

## Formation of Silica Nano-Fibers and Innovation During the Calcining Process

N. Sepehrian, M. Abbasi and M. Noori  
Department of Technical, University of Guilan, 3756 Rasht, Guilan, Iran

**Abstract:** In this study, Electrospinning and producing composite nanofibers of Polyvinyl Alcohol (PVA) Silica nano-Fibers ( $\text{SiO}_2$ ) were studied. The effect of concentrations of silica nanoparticles, concentration of PVA auxiliary polymer, calcining process temperature and duration of this process on the formation of composite silica nanofibers was examined. The morphology and structure of nanofibers produced were investigated by using light microscopy, Scanning Electron Microscope (SEM), X-Ray Diffraction (XRD) and Fourier Transform Infrared Spectroscopy (FTIR). Composite nanofibers with 6, 8, 10 and 12 weight percent of PVA auxiliary polymer along with silica nanoparticles of 50, 70, 90 and 100 weight percent of the auxiliary polymer were produced. Given that the PVA destroys at temperatures  $>494^\circ\text{C}$ , nanofibers were calcined at three temperatures of 500, 700 and  $900^\circ\text{C}$  for 3 h. The results showed that the composite nanofibers with initial concentration of 10 weight percent of the PVA auxiliary polymer and Silica nanoparticles of 90 weight percent of the auxiliary and calcined polymer at  $700^\circ\text{C}$  produce the best silica nanofibers. To evaluate the effect of calcining process duration, nanofibers were placed in the furnace for 7 h. Increasing the process duration reduced the crystallinity of nanofibers and nanoparticles loss.

**Key words:** Silica nano-fibers, calcining process, silica nanoparticles, calcining process

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### INTRODUCTION

Electrospinning is an easy and versatile technology to produce polymer fiber by polymer solutions and melted polymer. In this system, there is a high-voltage power supply which makes the polymer solution collide the front stacker page with high speed and the solvent evaporated during this process (Obradovic *et al.*, 2011). Silica nanofibers have interesting applications including their use in sensors, catalysts and dental composites. The sensors are capable of measuring different quantities such as rotation, pressure, electric field, temperature, pressure, sound, vibration, linear and angular position, strain, acceleration, humidity, adhesion and identifying chemicals (Finch, 1991; Doshi and Reneker, 1995). Shao *et al.* (2002) first produced silica nanofibers from electrospinning polyvinyl alcohol (PVA)/silica ( $\text{SiO}_2$ ) in 2002. The results showed that the calcining temperature has a big effect on the nanofiber morphology. The optimal temperature was  $550^\circ\text{C}$  to produce nanofibers. The solution was prepared from Tetraethyl Ortho Silicate (TEOS), PVA and Phosphoric Acid ( $\text{H}_3\text{PO}_4$ ) at a ratio of TEOS:  $\text{H}_3\text{PO}_4$ :  $\text{H}_2\text{O}$  = 1:0.01:11. This solution was given time for 5 h and then PVA with 10 wt% was slowly added to the solution and placed for 12 h at  $60^\circ\text{C}$  to reach the

appropriate viscosity. Produced nanofibers at 400, 550 and  $800^\circ\text{C}$  temperatures were calcined for 10 h. Nanofibers produced after calcining process have a rough surface and this phenomenon increases as the temperature goes up and the diameter of the nanofibers reduces (Choi *et al.* 2003; Sung *et al.* (2003) produced silica nanofibers from an electrospun solution which includes TEOS, distilled water, ethanol and Hydrochloric acid (HCL). Gel composition was mixed together in the ratio of 1:2:2:0.01 and nanofibers were produced at a voltage of 10 and 12 kV. The nanofiber's thickness decreased with increasing voltage. In this study, acid decomposition method was used for the production of nanofibers. Therefore in order to produce silica nanofibers, no calcining process was needed (Choi *et al.* 2003; Kameoka *et al.* (2004) prepared silica nanofibers through electrospinning and calcining process. Electrospinning system has two components: a silicon grooved source to deposit nanofibers and a rotating collector plate. Polymer solution was prepared by dissolving 5 wt% of the Polyvinyl Pyrrolidone (PVP) Polymer in the glass solution. Composite nanofibers produced deposit on grooved substrate attached to the rotating plate. The composite nanofibers were placed in the furnace at  $850^\circ\text{C}$  for calcining process. Crosslinking is created among.

