RESEARCH ARTICLE

The effect of adding different amount of spinning additives on preparation of nano Alumina fibers using a combined method of sol gel and electrospining

F. Mirjalili[1], F. Danafar[2], A. Zarei Mahmodabadi[1]*

[1] *Department of Material Engineering, Maybod Branch, Islamic Azad University, Maybod, Iran*

[2] *Department of Chemical Engineering, University of Shahid Bahonar, Kerman, Iran*

ARTICLE INFO

Article History:
Received 2020-5-28
Accepted 2020-7-14
Published 2020-08-01

Keywords:
Nano alumina fiber
sol-gel method
electrospinning

ABSTRACT

Alumina nano fibers were produced using a combined method of sol-gel and electros pinning method. The sol was first prepared by mixing of tri-isoproxide aluminum in aqueous solution of nitric acid. In the next step, ethylene glycol, acid lactic and poly vinyl alcohol were added to the solution .Finally, the sol was then heated at 80 °C to obtain gel. The resulting gel was converted to nano fibers of aluminum oxide by electrospinning method. The nano fibers were dried at 60 °C for 24 h and sintered at 1200 °C for 2 h. The results of XRD and FTIR showed that, in all samples the dominant phase was the corundum phase. To decrease the sintering temperature, magnesium nitrate hexahydrate (MgN_2O_6 * $6H_2O$) and sodium ethoxylate (SiC_8 H_2o O_4) were added to the sol containing 10% Polyvinyl alcohol. The XRD and FTIR analysis of this sample indicated the presence of corundum phase when the sintering temperature of 1000°C was used. Accordingly, the energy consumption was reduced as the sintering temperature decreased about 200°C by adding the additives. The SEM and TEM analysis also showed formation of non-agglomerated nano fibers with diameters of 41nm when 10% polyvinyl alcohol and 2% sodium ethoxylate (SiC_8 H_{20} O_4) was added to the sol.

How to cite this article
Mirjalili F., Danafar F., Zarei Mahmodabadi A. The effect of adding different amount of spinning additives on preparation of nano Alumina fibers using a combined method of sol gel and electrospining. J. Nanoanalysis., 2020; 7(3): 206-215. DOI: 10.22034/jna.005.

INTRODUCTION

Alumina is one of the most important materials because of, its high strength and modulus, resistance to attacks from molten metals and non-oxide materials, chemical inertness in both oxidizing and reducing atmospheres up to 1000 °C, and good electrical insulation [1-3]. Alumina (Al_2O_3) is a ceramic material with several metastable polymorphs (such as χ, κ,γ,δ,θ-Al_2O_3), and a stable phase, which is crystallized at high temperature (α-Al_2O_3 [4].

Alumina fibers are often used as high temperature insulating material due to their high melting point (Tm > 2040 °C) and low thermal conductivity (<0.5 W/m K) [5]. Convectional thermal insulating fibers are alumino silicate. An Alumina type can be used at high temperature, i.e.1700 °C, for a long period of time that provides 20% energy savings more than other types of alumino silicate fibers [6].There are two methods named melting-based method and sol–gel processing to produce ceramic fibers [7,8]. The later method (sol–gel) has several advantages, such as lower processing temperature, more homogeneity of products, uniform diameter of fibers, lower diameter and better control over final properties of fibers. The preparation of alumina fibers by spinning melt method is very difficult, due to the exceeded high melting temperature and low viscosity of the melt [9].Electrophoretic deposition

* Corresponding Author Email: *Fm.mirjalili@gmil.com*

(EPD) has increasingly attracted an interest as a materials processing technique for a wide range of technical applications. This technique enables the production of unique microstructures and nanostructures as well as novel and complex material combinations in a variety of macroscopic shapes, dimensions and arrangements starting from micron-sized or nanosized particles [10, 11]. Summary of relevant recent works on EPD describes the application of this technique in the processing of several traditional and advanced materials.

For example, EPD has been used for the preparation of metal oxide/ceramic nano fibers such as silica, zirconia, titania, nickel oxide, barium titanate, lead zirconate titanate and other oxide materials [12-15].

Poly(vinyl alcohol) (PVA) is used as a biomedical polymer due to their good chemical and physical properties like water solubility, chemical resistance, high melting point and biological compatibility. In addition, it is an inexpensive, non-toxic material having high tensile strength and flexibility. Electrospinning of PVA can be performed from aqueous solutions leading to generation of homogenous nanofibers [16].Chemical cross linking of the PVA nano fibers presents a suitable way for stabilization of the fibers towards aqueous surroundings. Synthesis of electrospun PVA fibers in term of their morphological and dimensional characteristics as well as their final concentration have been well studied to find relationships among various process parameters [17-19].

