EFFECTS OF THE POLYVINYL ALCOHOL (PVA) ON THE SYNTHESIS OF ALUMINA FIBERS THROUGH ELECTROSPINNING TECHNIQUE

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Abstract— Ceramic fiber products especially alumina has good properties such as high modulus and strength that were resistant toward the attacks of molten metals and non-oxide materials up to 1000°C and also have low thermal conductivity and high melting point. Alumina is a well-known structural material among functional ceramics in the area of catalysts, microelectronics and optic, both in pure or doped form as well as in combination with others. In this work, alumina fiber was synthesized by sol-gel electrospinning technique (e-spinning). The sol was prepared by mixing aluminum nitrate nonahydrate (ALN) and aluminum isopropoxide at 3 molar ratios. The precursor sol was stirred vigorously for 24 hours until ALP completely dissolved. After stirring, the precursor sol with PVA and without PVA was evaporating to obtain the suitable viscosity to spin. The rheology and viscosity were checked by viscometer. At the appropriate viscosity, the sol was spin into fibers using electrospinning machine. The green fibers were then dried at room temperature then calcined at 1200°C. The fibers produced were characterized using X-Ray Diffraction (XRD) and Field Emission Scanning Electron Microscope (FESEM). The XRD analysis had shown the α-alumina was formed at 1200 °C and SEM micrograph shows the formation of fibers with PVA addition.

Keywords: Electrospinning, alumina fibers, polyvinyl alcohol

I.INTRODUCTION

Alumina is a well-known structural material among functional ceramics in the area of catalysts, microelectronics and optic, both in pure or doped form as well as in combination with others [1]. Alumina is widely used as structural component for high temperature applications, heat engine and aerospace application, as electronics substrate. It is also used for adsorption of heavy toxic metal ions such as arsenic or arsenate. The adsorption depends on exposed surface area, so nanofibers with high surface area produced by electrospinning might be ideal for this application [2].

There are two methods named melting-based method and sol-gel processing in order to produce ceramic fibers products. The preparation of alumina nanofibers by spinning melt method is very difficult due to the exceeded high melting temperature and the low viscosity of the melt [3]. The molten ceramic should be viscous but have a low surface tension in order to be drawn into fiber form; even so a considerable fraction of the ceramic is not drawn and known as 'shot'. Even the sol-gel processes is more expensive than the melt blown process but greater control over the final products is possible and the fibers can be made with much higher alumina content [4]. The sol-gel processing has advantages such as much lower temperature processing, homogeneity of products, uniform diameter of fibers, fine grain size and better control over final properties [3].

In order to produce alumina fibers, electrospinning is of bottom-up approaches that are gaining increasing attention in recent years for the fabrication of ceramic nanostructures. Electrospinning is also relatively easy and fast process to produce nanofibers [5]. In

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electrospinning, a high voltage is applied to polymer fluids such that charges are induced within the fluid. When charges within the fluid reached a critical amount, a fluid jet will erupt from the droplet at the tip of the needle. The electrospinning jet will travel towards the region of lower potential, which in most cases, in a grounded collector [6]. Before the electrospinning were stated, the solution condition need to be considered because any changed or the differential between the solution will affected the properties of the fiber formed especially in fibers morphology [6].

There are many types of precursors that were used in producing fibers via electrospinning. Based on Hasmaliza's work (2008), it was found the sol was prepared by aluminum isopropoxide (ALP) which dissolved with 0.5M aluminum nirate nonahydrate (ALN) [7]. But, the technique that was used is conventional spinning where the fibers dimension formed are in micron size. Panda and Ramakrishna (2007) explored the used of other sol gel aluminum precursors such as aluminum nitrate and aluminum acetate in polymers, polyvinyl alcohol (PVA) and poly (ethylene oxide) (PEO). Salt to polymer concentration were set at 10, 50 and 100 wt%. These researchers reported issues with beading and structural deformities with all three loadings. In this work, the effect of producing alumina fibers through electrospinning technique using alkoxide route with and without PVA as additive on the fibers properties was studied.

II.EXPERIMENTAL PROCEDURE

Preparation of the Sol Precursor

The starting materials used for preparing the precursor sols are aluminum nitrate nonahydrate (Al(NO3)3.9H2O) and aluminum isopropoxide ($Al[OCH(CH_3)_2]_3$). Aluminum nitrate nonahydrate was obtained from Systerm (CAS No:7784-27-2 & 98.5% purity) and aluminum isopropoxide was produced by Merck and its properties shown asTable 1. The PVA that was used in this experiment is PVA with M_w=72000.

