

### 3. Characterization of experimental products.

#### 3.1 Characterization of nickel aluminate precursor

##### 3.1.1 Mass spectroscopy (MS)

The molecular masses of precursor products were characterized by Electrospray Ionization Mass Spectrometer (Bruker Esquire mass spectrometer). The mass range of sample was scanned from  $m/z = 100$  to 1000. The sample was dissolved in methanol.

##### 3.1.2 Nuclear Magnetic Resonance Spectroscopy (NMR)

The molecular structures of precursor products were investigated by  $^1\text{H}$ - and  $^{13}\text{C}$ -NMR (INOVA VARIAN NMR Spectrometer 400 MHz). The NMR spectrometer operating in the quadrature mode was used to obtain  $^1\text{H}$ - and  $^{13}\text{C}$ -NMR spectra. Tetramethylsilane [ $(\text{CH}_3)_4\text{Si}$ , TMS] and deuterated dimethylsulfoxide ( $\text{DMSO-d}_6$ ) were used as the reference and standard solvent; respectively. All chemical shifts are recorded in ppm. Typical  $^1\text{H}$ -NMR spectra consisted of 16 transients of 23,954 data points over a 6.39 KHz bandwidth using a 3.2  $\mu\text{s}$  at  $45^\circ$  pulse. Typical  $^{13}\text{C}$ -NMR spectra consisted of 25,600 transients of 30,144 data points over a 25.16 KHz bandwidth using a 7.25  $\mu\text{s}$  at  $45^\circ$  pulse.

##### 3.1.3 Thermogravimetric analysis (TGA)

Thermogravimetric Analyzer (Perkin-Elmer TGA 7) was used for thermal decomposition studies of organic contents in nickel aluminate precursors. Each 10 mg sample was placed in a platinum pan and heated in flowing  $\text{N}_2$  (20 psi). The heating range is  $27^\circ\text{C}$  to  $1000^\circ\text{C}$  and the heating rate is  $5^\circ\text{C}/\text{min}$ .

## 3.2 Characterization of nickel aluminate spinel

### 3.2.1 X-Ray Diffraction (XRD)

The X-ray Diffractometer (Philips P.W.1830) was used to characterize the purity of calcined products by using nickel-filtered  $\text{CuK}\alpha$  ( $\lambda = 0.154 \text{ nm}$ ) radiation as an X-ray source. The diffraction patterns were recorded over a range of  $2\theta$  angles from 15 to  $80^\circ$ . The crystalline phases of samples were identified by comparing the peak positions of X-ray diffraction pattern obtained with the Joint Committee on Powder Diffraction Standards (JCPDS) files.

### 3.2.2 Fourier Transform Infrared Spectroscopy (FTIR)

Fourier Transform Infrared Spectrophotometer (Perkin-Elmer 2000-FTIR) was used to record the infrared spectra of calcined samples. Potassium bromide (KBr), powdered with an argate morta and pestle, was used as the nonabsorbing media. Samples were prepared using 0.3-0.5 wt% analyte rigorously mixed with the powdered KBr.

### 3.2.3 Scanning Electron Microscopy (SEM)

The Scanning Electron Microscope (JEOL JSM-6301F) operating at an acceleration voltage of 20 kV, a work distance (w.d.) of 15 mm and magnification values in 10,000x – 50,000x was employed to investigate the microstructure of powder samples. The powder sample was mounted on alumina stubs using a liquid carbon paste. Samples were sputter coated with Au to avoid particle charging.

### 3.2.4 Surface Area and Porosimetry

The specific surface areas of the powder samples were determined by Surface Area and Porosimetry Analyzer (Micromeritics ASAP 2020 surface analyzer). The BET surface areas were calculated from the nitrogen adsorption isotherms at 77 K and a value of  $0.162 \text{ nm}^2$  for the cross-section of the nitrogen molecule. Prior to analysis, samples were degassed at 623 K under high vacuum for 20 h.

The total surface area ( $S_t$ ) of the sample can be expressed as:

$$S_t = \frac{W_m \cdot N \cdot A_{cr}}{M} \quad \dots(6)$$

where  $W_m$  = The weight of adsorbate constituting a monolayer of surface coverage.

$N$  = Avogadro's number ( $6.023 \times 10^{23}$  molecules/mol) and  $M$  is the molecular weight of the adsorbate.

$A_{cr}$  = The molecular cross-sectional area of the adsorbate molecule (nitrogen is  $16.2 \text{ \AA}$ ).

$M$  = The molecular weight of the adsorbate.

The specific surface area ( $S$ ) of the solid can be calculated from the total surface area  $S_t$  and the sample weight  $W$ , according to equation 7:

$$S = S_t / W \quad \dots(7)$$

The total pore volume ( $V_t$ ) is calculated by using equation 8:

$$V_t = P_a \cdot V_{ads} \cdot V_m / RT \quad \dots(8)$$

which  $P_a$  and  $T$  = Ambient pressure and temperature, respectively.

$V_{\text{ads}}$  = The volume of nitrogen adsorbed.

$V_m$  = The molar volume of the liquid adsorbate ( $34.7 \text{ cm}^3/\text{mol}$  for nitrogen).

$R$  = Gas constant ( $0.0821 \text{ L.atm/K.mol}$ )

The average pore radius ( $r_p$ ) can be expressed as

$$r_p = 2 V_t / S \quad \dots(9)$$

where  $V_t$  is obtained from equation 8 and  $S$  is the BET surface area.