CHAPTER III
EXPERIMENTAL

3.1 Materials

Aluminium hydroxide hydrate [$Al(OH)_3$ xH_2O] was purchased from Sigma Chemical Co. Triisopropanolamine (TIS, $N(CH_2CHCH_3OH)_3$) and nickel acetate [$(CH_3COO)_2Ni$] were obtained from Aldrich Chemical Co. Inc.(USA). They were used as a received. Ethylene glycol (EG, $HOCH_2CH_2OH$) used as solvent for the alumatrane synthesis was purchased from J.T. Baker Inc. (Phillipburg,USA). Acetronitrile (CH_3CN) and methanol were obtained from Lab-Scan Company Co., Ltd. and distilled before use. Nitric acid and ammonia solution used to adjust pH in the sol-gel process were purchased from Lab-Scan Company Co., Ltd., and used as received.

3.2 Equipment

Functional groups of materials were followed using FTIR spectrophotometer (Nicolet, NEXUS 670) with a resolution of 4 cm^{-1}. The solid samples were mixed and pelletized with dried KBr. Thermogravimetric analysis (TGA) was carried out using TG-DTA (Pyris Diamond Perkin Elmer) with a heating rate of 10°C/min in the range of room temperature to 750°C under nitrogen atmosphere to determine the thermal stability of alumatrane. Powder X-ray diffraction (XRD) patterns were carried out to characterize crystallinity of samples using a Rigaku X-ray diffractometer with CuKα as a source. A range from 5° to 90° was investigated using a step of 5°/min. The diffuse reflectance UV-VIS spectra were collected on a SHIMADZU UV 2550-VISIBLE spectrophotometer in the range of 190 to 900 nm. The reducibility was investigated by temperature-programmed reduction on TPD/R/O/MS Themo Finnigan 1100. The reducing gas is 5% H_2 in N_2 at a flow rate of 40 ml/min. The rate of temperature was carried out at the heating rate of 10°C/min ranging from room temperature up to 900°C. The BET surface area measurement, pore volume and pore size distribution were measured by using nitrogen at 77 K in

Autusorb-1 gas sorption system (Quantasorb JR). Samples were degassed at 250°C under a reduced pressure prior to each measurement. The morphology was studied on SEM using JEOL 5200-2AE scanning electron microscope.

3.3 Methodology

3.3.1 Synthesis of Alumatrane by the OOPS process

The preparation of alumatrane or tris(alumatranyloxy-*i*-propyl)amine was followed Wongkasemjit *et al*'s works [5,7] via the Oxide One Pot Synthesis (OOPS) process. Aluminium hydroxide, TIS and EG are added into a 250 ml two-necked round bottom flask. The mixture was homogeneously stirred at room temperature before being heated to 200°C under nitrogen in an oil bath for 10 h. Excess EG was removed under vacuum (10^{-2} Torr) at 110°C to obtain crude product. The crude solid was washed with acetonitrile and dried under vacuum at room temperature. Dried products were characterized using TGA and FTIR.

3.3.2 Nickel Loading onto Alumina Using Alumatrane Precursor via the Sol-gel Process

Alumatrane was used as an alumina source via the sol-gel process at various Ni/Al ratios, pH, hydrolysis ratios and calcinations temperature. Alumatrane and nickel acetate were dissolved in methanol for 1 h before adding water and adjusting pH. The pH value of the unadjusted mixture solution is pH 9. For acid conditions, pH 3, 5 and 7, HNO_3 was used whereas NH_4OH was added for obtaining pH 11. Three hydrolysis ratios of 9, 18, 27, as followed the previous work [5], were investigated. The solution was vigorously stirred at room temperature followed by heat treatment of the resulting gels at calcinations temperature ranging from 500°C to 900°C and held at the final temperature for 7 h.

3.3.3 Activity Testing on CO Oxidation Reaction

The catalytic tests of CO oxidation with O_2 were carried out in a fixed-bed flow reactor in the temperatures ranging from 200°-450°C. A 0.2 g of catalyst

was employed for each experiment. For the reaction condition, 180 ml/min total gas flow was maintained with feeding the reaction mixture of CO-O_2-He (1%-2%-97%).