SiO<sub>2</sub> molecules by PVP vaporization. Composite nanofiber's diameter was 107 nm and converted into 85 nm after calcination process (Kameoka *et al.*, 2004). Korean researchers produced silica nanofibers from polymer mixture of PVP/P123 in 2008. In this study, a three-block copolymer of Pluronic P123, TEOS, PVP, hydrochloric acid and ethanol was used. The PVP/ SiO<sub>2</sub> nanofibers produced for calcining process and for the production of calcined silica nanofibers were placed in the furnace at 550°C for 2 h. (Cancer *et al.*, 2008; Shah *et al.* (2012) produced silica nanofibers by the two dimensional and three-dimensional colloidal solution. Colloidal solution was made of industrial silica colloidal solution 30% (SiO<sub>2</sub>) 30% and polymer solution 5% wt of Polyethylene Oxide (PEO). Then, electrospinning and the production of composite nanofibers PEO/SiO<sub>2</sub> with a ratio of 80:20 from SiO<sub>2</sub> and PEO solution were performed. Pyrolysis process was carried out for fibers produced at 700, 800, 900, 1000 and 1100°C for 2 h and silica nanofibers were produced. The optimal temperature of 800°C was selected and the structure was falling apart from 900°C due to melting adjacent nanofibers. The nanofiber produced by a ratio of 50:50 of PEO and solutions had a three-dimensional structure similar to the structure of the wool (Shah *et al.*, 2012; Praeger *et al.* (2012) produced glass nanofibers through direct electrospinning from the molten solution. In this production method, an polymeric material with a high melting point and surface tension was of essence; therefore, bromine oxide (B<sub>2</sub>O<sub>3</sub>) was selected as the molten solution for electrospinning. Because the melting temperature of B<sub>2</sub>O<sub>3</sub> is 450°C, electrospinning was performed to control the viscosity and surface tension at 850°C and B<sub>2</sub>O<sub>3</sub> Nanofibres with a diameter of 100 nm were produced (Praeger *et al.*, 2012).

This study aims to investigate the feasibility of producing silica nanofibers by electrospinning with the use of silica nanoparticles and polyvinyl alcohol as an auxiliary polymer. The difference between this method and the earlier ones is the direct use of silica nanoparticles as well as the investigation of the effects of nanoparticles concentration, auxiliary polymer concentration, calcining process temperature and duration of this process on the formation of silica nanofibers.

## MATERIALS AND METHODS

**Raw material:** Silica nanoparticles with particle size of 10-15 nm were purchased from sigma-Aldrich Corporation and polyvinyl alcohol with a molecular weight of 72,000 was purchased from merck company.

**Instruments:** The production of nanofibers was done by an electrospinning device consisting of gamma high voltage research (USA) and pump systems New Era (USA).

To study the physical structure of the fiber, Nikon Microphot-FXA optical microscope made in Japan was used 2100AIS? Korean Seron Technologies (SEM) Scanning Electron Microscopy and the mini sputter coater 7620SC? Quorum Technologies gold coating smade in England were also used the to examine surface morphology. In order to identify the structure of the nanoparticles in nanofibers, Fourier Transform Infrared (FTIR) Nicolet Magna 560 made in America which can scan in the range of (400-4000) cm<sup>-1</sup> was used. Nabertherm furnaces were used for calcining the nanofibers and 3000 Equinox X-ray diffraction made by France INEL company was used to measure the amount of nanofiber's crystallinity.