Fibers of PVA/SiO_2 have higher water solubility and thermal stability comparing to pure PVA nano fibers [10, 20]. It is generally important to obtain continuous fibers of high strength. Fibers used in thermal insulation are short. Which are stable. The stable fibers are usually post-processed to obtain the necessary forms [21-23]. Obviously; it is good to couple the fiber preparation and shaping steps to get rid of or to minimize any post-processed step in the centrifugal spinning method of sol–gel technology[24,25]. Chandradass et al (2006) synthesized composite fibers (Zro_2/Al_2O_3) from an aqueous solution of aluminum powder, aluminum chloride hexahydrate and yttrium oxide by sol-gel method. They prepared nano fibers without any agglomeration in the size range of 30-35 nm [1]. Sub micrometric alumina fibers were produced by the electro-blown spinning (EBS) technique and compared with alumina fiber mats prepared using the solution blowing spinning (SBS) method. This method was successful in producing excellent flexible and continuous alumina fibers with mean diameter of about 2.75 μm. These fibers have smaller diameter compared to SBS alumina fibers, which had a diameter of about 4.12 μm. Moreover, diameter distribution of the EBS fibesr was more uniform than the SBS's [26].Venkatesh et al (2002) prepared alumina nanofibers by hydrolyzing aluminum nitrate in the presence of hexamethylenetetramine (HMTA) followed by the supercritical fluid drying. These nanofibers were recommended to be used for biomaterials and tissue engineering [27]. Tepper et al., reported the effects of processing variables on the microstructure of alumina fibes. They used organics and spin-assist materials, which changed the required heating, timing and resulted in formation of high-strength fibers [17]. When organic materials are used in electro- spinning method, the surface roughness increases after bonding and thus the fibers strength decrease. Changing of heating scheduling also has an important impact on the formation of alumina fibers and a suitable scheduling leads to the formation of high-strength, high-strength fibers [28]. The researchers fabricated long fibers of alumina using the Al source in low cost and distilled water as a solvent by sol-gel method. Aluminum nitrate (AN) and malic acid (MA) were used as raw materials and, polyvinylpyrrolidone (PVP) was added as a spinning additive [29,30]. The δ-Al_2O_3 nano fibers were successfully synthesized, with a diameter of 2 nm and a length of 50 nm. The long quartz fibers have been produced by some factories, such as Mingda Co. Ltd, Jingzhou [31-32]. Continuous α-Al_2O_3 fibers were grown by seeding of α-Al_2O_3 suspension with in-situ suspension. The results showed that the calcination of 1200 °C was required for preparation of α-Al_2O_3 fiber with high density [33].

Dense alumina fibers were produced at 1600°C by sol-gel and electrospinning at low cost. Aluminum nitrate and polyvinyl pyrrolidone were used as a precursor ad spinning polymer, respectively. Plutonic 127 was also used as an additive to achieve a porous structure. These fibers with a high mole ratio presented a mean diameter close to nanometric scale 153 ± 39 nm [4]. In other work, Alumina borate nanofibers were fabricated by a method of electrospinning and sol-gel method combination. The results showed that if the content of PVP increased, the diameter of the

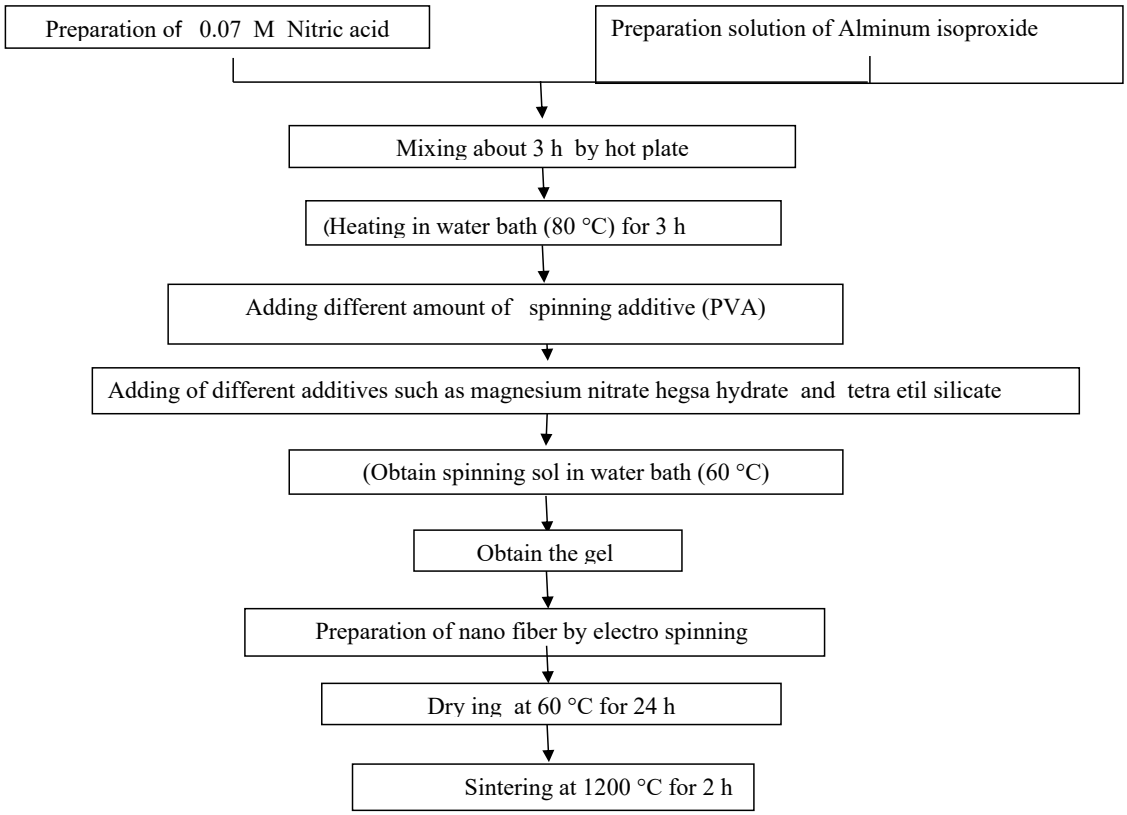

Fig. 1. Schematic view of production route for alumina fibers

alumina borate nano fibers increased. However, the crystallinity of the calcined nano fibers decreased with the increase of the PVP content [34]. Thamarai and Parag produced alumina fibers with a diameter of 5–15 μm by sol-gel process through centrifugal spinning. It was concluded that viscosity between 13 and 42 Pa.s with 3000 rpm was favored for obtaining fibers containing less shots [35].