Alumina fibers were prepared through sol-gel electrospinning. Firstly, 0.5M ALN was prepared by using distilled water. Then, aluminum isopropoxide were added to the 0.5M ALN at molar ratio of 3. The precursor sol was stirred with vigorously stirred for 24 hours.

Preparation of the Fibers

After stirring, the PVA was added then the mixture was evaporated by Heidolph Laborota Rotary Evaporator at controlled water bath temperature (<60°C below than ALN melting point). The spinnability of the sols was determined from the capability of fiber formation by immersing the glass rod into the sols, then pulling it up quickly. The spinning was done by control the spinning parameters and then the fibers were collected on the metal collector. They were then put in alumina crucible and calcined at 1200 °C with 5 °C/min heating rate.

Table 2.1: The properties both of two main raw materials

Properties	Raw Materials	
	ALP	**ALN**
Chemical formula	$Al[OCH(CH_3)_2]_3$	$Al(NO_3)_3.9H_2O$
Molecular weight	204.25 g/mol	375.13 g/mol
Density	1.035 g/cm^3	0.88 g/cm^3
Boiling point	94 °C	73 °C

Characterizations

In order to determine the viscosity of the alkoxide-water based alumina sol, the sol was analysed by *Haake Viscometer model VT550*. X-Ray diffraction test (XRD) was performed with the *model Bruker AXS D8 Diffractometer* to identify the phase transformation in alumina fibers. Microstructure and diameter of fibers were observed under Scanning Electron Microscope *model ZEISS SUPRA 35VP* to observe the effect of PVA addition on the fibers morphology.

III.RESULT AND DISCUSSION

It has been observed that the viscosity of the sol with PVA addition to produce fibers via electrospinning technique is in lower range, which is 0.2 Poise to 7 Poise. According to previous studies, the viscosity of the sol must be in narrow range where the viscosity values between 1.6 Poise to 20 Poise will form fibers [8]. The viscosity of the sol should not exceed the maximum value because the solidification takes place during spinning and the sol cannot be spun through the needle. If the viscosity becomes lower than

minimum value, the sol just passes the needle without formed any fibers. Whereas the viscosity of the spinnable sol without PVA addition was higher (about 70 Poise). But it was difficult to form fibers because the viscosity were exceeds the maximum value. The small amount of solution was passing through the needle and the rest was stucked in needle. The fibers formations need an interparticles bonding in solution to create a long chain of particles to improve the spinnabilitity. The PVA addition was improve the spinnability of the sol because PVA was assist the particles inside the sol to create a bonding, whereas the sol without the PVA need to create a bonding between the particles by their own by remove the water. The interacting was growth in low water content and automatically increase the viscosity [9]. Figure 1 show the fiber formation using spinnability sol (with PVA addition).

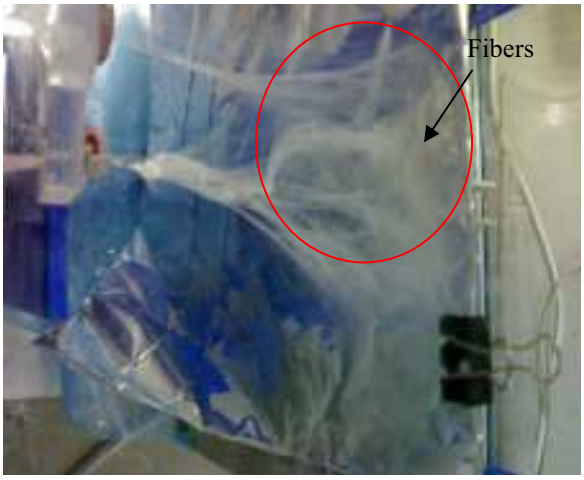

Figure 1: The fiber formation during electrospinning process

Whereas Figure 2 shows the alumina fibers produced without the Polyvinyl Alcohol (PVA) addition. The fibers look dry, ununiform diameter and sticking with each others. Cracks were also observed on the fibers due to the high viscosity of the sol during electrospinning. The higher viscosity was increased the diameter of fibers [6].