**Methods: preparing the PVA/nano SiO<sub>2</sub> solution:** After choosing the Polyvinyl Alcohol (PVA) as an auxiliary polymer, the PVA solution with 6, 10, 8 and 12 wt% was prepared by dissolving the polymer powder in hot distilled water. Choosing the consuming percentages of silica nanoparticles was done with the assumption that, after the removal of the auxiliary polymer, nanoparticles distance should be enough to come together and establish a connection. This nanoparticle was used in 50, 70, 90 and 100 wt% of polyvinyl alcohol. Each of the solutions was placed on the stirrer for 24 h at medium speed until a uniform and homogenized solution was obtained.

**Electrospinning process:** Three important electrospinning parameters including feeding rate, voltage and the distance from the tip of the needle to collector plate were examined. As a result, feeding rate of 0.01 m<sup>2</sup>h<sup>-1</sup>, the distance of 10 cm and electrospinning voltage of 7kv were chosen. Nanofiber web was collected on aluminum foil.

**Calcining process:** For calcining process, the web produced was removed from the foil and placed within a specified time in the oven under 500, 700 and 900°C. Since the evaporation temperature of polyvinyl alcohol is <494°C (Wannapopa *et al.*, 2012), Polymer is completely removed at these temperatures and causes the nanoparticles to remain in the fibrous direction and crosslinking is established among silica nanoparticles during the calcining process (Kameoka *et al.*, 2004). As a result, silica nanoparticles are produced.

## RESULTS AND DISCUSSION

**Effect of concentration of PVA/nano SiO<sub>2</sub> on the formation of electrospun nanofibers:** Electrospinning of

nanofibers was first initiated with 6% of polyvinyl alcohol along with different percentages of silica nanoparticles under electrospinning conditions mentioned. Results showed that in this concentration (6%) of the auxiliary polymer, the viscosity of the fluid is to the extent that it can produce sustained flow for sufficient time. As a result, the particles produced are a lot in nanofibers and some solution was thrown forward as drops. That's why the work was not done with this percentage of the auxiliary polymer. Then, 8 wt% of the auxiliary polymer was used along with different percentages of nanoparticles. Because of the increased concentration, stability of the solution further increases and inter-chain interaction also increases. Finally, the solution viscosity is to the extent that it can move like a stable jet toward the plate and drop splashes on the collector plate was less than before (auxiliary polymer at a concentration of 6 wt%). Hence, it was selected as the initial concentration. A sample of a composite nanofiber containing 6 and 8 wt% of the auxiliary polymer provided by the optical microscope is shown in Fig. 1. As it can be seen in Fig. 1 a, there are many particles in the fibers; however, the number of particles is less and more continuous nanofibers have been created in Fig.1 b.

#### **Examining the effect of nanoparticle concentration and calcining temperature on the morphology of nanofibers**

##### **Examining the effect of nanoparticle concentration:**

When the polyvinyl alcohol at a concentration of 8% was selected as the initial concentration, nanoparticles with 50, 70, 90 and 100 wt% of the auxiliary polymer were added to this solution. Figure 2a shows composite nanofiber's image produced by 8 wt% of polyvinyl alcohol and nanoparticles of 50 wt% of the auxiliary polymer. The average diameter of the nanofibers was 228 nm. Figure 2b shows the average diameter is 237 nm.

It is observed that their diameter distribution compared to the sample with 8 wt% of polyvinyl alcohol and nanoparticles of 50 wt% of the auxiliary polymer had increased due to the increased concentrations of nanoparticles. Increasing concentration of nanoparticles in solution gradually led to concentration of nanoparticles and creation of particles in nanofibers. This causes non-uniformity of the nanofiber diameter.

As Fig. 2c shows the diameter mean of the samples with 8 wt% of polyvinyl alcohol and nanoparticles of 90 wt% of the auxiliary polymer is 229 nm.