This study has driven us to verify and prepare alumina nano fiber by sol–gel centrifugal spinning route and electrospining method. The novelties of this research are investigating

• The effects of different percentages of organic matter and spin-aid on spin ability of sol as well as on the morphology and the microstructure properties of fibers produced.

• The effects of adding different amounts of spinning additive (PVA) on the morphology and microstructure of the fibers are studied.

• The effect of magnesium nitrate hexahydrate ($MgN_2O_6 * 6H_2O$) and sodium ethoxylate ($SiC_8 H_2o O_4$) on phase transformation of alumina nano fibers by determining the weight change percentages of additives.

• The effect of different additives on required sintering temperature.

The target of this project is developing and evaluating of alumina nano fiber with the best properties for industrial uses.

EXPERIMENTAL PROCEDURE

Preparation of nano alumina fiber

Nano Alumina fibers were prepared by sol-gel and electro spinning method using Aliminum iso proxide (C_9 H_{21} ALO_{30} , Merck KGaA 8.01079.0250), NHO_3 (Merck KGaA 1.00456.1000) , Lactic acid (Merck KGaA 1.00366.1000), Hydroxylethyil cellulose (Merck KGaA 8.220.68.0100) and polyvinilalcohol (Merck KGaA 8.21038.01000) as starting materials. The alumina fibers were prepared in the processing steps as depicted in Fig. 1. The alumina sol was prepared by mixing H_2O, aluminum isoproxide and nitric acid, followed by heating in water bath (80 °C) for 3 h. A proper amount of water and spinning additive (PVP) were added into the alumina gel.

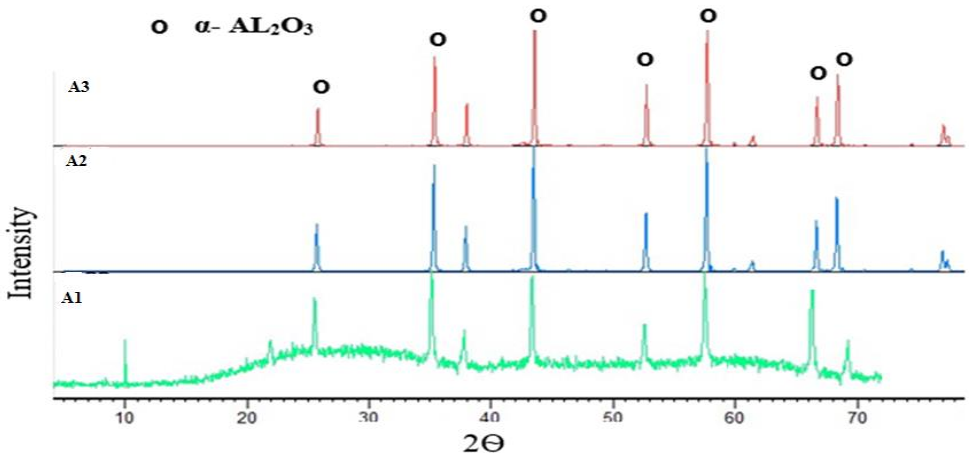

Fig. 2. X-ray diffraction pattern of α-AL_2O_3 synthesized after calcination at 1200 ° C for 2 h.

Then, the precursor solution was concentrated to obtain spinning sol in water bath (60 °C) for 3 h. The sol fibers were prepared by eletrospinning. Then, the sol fibers were dried at 60 °C for 24 h in an oven. The gel fibers were then sintered at 1200 °C for 2 h, with a heating rate of 2 °C/min. In the next step, different additives such as magnesium nitrate hegsahydrate ($MgN_2O_6 * 6H_2O$, Merck KGaA 1.05853.0100) and tetra etyle silicate ($Sic_8H_{20}O_4$, Merck KGaA 8.00658.0250) were added. After preparation of sol, about 2 Wt% of the additives was added to the alumina gels. Then, the precursor solution was concentrated to obtain spinning sol in water bath (60 °C) for 3 h. The sol fibers were prepared by eletrospinning and dried at 60 °C for 24 h in an oven. The gel fibers were then sintered at 1000 °C for 2 h, with a heating rate of 2 °C/ min. Phase identification was performed by X-ray diffraction (XRD) PW1800, of Philips Company, using nickel filtered Cu Ka radiation in the range of 2θ = 10°-60° with a scanning speed of 5° per minute. A Fourier transform infrared spectrometer (FTIR) by Perkin Elmer Spectrum 100 series was used with the universal attenuated total reflection (UATR) method. Microstructures of powders were identified by transmission electron microscope (Philips –Zeiss- Germany) and scanning electron microscopy (SEM PHENOM).

RESULTS AND DISCUSSIONS

XRD results for preparation of nanofiber

The X-ray diffraction patterns (XRD) of samples with 14 % PVA(A3), sample with 10 % PVA(A2) and sample without PVA(A1) after calcining at 1200 ° C for 2 h are shown in Fig. 2. The straight base line and sharp peaks of the diffractogram in Fig 2 confirmed that, the products were well crystallized. The XRD pattern in Fig 2 indicated that, the main phase in all samples are α-AL_2O_3 phase (corundum) which is consisted with the information contained in the standard cards (0173-10) and (10-44) and (1484-43). Consequently, in all samples, the α-AL_2O_3 phase (corundum) was formed. Hyuk-Joon and Kug (1999) observed that completion of the most stable phase, α-alumina occurred at 1200°C. Although the thermodynamically stable α-Al2O3 phase could be obtained through a reconstructive transformations (boehmite), the morphology remains unchanged and the final products have the same shape as in the initial phases (36,37).

