowledgement

This study was supported by projects LTI17023 (INTER EXCELENCE, INTER INFORM), LO1203 "Regional Materials Science and Technology Centre - Feasibility Program" and SP2018/81(Ministry of Education, Youth and Sports of the Czech Republic).