Then, a solution with 8 wt% of polyvinyl alcohol and nanoparticles of 100 wt% of the auxiliary polymer was used to produce nanofibers with the same conditions mentioned. As it is shown in Fig. 2D increasing concentrations

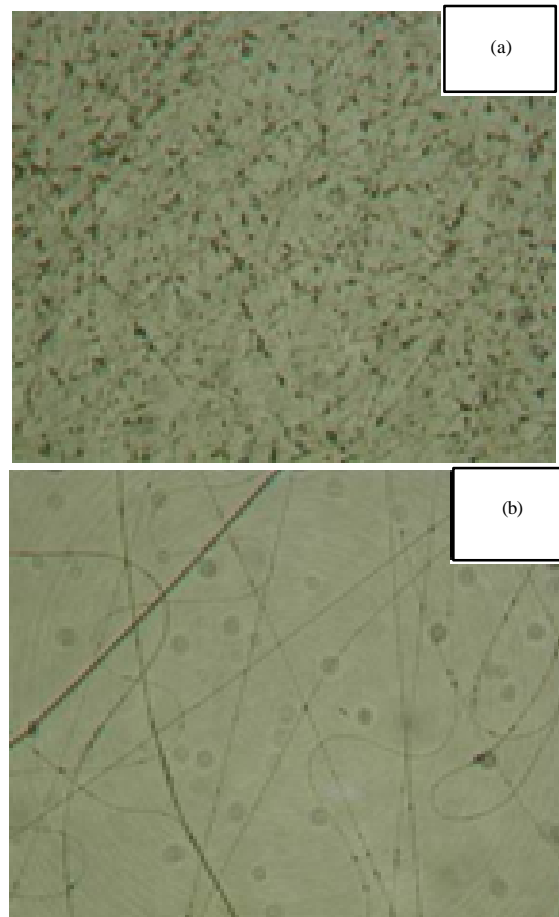


Fig. 1: Optical microscopy Image of nano-composite fiber; a) 6 wt% PVA; b) 8 wt% PVA

of nanoparticles leads to an increased number of larger particles and appearance of nanoparticles on the surface of the fibers. This factor was effective in the electrospinning process as thrown drops and non continuous liquid flow during the process. Moreover, the produced web compared to previous samples was more brittle and the same percentage of nanoparticles in later stages was discarded. The mean diameter is 231 nm.

**Investigating the effect of calcined temperature:** Of the samples produced, composite Nanofibers with the highest and lowest concentrations of nanoparticles (nanoparticle equal to 50 and 90 wt% of the auxiliary polymer) were selected for calcining process in the furnace under 900°C for 3 h. Auxiliary polymer role in this process 0 in addition to participation in electrospinning process is keeping with nanoparticles up to high temperatures and prior to calcining temperature and keep track of

