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# Synthesis, Characterization and Catalytic Activity of $Ni_xCo_{1-x}Al_2O_4$ Spinel

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**Abstract** – Complex oxides with spinel structure are of intense interests in material research because of their remarkable optical, electrical, magnetic, catalytic properties and widespread applications in science and engineering. In this work, $Ni_xCo_{1-x}Al_2O_4$ (x= 0; 0,3; 0,5; 0,7; 1) were prepared by combustion synthesis. All catalysts were nanometer scale and presented the spinel structure with the presence of $Al_2O_3$, NiO and/or CoO as impurity with high conversion of n-heptane to produce syngas.

Complex oxides (containing two or more types of cations) with spinel structure are of intense interests in material research because of their remarkable optical, electrical, magnetic, catalytic properties and widespread applications in science and engineering [1]. The spinel structure, featuring the general formula $AB_2O_4$ displays 64 tetrahedral sites and 32 octahedral sites, of which only 8 tetrahedral sites and 16 octahedral sites are occupied by the cations $A^{2+}$ and $B^{3+}$, respectively.

Nickel aluminate ($NiAl_2O_4$) is a mixed cation oxide with normal spinel structure, where $Al^{3+}$ occupies the octahedral sites and $Ni^{2+}$ occupies the tetrahedral sites. Cobalt aluminate ($CoAl_2O_4$) is a double oxide with a normal spinel type structure too, in which $Co^{2+}$ ions are accommodated in tetrahedral positions while $Al^{3+}$ ions are in octahedral positions [2]. It has been widely used as a ceramic blue pigment as well as heterogeneous catalyst. Several preparation methods have been studied to obtain nanocrystalline nickel aluminate with small particle size, such as ultrasound irradiation and microwave heating. The solgel method provides powder with small particle size and high surface area, however, this method releases dangerous gases which require great care during sintering [3].

In this work, $Ni_xCo_{1-x}Al_2O_4$ (x= 0; 0,3; 0,5; 0,7; 1) were prepared by combustion synthesis and characterized by X-ray diffraction (XRD), thermal analysis (TGA), differential scanning calorimetry (DSC), scanning electron microscopy (SEM), energy dispersive X-ray spectrometry (EDX) and superficial area by $N_2$ adsorption method (BET).

Catalytic activity was evaluated through the partial oxidation of n-heptane. The reactions were performed in a borosilicate U-tube containing 250 mg of each catalyst which was activated at 200ºC before reaction. The samples was then heated to the reaction temperature of 240ºC and the flow was switched to obtain 12,6 mol $h^{-1}$ $m_{cat}^{-1}$ molar flow (n-heptane per catalyst weight – F/W). The end of reactor is connected in a gas chromatography Varian CP3800 coupled with thermal conductivity detection through stainless-steel line.

All catalysts presented the spinel structure with the presence of $Al_2O_3$, NiO and/or CoO as impurity with high conversion of n-heptane to produce syngas (CO and $H_2$).

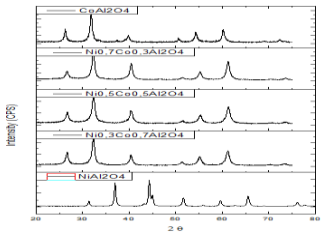


Figure 1: XRD patterns of $Ni_xCo_{1-x}Al_2O_4$ spinels.

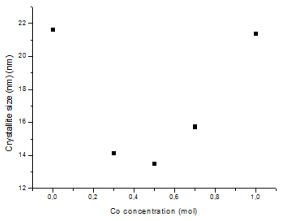


Figure 2: Crystallite size variation with Co concentration.

## References

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