The XRD pattern of the α-Al_2O_3 nano fiber powder containing 2% magnesium nitrate hexahydrate ($MgN_2O_6 * 6H_2O$) (B1) and 2% tetraethyl ortho-silicate ($SiC_8 H_{20} O_4$) (B2), after calcination at 1000 °C for 2 h are shown in Fig 3.

Comparing diffraction patterns of two samples, the formation of α-Al_2O_3 phase could be accounted as the main phase in the samples of B1 and B2, which is in agreement with standard cards of (10-04-04), (1484-43(. Magnesium nitrate hexahydrate ($MgN_2O_6 * 6H_2O$) (B1) and tetraethyl ortho-silicate ($SiC_8 H_{20} O_4$) (B2) additives have the potential synergistic property to reduce the transformation temperature. They can cause to reduce the transition alumina transformation temperature as well as the ability to modify the grain morphology. Its calcination temperature was about 1000 °C, which was 200 °C lower than the general calcination

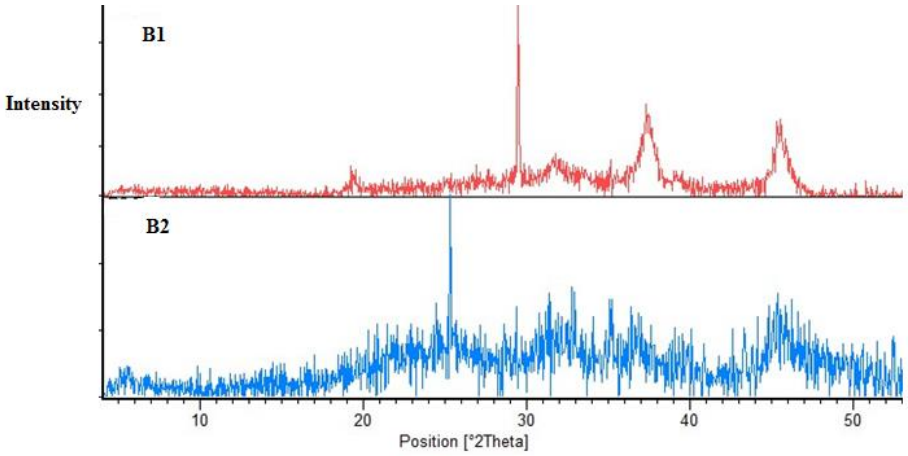

Fig. 3. X-ray diffraction pattern of B1 and B2 samples after calcination at 1000 °C temperature for 1 h

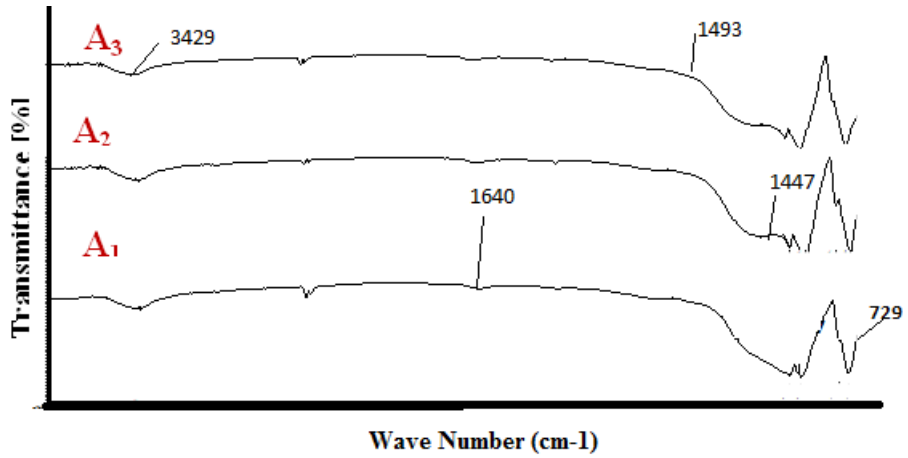

Fig. 4. FTIR curves powder samples of A1, A2, A3

temperature for preparing α-Al_2O_3 fibers and thus led to save the energy consumption. Ions of Mg^{2+} and Si^{4+} in solid solution could provide solid solution with Al_2O_3 .Integration of limited Mg^{2+} and Si^{4+} ions in solid solution might create interfacial energy difference. The interfacial energy difference improved the interface reaction of alumina and speeded up the grain growth in some directions [37].

FTIR results of the a-Al_2O_3nano fiber

Fig 4 shows the FTIR spectrum of samples A1, A2 and A3 after calcining at 1200 ° C for 2 h. The FTIR shows the existence of ((AL-O)) bonds at 1447 cm^{-1}, (α-AL_2O_3) bands at 1493 cm^{-1}, (ALO_4) bands at 1590 cm^{-1}, (AL-O-AL) bands at 1640 cm^{-1} and (ALO_6) band at 729 cm^{-1} in all samples [12,18].The peaks marked with (AL-OH) at wave number of 1400 cm^{-1} and (CO) and (C = C) at wave numbers of, 1450-1600 cm^{-1} respectively related to the additives. The peak marked with H_2O in 3429 cm^{-1} belonged to the stretching vibration as well as the deformation vibration characteristics of hydroxylate group (O-H) of water [20,26].

In addition, by comparing the graphs, it was observed that, when the fibers were formed the intensity of the peaks was decreased. Moreover, by comparing bands and the existence of narrower widths in the range of 400-750 cm^{-1}, A3 sample was selected as an optimal sample.

