

CHAPTER III EXPERIMENTAL

3.1 Materials

Gamma alumina, $\gamma\text{-Al}_2\text{O}_3$, used as a catalyst support was purchased from Saint-Gobian NorPro Corp. Alpha alumina, $\alpha\text{-Al}_2\text{O}_3$, used as an inert catalyst bed support for keeping the height of catalyst bed was obtained by Saint-Gobian NorPro Corp. Nickel nitrate, $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, used as a nickel source of catalyst was purchased from BDH. Hydrogen with 99.99% purity and tetralin (>97%, Fluka) were used as reactants. Nitrogen with 99.99% purity was used as the inert gas for building up the pressure and cleaning line.

3.2 Experimental

3.2.1 Catalyst Preparation

$\text{Ni}/\text{Al}_2\text{O}_3$ catalyst was prepared by impregnating supports ($\gamma\text{-Al}_2\text{O}_3$) with an aqueous solution of $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$. Impregnated resultant was left in air for 5 hours and then dried in an oven at temperature of 110 °C for 12 hours. Finally, it was calcined at 400 °C for 2 hours.

3.2.2 Catalyst Characterization

Atomic Absorption Spectrometric Analysis (AAS)

The metal loading on support was determined by using AAS (Spectr-300, Varian). The flame was burned with air-acetylene. Nickel solution was prepared by digesting $\text{Ni}/\text{Al}_2\text{O}_3$ with a mixture of 1 ml HNO_3/HCl (5 : 1) and dilution with water.

Surface Area Analysis

Catalyst surface areas were estimated by using BET surface area analyzer (Autosorb-1, Quantachrome). The surface areas were analyzed by using

nitrogen gas as adsorbate. To eliminate adsorbed volatile compounds, adsorbents were dried and evacuated at 250 °C for 4 hours.

3.2.3 Hydrogenation Procedure

The catalyst was packed in the ¾” stainless steel packed reactor equipped with a thermowell that allows placing a thermocouple in the center of the bed. Before the experiments the catalyst was reduced in flowing hydrogen (250 ml/min) for 1.5 hours at 450 °C. The reactant and product concentrations were monitored using an on-line GC. In this work, hydrogen and tetralin were fed to the top of the reactor. At the conditions of this study, 250-300 °C and 300 psig, simulations carried out via PRO II® indicate that the feed will be in vapor phase. However, to ensure proper vaporization a layer of 5 cm glass beads was placed before the catalyst bed.

3.2.4 Fitting of the Model

The reactor was modeled as an isothermal PFR. Due to the high activity of the catalyst the integral reactor analysis was used. The reaction rates were obtained at the steady state conditions. The estimation of kinetic parameters was performed by minimizing the sum of errors of the calculated and experiment mole fractions as shown Eq. (3.1), which y_i and $y_{cal,i}$ are the experimental and calculated mole fractions, respectively.

$$\sum_{\min} [(y_i - y_{cal,i})]^2 \quad (3.1)$$