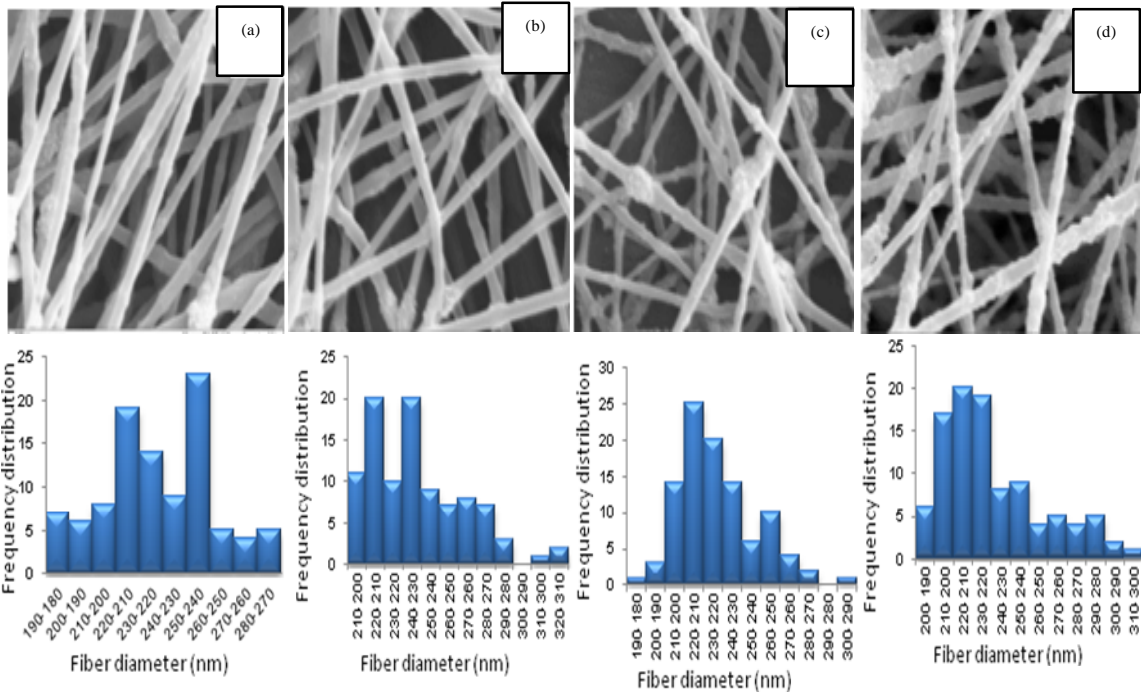


Fig. 2: SEM image of nano-composite fiber with 8 wt% PVA and nanoparticles: a) 50; b) 70; C) 90; d) 100 wt% of PVA and diameter distribution graph

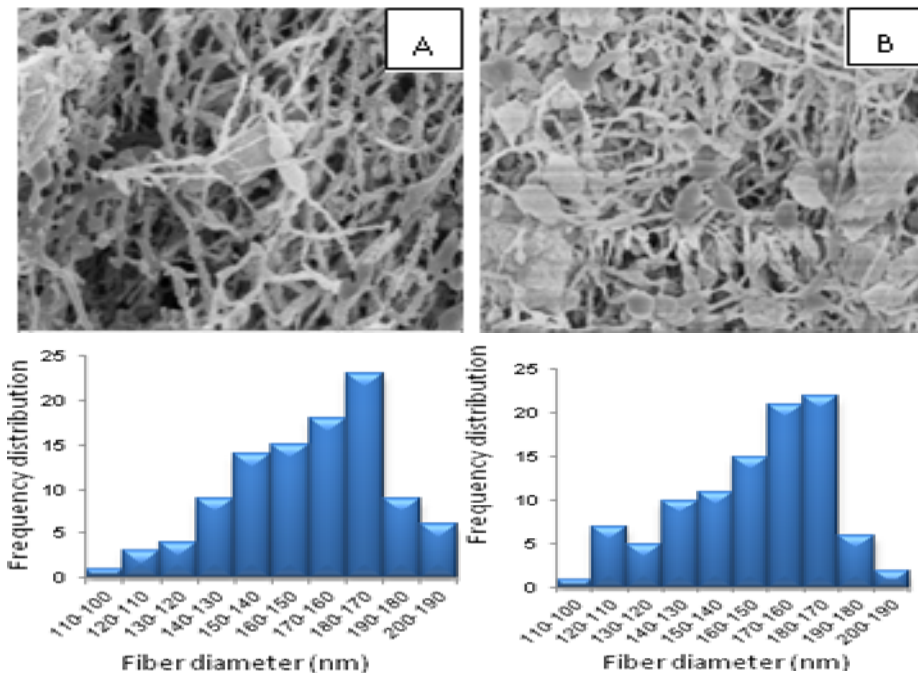


Fig. 3: SEM image of nanofibers silica with 8 wt% PVA after calcining process in 9000c and nanoparticles: a) 50; b) 90 wt% of PVA and diameter distribution graph

fiber-shaped directions of nanoparticles. Figure 3a shows the SEM image of the sample calcined at 900°C for 3 h

along with a diameter distribution graph with initial concentration of 8 wt% of polyvinyl alcohol and