Structural changes at B1 and B2 samples were

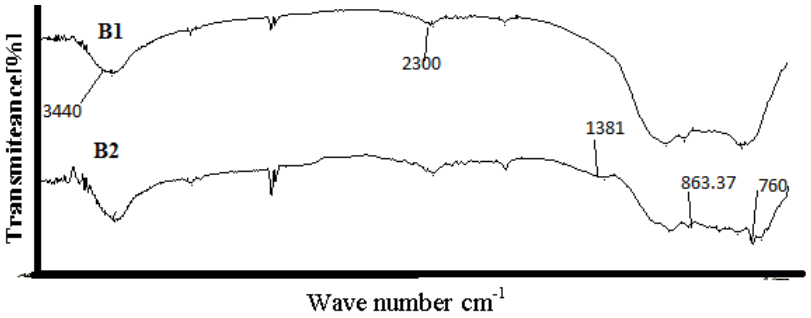

Fig. 5. FTIR curves powder samples B1, B2

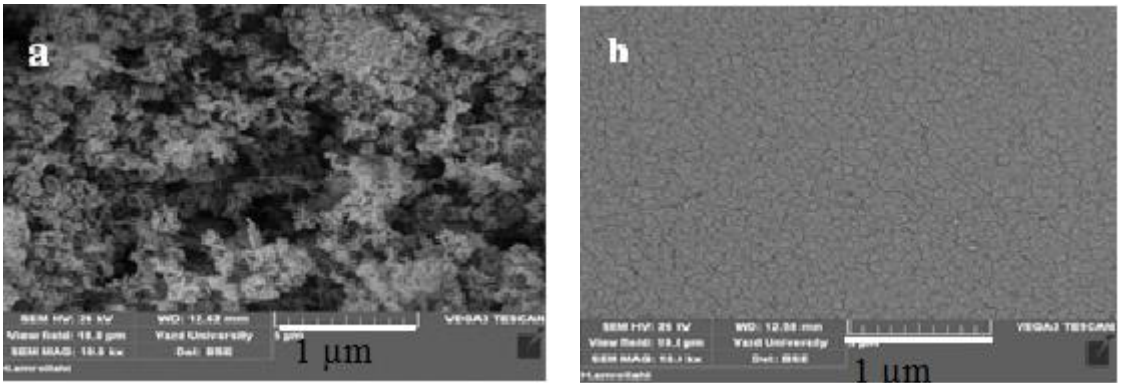

Fig. 6. SEM image of sample A1 (before calcined a), sample A1 (after calcined ,b)

investigated by FTIR, which could be observed in Fig 5. Peaks in the wave number of 150-500 cm^{-1}were related to Al-O band. The (α-AL_2O_3) bands at 560 cm^{-1} and ALO_4 band at 645 cm^{-1} were excited [2,18,27]. The peaks marked with (AL-O-AL) , ALO_6 and AL-OH were at 760 , 825 and 1381 cm^{-1} related to alumina groups , respectively. Peaks appeared in 1450-1627 cm^{-1} were related to the (C-O) and (C-C) bonds[26-28].There were the peaks at 2300cm^{-1} and 3448 cm^{-1} which were associated to (O-N) band and (O-H) bands separately. According to Fig 5, with increase of magnesium nitrate hexahydrate and tetraethyl ortho-silicate additives, the absorption band of α -Al_2O_3 appeared at 863·37 cm^{-1}.

Moreover, the significant absorption bands of α-Al_2O_3 at 583·15 and 517·19 cm^{-1} to 572 and 482 cm^{-1}, respectively. This observation showed that, the phase transformation of α-Al_2O_3 was promoted by the addition of additives. Also, with increasing weight percentage of additives, the intensity peak of Al-O decreased. Accordingly, the phase transformation of α-Al_2O_3 was promoted by the addition of additives.

Morphological properties of the a-Al_2O_3nano fiber

Fig 6 shows the scanning electron microscopy (SEM) images of nano alumina fiber before calcined and after calcined. As it could be seen, the fiber could not grow very well and a network of interlinked fibers form a continuous web-like structure. It might be due to insufficient amount of spinning aid (PVA), as with increasing the amount of PVA the fibers were grown and formed.

Fig 7 shows SEM of A2 (10% PVA) sample with good appearance and fiber shape without any defect and agglomeration. They had smooth surfaces, uniform diameters and were flexible with average diameter of fibers was about 65-80 nm.

Fig 8 shows SEM micrograph of sample A3 (14 % PVA). As it was shown, the growth of fiber was achieved and the shape of fiber was formed.

Thus, nano fiber with reasonable strength and small amount of dust formation was normally obtained. Average diameter of fibers was about 160-200nm with regular shape and some agglomeration. Regarding to Fig.6 and Fig. 7 by increasing the PVA percentages, the average diameter of fibers increased because the average diameter of gel

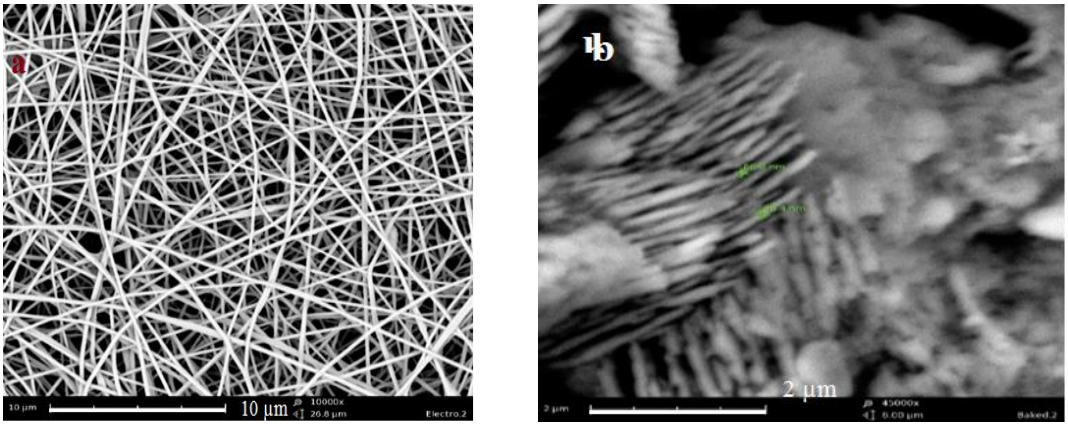

Fig. 7. SEM image of sample A2 (before calcined a), sample A2 (after calcined ,b)

