**ABSTRACT**

Reaction of hydrazine and mercaptosuccinic acid with metal ions forms complexes with general formula [Ln(N2H4)2CH2(COO)CH(SH)(COO)1.5]·(H2O), where Ln = La(III), Pr(III), Nd(III), Sm(III), and Gd(III) at pH 5. The complexes have been characterized by elemental analysis, IR and UV-visible spectroscopic, thermal and X-ray diffraction studies. The IR data reveal that the acid moiety in the complexes is present as dianion due to the deprotonation of COOH groups by lanthanides in these complexes, leaving –SH group unionized and hydrazine as bidental neutral ligand showing absorptions in the range of 945–948 cm−1. The thermoanalytical data evince that the complexes are stable up to 103°C and undergo complete decomposition in the range of 550–594°C resulting in metal oxides. SEM images of La2O3 and Gd2O3 residues show their nano sized clusters suggesting that the complexes may be used as precursors for nano La2O3 and Gd2O3, respectively. X-ray powder diffraction patterns show isomorphism among the complexes. The kinetic parameters of the decomposition of the complexes have been computed by Coats-Redfern equation