

Panitat Hasin 2007: Synthesis and Characterization of NiAl₂O₄ Spinel.
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Chemistry. Thesis Advisor: Assistant Professor Nattamon Koonsaeng, Ph.D.
177 pages.

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 (NiAl₂O₄). By using Ni(NO₃)₂·6H₂O, NiCl₂·6H₂O, Ni(OH)₂, or Ni(CH₃COO)₂·4H₂O in the one step reactions, four metallo-organic precursors, namely SPNO, SPCI, SPOH and SPAC, respectively were prepared. On the basis of mass spectroscopy, ¹H-NMR, ¹³C-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 Ni²⁺ cation. All metallo-organic precursors were converted to nickel aluminate powder by calcinations. FTIR and XRD results confirmed the single phase of NiAl₂O₄ powder after calcinations of SPNO and SPAC precursors at 1000°C for 5 h. However, the XRD patterns of the SPCI and SPOH calcined products showed two mixed crystalline phase, NiO and NiAl₂O₄. The morphology as well as surface area and porosity characteristics of the NiAl₂O₄ spinel powders obtained from SPNO, SPCI, 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 m²/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°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 NiAl₂O₄ powders showed homogenous variation in microstructures and has slightly different surface area as compared to that directly calcined spinel precursor.

Student's signature

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