

Sol-gel auto combustion synthesis of nanostructured cobalt aluminate

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There are many methods of preparing CoAl_2O_4 nanosized powders such as co-precipitation method, hydrothermal method, solid state reactions method and sol-gel method which has some advantage.

In this report cobalt aluminate powder was prepared by sol-gel citrate route. Metal nitrates and citric acid as complexing were dissolved in distilled water with following addition ammonia solution to get $\text{pH} = 7$. After being evaporated obtained sol solution had been transformed to polymeric gel and auto combustion process occurred. Further calcined didn't carry out. In the result dark gray product was obtained. Synthesized sample was characterized by XRD, FT-IR, TG-DSC and colorimetric methods.

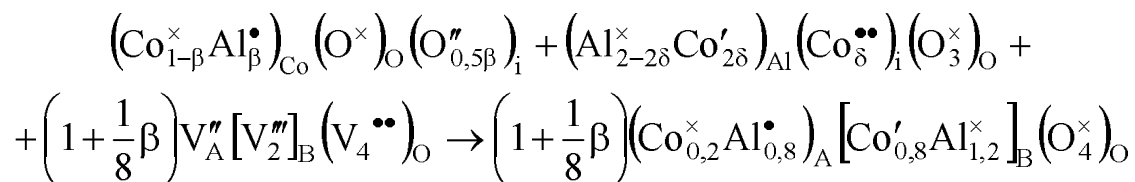
The main peaks of XRD pattern are indexed in accordance with spinel structure of CoAl_2O_4 and $\text{Fd}3\text{m}$ space group. The average crystallite size of the cobalt aluminate was estimated by Scherrer's formula. Crystallographic data (X-ray density (ρ_{XRD}), specific surface area (S), radius of the ions at octahedral (r_{oct}) and tetrahedral (r_{tet}) sites values were calculated using XRD data. Partially inversion of structure also was detected by this analysis.

The IR spectrum was recorded in the $4000\text{--}400\text{ cm}^{-1}$ region with an FTIR Brucker Alpha-P spectrometer. Several peaks in $700\text{--}450\text{ cm}^{-1}$ range on IR spectrum indicate the formation of CoAl_2O_4 spinel. These peaks belonging to vibrations of Me-O groups in tetrahedral and octahedral sublattices.

DSC curve of dried precursor showed two exothermic peaks, first of which corresponding to decomposition of metal complexes and metal oxides formation and the second on spinel phase formation. TG curve of precursor showed weight loss is 87 %.

For colorimetric analysis CoAl_2O_4 samples was calcined at 400, 600, 800, 1000°C and investigated using an X-Rite Color i7 Benchtop spectrophotometer with standard lighting D65. Results showed appreciable color changes depending on calcination temperature. CIE $L^*a^*b^*$ colorimetric parameters of samples were determined.

For prepared spinel antistructure model of formation was described:



Sol-gel route is very useful to preparing spinel oxide powders and it gives good results requiring not much time and costs.