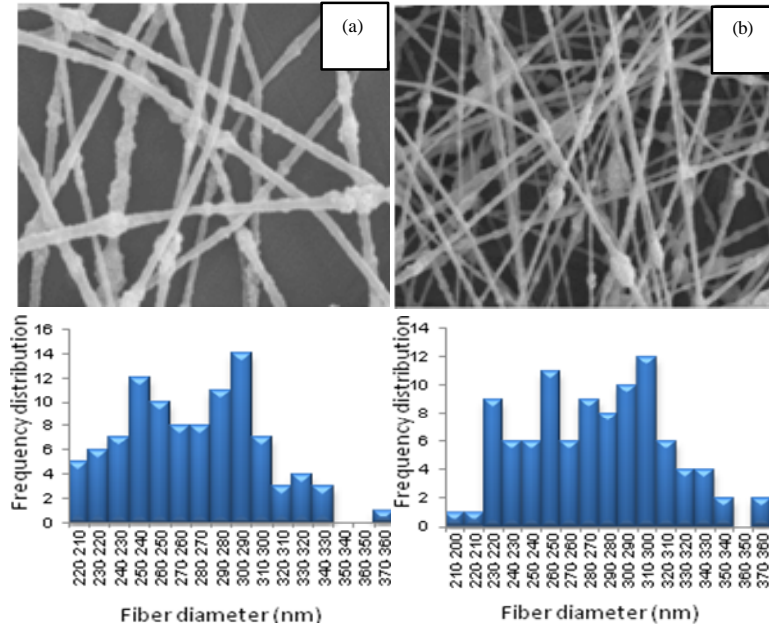


Fig.4: SEM image of nanofibers Silica with: a) 10; b) 12 wt% PVA and nanoparticles with 90 wt% of PVA and diameter distribution graph

nanoparticles with 50% of the auxiliary polymer. Web produced after this process was fragile and light with no flexibility and strength. The mean diameter was 159 nm.

As it is seen, nanoparticles are stuck together and lumped because of approaching the melting point. The diameter of the nanofibers after calcining process compared to earlier stages has reduced due to auxiliary polymer removal. Reduction in the sample is 30.26 compared to alcohol and nanoparticles with 90 wt% of the auxiliary polymer. Web produced after this process was also fragile and light with no flexibility and strength. The mean diameter was 155 nm. There was also 32.31% diameter decrease compared to the mean diameter of non-calcined sample.

On the other hand in the calcined samples with the initial nanoparticle concentration as much as 50 wt% of the auxiliary polymer with samples containing nanoparticles at a concentration of 90 wt% of the consumed polymer is used. It seems that containing nanoparticles at a concentration of 90 wt% of the consumed polymer (Fig. 3B), created nanofibers are in greater numbers and with greater coherence. It is due to the greater closeness of nanoparticles in Nanofibers because of the greater concentration of nanoparticles. The results obtained providing the stationary conditions of the auxiliary polymer concentration after calcining process suggest that with increasing concentrations of nanoparticles, nanofibers are created better with higher

frequency and greater coherence. The problem caused by increasing concentrations of nanoparticles is increased willow and disorganization of nanofiber structure during electrospinning process. Therefore, the project continued by using nanoparticles at a concentration of 90 wt% of the consumed polymer.