Figure 3 shows the alumina fibers that were produced with PVA addition. The purpose of PVA in this study was act as a binder to assist in enhancing spinnability. PVA is a water-soluble synthetic polymer which is odorless and non-toxic. It is fully degradable and is quick dissolver [10]. The addition of PVA was produced the uniform fibers diameters where the size are thinner compared to the fibers produced without the PVA. The PVA inside the solution will create a bonding between the particles to assist spinnability at lower viscosity. The fibers surface also smooth with the PVA addition due to the small amout of PVA used. The amount of fibers also increased with the decreasing of viscosity because the solutions easy to jet over the needle tip to the collector [11]. The jet was dried during flying and as a result, the fibers not stick to each other.

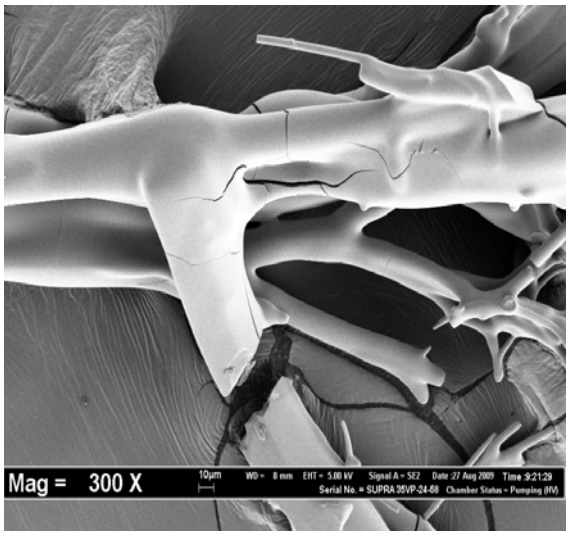

Figure 2: Alumina fibers without PVA

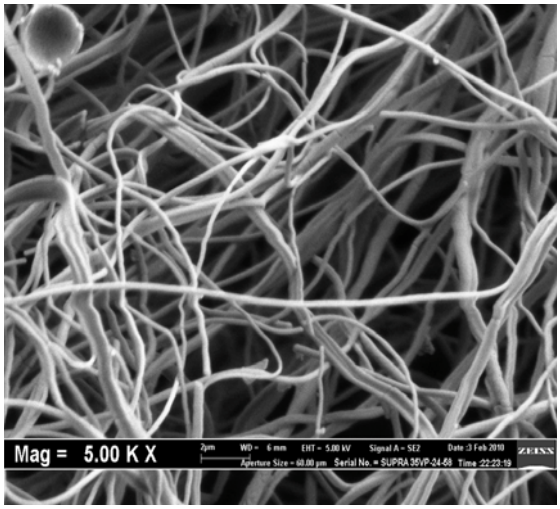

Figure 3: Alumina fibers with PVA

The XRD result for the fibers calcined at 1200°C with and without PVA addition was shown in Figure 4. It was observed that pure α-alumina phase was occurred. The phase transformation of α-alumina was completely occurring at 1200°C [12]. Hasmaliza (2008) was study that the formation of single phase of α-alumina was started at 1100°C and become stable at 1200°C. The PVA

totally removed during calcinations process due to the PVA properties which can evaporate at 280°C. So, the addition of PVA was not effect the purity of the fibers phase.

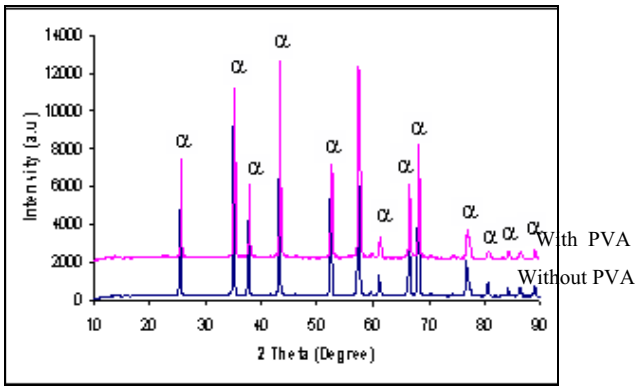

Figure 4: XRD data of Alumina Fibers.

IV. CONCLUSION

PVA addition gives significant effect on the fibers formation. Uniform fibers were produced using sol-gel alkoxide system with addition of PVA as a binder via electrospinning technique. Purity of the alumina was not effected by the PVA addition where the pure α-alumina phase was observed at 1200°C.

ACKNOWLEDGEMENT

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