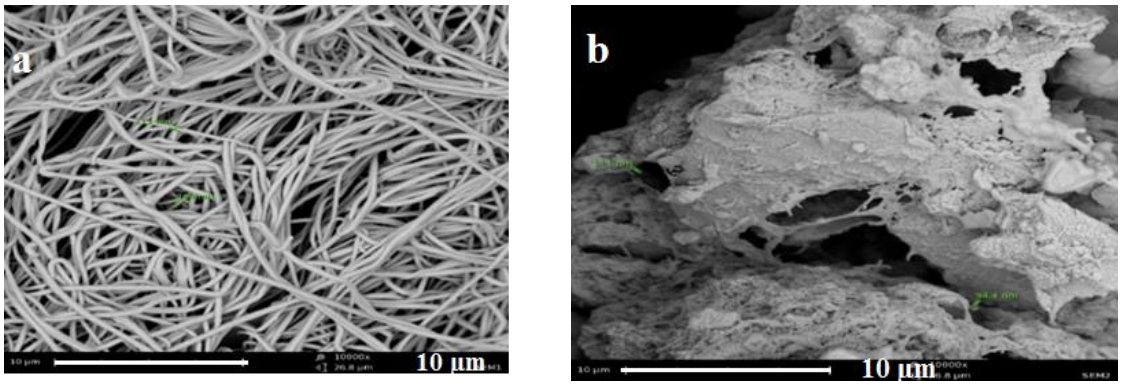

Fig. 8. SEM image of sample A3 (before calcined a), sample A3 (after calcined ,b)

fiber remarkably depends on the amount of PVA. This observation is consisted with the presented results by Xiaoling et al., who prepared magnesia-alumina spinel/yttrium aluminum garnet composite fibers by sol-gel method. They showed the diameter of fibers depends on the amount of polyvinylpyrrolidone (PVP) and with increasing of PVP amount the diameter of fibers increased [38].

Fig 9 shows SEM images of sample B1(containing magnesium hexahydrate nitrate (MgN2O6 * 6H2O)) and B2 (Containing tetraethyl octosilicate (SiC8 H_{20} O_4). As it could be seen the shape of both samples were entirely fibrous. Sample B1 had a certain amount of agglomeration with a diameter of about 90-100 nm, but sample B2 shows that, the fibers were more uniform without any agglomeration with a diameter about 40-50 nm.

As shown in Fig. 9, adding of different additives influenced stabilization of transition forms of alumina and retarded the conversion to a-alumina by incorporating dopant contains and vary the number of point defects (impurity ions) in the structure. Tetraethyl octosilicate had better effect on decreasing the diameters of alumina nano fiber. The silica in tetraethyl octosilicate affect the size asa grain growth inhibitor instead of varying fiber alumina diameter . Ion of Si^{4+} formed more liquid and enhanced the directional growth of the alumina nanostructures and hence increased the transition forms of alumina to α-alumina.

Transmission electron microscopy (TEM) images of samples A1and B2 were used for investigating the particle size and morphology of nano alumina fibers. As Fig 10 shows, in sample A1 the fibers did not grow very well and there was a lot of agglomeration. However, sample B2 had completely fiber form without any agglomeration with average diameter about 40 nm which is consisted with SEM results.