**Effect of (PVA) auxiliary polymer concentration and calcined temperature on the morphology of nanofibers**

**Investigating the effect of auxiliary polymer concentration:** Figure. 4a represents the SEM image of the composite nanofibers with the concentration of 12 wt% of polyvinyl alcohol and nanoparticles of 90 wt% of the auxiliary polymer as well as the diameter distribution graph. In this concentration, diameter mean was 268 nm. As it is observed, like earlier, the particles are created in nanofibers. The diameter average in this sample compared to the composite nanofibers with 8wt% of the auxiliary polymer has increased. Moreover, fragmentation and heterogeneity have also increased in nanofiber's diameters. Then, composite nanofibers with the concentration of 12 wt% of polyvinyl alcohol and nanoparticles of 90 wt% of the auxiliary polymer were prepared and its SEM image and diameter distribution graph is shown in Fig. 4B. The diameter is mean of 279 nm. Heterogeneity also increased in nanofiber's diameters. As it can be seen increasing the concentrations of auxiliary polymer has also increased the nanofiber's diameters. And diameter mean of 279 nm. Heterogeneity also

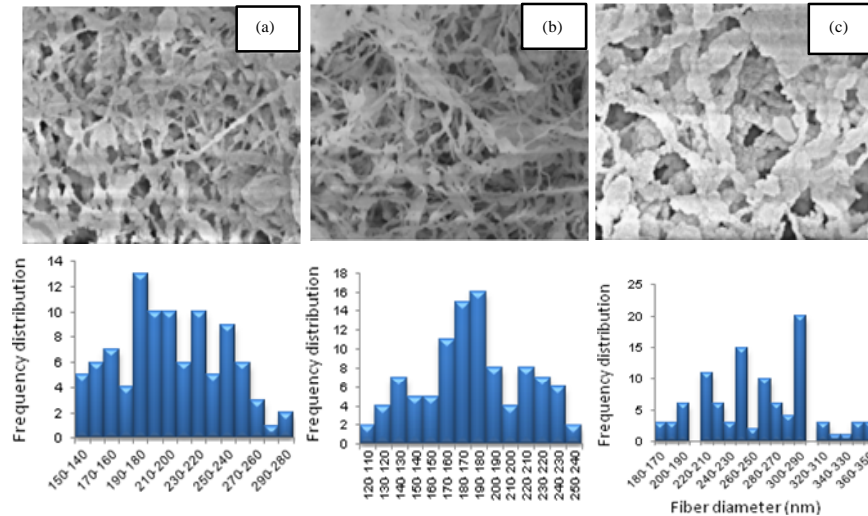


Fig. 5: SEM image of nanofibers Silica after calcining process in 500°C and nanoparticles with 90wt% of PVA and: a) 8; b)10, C)12 wt% PVA and diameter distribution graph

increased in nanofiber’s diameters. As it can be seen increasing the concentrations of auxiliary polymer has also increased the nanofiber’s diameters.

**Effect of calcining temperature:** After the production of composite Nanofibers at different concentrations, calcining operation was performed on nanofibers due to evaporation temperature of the auxiliary polymer which is  $>494^{\circ}\text{C}$  (Praeger *et al.*, 2012). Generated web were removed from the aluminum foil and placed in a furnace for 3 h at three different temperatures of 500, 700 and  $900^{\circ}\text{C}$ .

Figure 5a represents the SEM image of the silica Nanofibers after calcining the composite Nanofibers with the concentration of 8 wt% of polyvinyl alcohol and nanoparticles of 90 wt% of the auxiliary polymer as well as the diameter distribution graph. The diameter mean was 207 nm. There was a diameter decrease (9.6%) compared to pre-clacining process. Figure 5b represents the SEM image and the diameter distribution graph of the composite nanofibers with the concentration of 10 wt% of polyvinyl alcohol and nanoparticles of 90 wt% of the auxiliary polymer. The diameter mean was 182 nm. There was a diameter decrease (32%) compared to the initial diameter mean. Silica nanofibers were prepared by calcining the composite nanofibers with the concentration of 12 wt% of polyvinyl alcohol and nanoparticles of 90 wt% of the auxiliary polymer and its SEM image and diameter distribution graph is shown in Fig. 5c. As it is shown, nanoparticles are losing after the calcining process and this causes a great heterogeneity in nanofibers diameters. As a result, the diameter mean of 257 nm was created. There was a diameter decrease (7.88%) compared to the initial diameter mean.

