3 MATERIALS AND METHODS

3.1 Introduction

This chapter provides information and application on chemicals and gases used during catalyst preparation and dry reforming experimental work. It also includes theoretical and procedures of catalyst preparation by impregnation method and catalyst characterization instruments in detail. Lastly, detail description on dry reforming experimental work was also presented in this chapter.

3.1.1 Chemicals

Glycerol (Fisher Scientific, 99.95% purity, liquid), nickel (II) nitrate hexahydrate (Acros Organics, 99%, powder), cobalt (II) nitrate hexahydrate (Sigma Aldrich, 98%, powder) and lanthanum (III) oxide (Acros Organics, 99.99%, powder) was employed for this work. Glycerol is used as a feedstock in dry reforming, nickel powder and cobalt powder as based catalyst whereas lanthanum oxide were used as a catalyst support. The list of gases used in this research are carbon dioxide (CO_2) and nitrogen (N_2). All this gases were provided by Faculty of Chemical and Natural Resources Engineering Laboratory, Universiti Malaysia Pahang. Carbon dioxide is used as reactants of dry reforming process and nitrogen gas is used as reforming agent carrier gas for the glycerol dry reforming process.

3.2 Catalyst Preparation

5 wt % Ni/La_2O_3, 10 wt % Ni/La_2O_3, 15 wt % Ni/La_2O_3 and 10 wt % Co/La_2O_3 was prepared by wet impregnation method. Initially, La_2O_3 support from Acros Organics was calcined at 1073 K for 6 hours in a Carbolite furnace. Nickel powder was weighed and filled in the beaker with deionized (DI) water. After that, an accurately weighted calcined La_2O_3 was mixed with $Ni(NO_3)_2.6H_2O$ aqueous solution and then magnetically-stirred for 3 hours. Then, the slurry was dried overnight in an oven at 383 K for 12 hours to remove excess water. The product was crushed and calcined again for 6 hours at 1073 K. It was then cooled down and sieved to a particle size of 90-200 µm for characterization and reaction studies. The procedures was repeated for 10 wt % Co/La_2O_3.

3.3   Catalyst Characterization

The catalyst were undergo the characterization analysis using Scanning Electron Microscopy (SEM), Brunauer-Emmet-Teller (BET), X-Ray Diffraction (XRD) and Fourier Transform Infrared Spectroscopy (FTIR). The surface structure of catalysts were determined by SEM, specific surface area of catalyst is obtained from BET and crystalline structure of catalysts were obtained from XRD analysis. Then, FTIR was also performed to obtain the functional group of catalysts.

3.3.1   Scanning electron microscopy (SEM)

Scanning electron microscopy (SEM) is performed on catalyst to analyse the catalyst morphology. The surface structure of the catalysts were captured by using Scanning Electron Microscopy (SEM) unit JOEL.JSM- 7800F model. The analysis was carried at 3kV with 10,000 × magnification.

Figure 3-1: Scanning electron microscopy (SEM)

3.3.2 Bruanauer-Emmett-Teller (BET)

Brunauer-Emmett-Teller (BET) analysis was carried out using Thermo-Scientific Surfer to determine the specific surface area of the calcined catalysts. In the analysis, liquid N_2 with a cross-sectional area of 16.2 $\mathring{A}^2$ was used as the adsorbate at temperature 77 K. Temperature ramping for this analysis is 573 K.

Figure 3-2: Bruanauer-Emmett-Teller (BET)

3.3.3 X-ray Diffraction (XRD)

X-Ray Diffraction (XRD) analysis was conducted using Rigaku Miniflex II to obtain crystalline structure of the catalysts. This analysis was carried out using CaKα radiation (λ = 1542 Aᵒ) at 15 mA and 30 kV. The scan rate was 1° min⁻¹ for values between 2θ = 10° and 80°.