

Panitat Hasin 2007: Synthesis and Characterization of  $\text{NiAl}_2\text{O}_4$  Spinel.  
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Reaction of aluminium hydroxide, triethanolamine and various nickel(II) salts in ethylene glycol provides, in one step, an effective metallo-organic precursor for nickel aluminate spinel ( $\text{NiAl}_2\text{O}_4$ ). By using  $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ,  $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ ,  $\text{Ni}(\text{OH})_2$ , or  $\text{Ni}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$  in the one step reactions, four metallo-organic precursors, namely SPNO, SPCl, SPOH and SPAC, respectively were prepared. On the basis of mass spectroscopy,  $^1\text{H-NMR}$ ,  $^{13}\text{C-NMR}$  and infrared spectroscopy, the precursor structure was proposed to be a trimetallic double alkoxide consisting of two four-coordinate TEA-Al (alumatrane) moieties linked via a bridging TEA group that enfolding  $\text{Ni}^{2+}$  cation. All metallo-organic precursors were converted to nickel aluminate powder by calcinations. FTIR and XRD results confirmed the single phase of  $\text{NiAl}_2\text{O}_4$  powder after calcinations of SPNO and SPAC precursors at  $1000^\circ\text{C}$  for 5 h. However, the XRD patterns of the SPCl and SPOH calcined products showed two mixed crystalline phase, NiO and  $\text{NiAl}_2\text{O}_4$ . The morphology as well as surface area and porosity characteristics of the  $\text{NiAl}_2\text{O}_4$  spinel powders obtained from SPNO, SPCl, SPOH and SPAC precursors were characterized by SEM and BET surface area measurement. The pyrolyzed products exhibit porous microstructure with irregular shaped of block-like particles and have not high surface area ( $20 - 50 \text{ m}^2/\text{g}$ ). Nickel aluminate spinel powder was alternatively prepared via sol-gel process, using SPNO and SPAC precursor solutions. At room temperature, the optimal conditions for SPNO gel formation were found in ethanolic solutions with concentration range of 32.0 %, 28.0 – 32.0 %, 24.0 – 32.0% and 20.0 – 32.0 % (w/v), for pH 6, 7, 8 and 10, respectively. Whereas SPAC gels formed in i-propanolic solutions, pH 7.0 with concentration of 20.0 – 32.0 % (w/v) and 16.0 % (w/v), at pH 8.0. Heat treatment of obtained SPNO and SPAC gels at  $1000^\circ\text{C}$  for 5 h in air produced mixed phases of nickel aluminate spinel and nickel oxide which were confirmed by XRD. From SEM and BET surface area analysis, it was found that  $\text{NiAl}_2\text{O}_4$  powders showed homogenous variation in microstructures and has slightly different surface area as compared to that directly calcined spinel precursor.

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