CHAPTER III

EXPERIMENTAL

This chapter describes experimental system and procedures for alumina nanofibers. It is divided into three parts, i.e. chemicals used, preparation of samples and characterization of the obtained products, respectively.

3.1 CHEMICALS

All chemicals using in this research are as follows:

1. Aluminum isopropoxide (98%+) ($[CH_3]_2CHO)_3Al$) available from Aldrich Chemical Co., Ltd., Germany.
2. Polyvinyl alcohol with molecular weight of 72,000 available from Fluka Chemical Co., Ltd., USA.
3. Acetic acid (99.5%) (CH_3COOH) available from BDH Chemical Co., Ltd., England.

3.2 EXPERIMENTAL PROCEDURES

3.2.1 Preparation of spinning solution

The method to produce alumina sol from aluminum isopropoxide ($[CH_3]_2CHO)_3Al$), has been proposed by Yoldas since 1974. Boehmite (AlOOH) is synthesized from a reaction between solution of aluminum isopropoxide and water. [Kobayashi et al.2005; Yoldas 1974; Kolaczkowski and Kim 2006]:

In this research, aluminum isopropoxide (AIP) was used as the source of alumina. Polyvinyl alcohol (PVA) was used as the spinning aid. Acetic acid was used as peptizing agent. For the preparation of the spinning solution, AIP was first hydrolyzed with water at temperature in range of 80-85°C under vigorous stirring for 1 h. The molar ratio of water, AIP, and acetic acid was normally set at 100:1:0.15.

The solution was further stirred at 80-85°C for desired period of aging time, in the range of 0 to 48 hrs. Then, 5.75 wt% of PVA was dissolved into the water which is equivalent to the solution. The mixture was stirred under mild heating for a sufficient time to ensure complete dissolve of PVA. The mixture obtained was referred to as the spinning solution.

3.2.2 Preparing of alumina composite fibers

The electrospinning system is consisted of a syringe, a stainless steel needle, a collector (stainless steel net) and two electrodes attached to high voltage DC power supply, Protek DC Power Supply DF1730SB3A. A voltage of 8.5 kV was applied between tip of the syringe tip and the grounded collector, which were set at ca. 12 cm apart. The electrospun fibers were collected as sheet of non-woven fibers on the collector. In the comparative study, alumina powder was prepared by hydrolyzing AIP and subsequently ageing for 24 hrs, in the same manner as that previously described. Then the mixture was placed in an oven at 110°C to remove all the solvent.

The powder and fibers were calcined at temperature in the range of 500-1200°C for 2 hrs using the heating rate of 10°C/min. The calcined products were collected and characterized by various analytical techniques, such as, X-ray diffraction, FTIR spectroscopy, scanning electron microscopy, transmission electron microscopy, and thermogravimetric and differential thermal analysis.

3.3 CHARACTERIZATIONS

3.3.1 X-ray diffraction (XRD)

The X-ray diffraction (XRD) analysis of the product was performed by a SIEMENS D5000 X-ray diffractometer, using Ni-filtered CuKα radiation. The scan was performed over the 2θ range from 20° to 80°.

Crystallite size of the powder was estimated from XRD line broadening according to the Scherrer equation. The value of shape factor, *K*, was taken to be 0.9 and α-alumina was used as an external standard.

3.3.2 Scanning electron microscopy (SEM)

Morphology of the samples was observed on JEOL JSM-5410LV scanning electron microscope at the Scientific and Technological Research Equipment Center (STREC), Chulalongkorn University. The SEM was operated using the secondary electron mode at 15 kV.

3.3.3 Fourier transforms infrared spectroscopy (FTIR)

Functional groups in the samples were identified by using an infrared spectroscopy (Nicolet Impact 400). Before measurement, the sample was mixed with KBr and formed into a thin pellet. The scan was performed over the Wavenumber from 400 to 4000 cm^{-1}.

3.3.4 Thermogravimetric and differential thermal analysis (TG-DTA)

The as-spun alumina fibers was subjected to the thermogravimetric and differential thermal analysis (Diamond Thermogravimetric and Differential Thermal Analyzer, TA Instruments SDT Q600) to determine the carbon content in the sample, as well as their thermal behaviors in the range of 20-1300°C. The analysis was performed at a heating rate of 10°C/min in 100 ml/min flow of air.

3.3.5 Transmission Electron Microscope (TEM)

The morphology of an individual grain in the samples was observed on a JEOL JEM-2100 Analytical Transmission Electron Microscope, operated at 80-200 keV at the National Metal and Materials Technology Center, Thailand. The crystallographic information was also obtained from the selected area electron diffraction (SAED) analysis performed in the same instrument.

3.3.6 Viscosity measurement

The viscosity of electrospinning solutions were measured on a Brookfield Model DV III viscometer at The Petroleum and Petrochemical College, Chulalongkorn University, Thailand. The samples were prepared at room temperature and the percentage of torque performed was over 90% throughout the measurement.