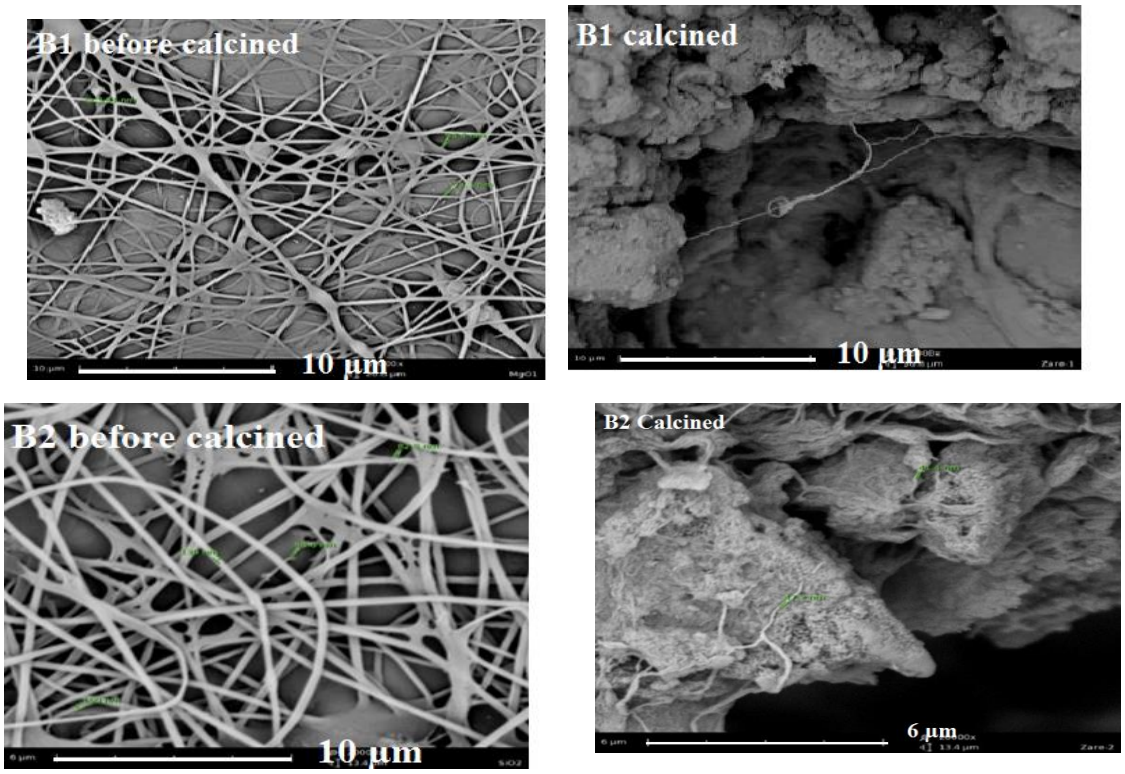

Fig. 9. SEM image of sample B1 (a: before calcined), (b:after calcined), sample B2 (a: before calcined), (b:after calcined)

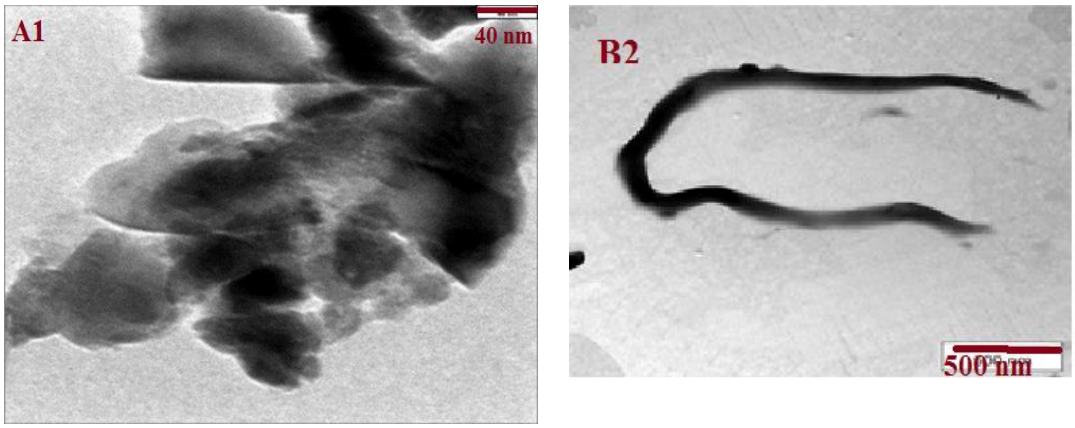

Fig. 10. TEM images of sample A1 and sample B2

In summary, liquid-forming additives enhance the γ -to-α transformation of alumina. Additive effects on sintering and shrinkage were almost similar except for the added effect of reaction sintering and faster transformation kinetics due to the better contact between powders in a compact [37]. Moreover, the average diameter of gel fiber remarkably depended on the PVA percentages and with increasing of PVA addition amount the average diameter was increased.

CONCLUSION

1- Nano α-Al_2O_3 nano fiber powder with using different additives was synthesized through sol-gel

and electro spinning method at 1200 °C.

2- Using a combination of sol-gel and electro spinning techniques, different amounts of PVA (1, 10,14 %wt) were tested. The best result was obtained when 10% of PVA was used as spinning aid.

3- The formation of α-Al_2O_3nano fibers were achieved with 2% additives of magnesium hexahydrate nitrate and tetraethyl octosilicate .

4- The results of XRD showed that with the increasing amount of magnesium nitrate hexahydrate (MgN_2O_6 * $6H_2O$) and tetraethyl erythrocylate (SiC_8 H_{20} O_4), the sintering temperature reduced from 1200°C to 1000°C and α-Al_2O_3 was formed which caused to decrease the energy consumption .

5- By using 10% PVA as spinning aid and 2% tetraethyl octosilicate as an additive , the diameter of nano fibers became about 41nm with regular shape and good dispersion.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

REFERENCES

1.Chandradass M, Balasubra M. Effect of magnesium oxide on sol-gel spun alumina-zirconia fibres. Journal of the European. 2006; 26: 2611-2617.

2.Bahgat R . New Electrospun Polystyrene/Al2O3 nanocomposite superhydrophobic Coatings; Synthesis. Characterization, and Application. Coatings. 2018; 8: 65-83.

3. Rastetter C, Symes W . Alumina Fiber-a polycrystalline refractory fibre for use up to 1600. Inter Ceram . 1982; 31:3-6.

4..José H ,Nahúm A ,Simón Y . Fabrication of α-alumina fibers by sol-gel and electrospinning of aluminum nitrate precursor solutions. Results in Physics.2019; 12:193-204.

5. Makki T, Sakaa S . Formation of alumina fibers by unidirectional freezing of gel. J.Non cryst. Solids.1986; 82: 239-245.

6.Xuan Z, Bin D , Tong S , Wei L, Chang D. Preparation of alumina nanorods from chromium-containing alumina sludge. Nanoscale Research Letters.2017;12:392-403.

7.Chatteijes M K , Naskar C, D.Ganguli. Sol – gel alumina Fiber mats for high- temperature application . Mater. Lett. 2002; 57:87-93.

8.Chandradass J, Balasubramanin M: Sol-gel processing of alumina fibers. Journal of Materials processing Technology.2006; 173: 275-280.

9.Andrei S , Anthony B ,Pavel P ,Tomáš K ,David L. Nanofibrous alumina structures fabricated using high-yield alternating current electrospinning. Ceramics International.2016; 15: 17154-17161.

10.Syed Danish A ، Chong W ، Qinjun S ، Jing G ، Shengdong Z ، Haifeng Y ، Jian C. Polymer-free electrospun separator film comprising silica nanofibers and alumina nanoparticles for Li-ion full cell, Journal of Energy Chemistry.2020;42: 217-226.

11.Corni I, Mary P, Aldo R. Electrophoretic deposition: From traditional ceramics to nanotechnology. Journal of the European Ceramic Society.2008; 28 :1353-1367.

12.Dan L, Younan X. Fabrication of titania nanofibers by electrospinning. Nano Letters. 2003;**3**: 555-560.

13. Jingyi S ,Zehua L ,Yinan W ,Bingru Z ,Fengting L . Dendrimer-based preparation of mesoporous alumina nanofibers by electrospinning and their application in dye adsorption. Chemical Engineering Journal.2015; 264: 48-55.

14.Kim J , Chio Y C. Direct observation of alumina nanowire formation from porous anodic alumina membrane via the droplet etching.Nanotechnology.2005;**17**: 2-12.

15. Alveera K, Mohammad Ayaz A, Shirish J, Vyacheslav L. Synthesis of alumina fiber by annealing method using coir fiber , American Chemical Science Journal.2016; 15(2): 1-7.

16.Thomasj W. Increased os tedolast Functions On theta+delta nano fiber alumina. Biomaterial. 2005; 26: 953-960.

17.Tepper F, Lerner M , Ginley D. Nanosized alumina fibers. AM ceram soc Bull.2001;151-158.

18. Momohjimoh I , Mohamed A. Hussein Al . Recent Advances in the Processing and Properties of Alumina–CNT/SiC Nanocomposites.Nanomaterials .2019; 9, 86-96.

19.Naskar M K , Chatterjee M , Dey A , Basu K. Tissue Effects of processing parameters on the fabrication of near-net-shape fiber reinforced oxide ceramic matrix composites via sol-gel route;Ceramic International.2004; 30:257-265.

20.Naskar M K , Ganguli D.Rare- earth doped Alumina zirconia fibers by sol-gel processing. J.Mater. sci.2001;21: 355-361.

21.Panda P K, Ramakrishna S. Electro spinning of alumina nan fibers using different precursors. J Mater Sci. 2007; 42:2189-2193.

22.Saras V, Rama G.V. Structural evolution in alumina gel. J Mater Sci. 1987; 22:28529-2534.

23.Scherer G , lung J. Glasses From colloids. J.Non Cryst. Solids.1984; 63:163-172.

24.S.sakka. Sol-gel glasses and their application. Trans. Ind ceram.Soc.1987;46,1-11.

25.Thomasj W. Increased osteoblast functions on θ+δ nanofiber alumina. Biomaterial.2005; 25: 953-960.

26.LeiLi W, Kang X, Zhuang J, Shi Y, Bowen C. A comparative study of alumina fibers prepared by electro-blown spinning (EBS) and solution blowing spinning (SBS).
Materials Letters .2015; 160: 533-536.

27.Venkatesh R, Ramanan S R. Influence of processing variables on the microstructure of sol-gel spun alumina fibers. Materials Letters.2002; 55:189-195.

28.Venkatesh R, Ramanan S R. Effect of organic additives on the properties of sol-gel spun alumina fibers. Journal of the European ceramic society. 2000; 20, 2543-2549.

29.Yogo T , Wahara H .Synthesis of a- alumina fiber from

modified aluminum alkoxide precursor.J.mater. Sci;1991:5292- 5296.
30.Wang Y ,Wei L , Xiuling J. Electro spinning preparation and adsorption properties of mesoporous alumina fibers. Journal of Materials Chemistry A.2013; **1**:10720-10726.
31. Shafqat A , Yasir A , Zareen Z .Synthesis of γ-alumina (Al_2O_3) nanoparticles and their potential for use as an adsorbent in the removal of methylene blue dye from industrial wastewater. Nano ascale advance.2009;1: 213-219.
32. EmilieP ,Leif N ,Torbjörn G , Patrice S. Direct electro deposition of aluminium nano-rods. Electrochemistry communications. 2008; 10: 1467-1470.
33.Xiangdong L , Xiaohui S, Hongrui X, Lifu C, Siwei L,Ming T. 2020, Continuous α-Al_2O_3 fibers grown by seeding with in-situ suspension. Ceramics International.2020;1-12.
34. Xiaolei S, Wen L, Juan W, Shuheng X, Bing L, Yunzhu M. Microstructural differences between electrospun alumina borate nanofibers prepared by solutions with different PVP contents.ceramic international. 2017; 43: 9831-9837.
35.Thamarai N , Parag B**.** Influence of spinning parameters on synthesis of alumina fibers by centrifugal spinning. Ceramics International.2018;44:11644-11649.
36. Hyuk-Joon Y, Wook J, Tae K , Kug S. Low-Temperature Formation of α-Alumina by Doping of an Alumina-Sol. Journal of Colloid and Interface Science.1999;21:110-113
37. Mirjalili F.Effect of fluoride additives on production and characterization of
nano-α-Al_2O_3 particles. Bull. Mater. Sci.2014; 37(7)1709–1716.
38. Xiaoling M, Zhenlin L,Hongbin T, Jiano N, Caixia W, Preparation and grian-growth of magnesia-alminum spinel/yttrium aluminum grant composite fibers. Ceramics-Silikáty .2018;62 (3):279-284.