**Calcination process at  $700^{\circ}\text{C}$ :** In Fig. 6A, the SEM image of silica nanofibers on the composite nanofibers with the concentration of 8 wt% of polyvinyl alcohol and nanoparticles of 90 wt% of the auxiliary polymer at  $700^{\circ}\text{C}$  as well as the diameter distribution graph is shown. As it can be observed, nanofibers are not well created. Measuring the diameter of nanofibers in some areas showed that the diameter distribution is placed between 100-300 nm with the highest frequency in 180-190 nm and the diameter mean of 211 nm. There was a diameter decrease (7.8%) compared to pre-clacining process. Figure 6b shows the SEM image and the diameter distribution graph of silica nanofibers obtaining from the calcination process at  $700^{\circ}\text{C}$  and from the composite nanofibers with the concentration of 10 wt% of polyvinyl alcohol and nanoparticles of 90 wt% of the auxiliary polymer. It is shown that nanoparticles could maintain their fibrous form without loss during the calcining process and form nanofibers. However in spite of the nanofibers formation, the web produced after these processes were also fragile and light with no flexibility and strength. the diameter mean was 156 nm. There was a diameter decrease (41.79%) compared to pre-clacining process. In Fig. 6c shows the image of silica nanofibers produced from calcination process on the composite nanofibers with the concentration of 12 wt% of polyvinyl alcohol and nanoparticles of 90 wt% of the auxiliary polymer at  $700^{\circ}\text{C}$  as well as the diameter distribution graph. At this stage, nanofibers have no appropriate form due to aggregation and attachment of nanoparticles. Measuring the diameter of nanofibers in some areas showed that the diameter distribution is placed between 130-320 nm with the highest frequency in 210-220 nm and the diameter mean of 231 nm.

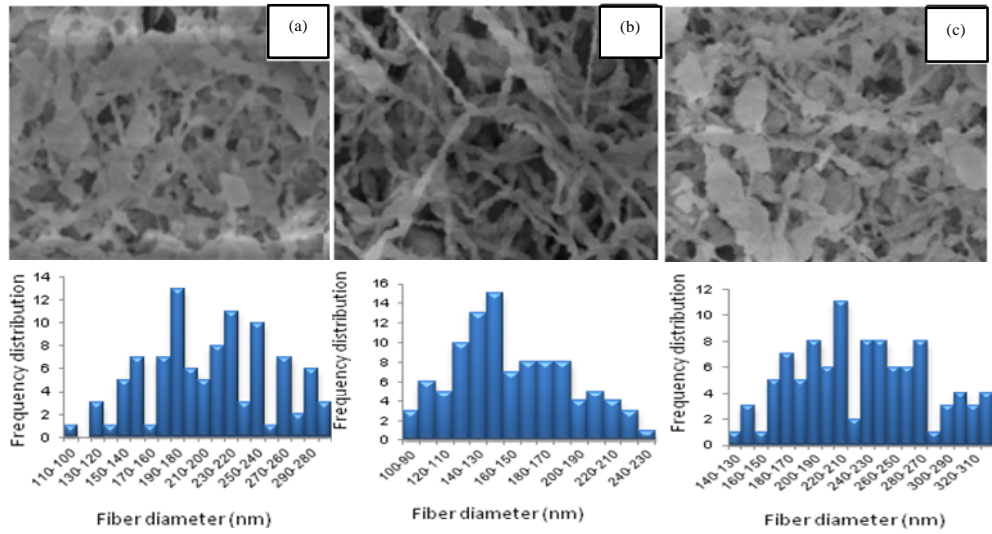


Fig. 6: SEM image of nanofibers Silica after calcining process in 7000c and nanoparticles with 90wt% of PVA and: a) 8; b)10 c)12 wt% PVA and diameter distribution graph

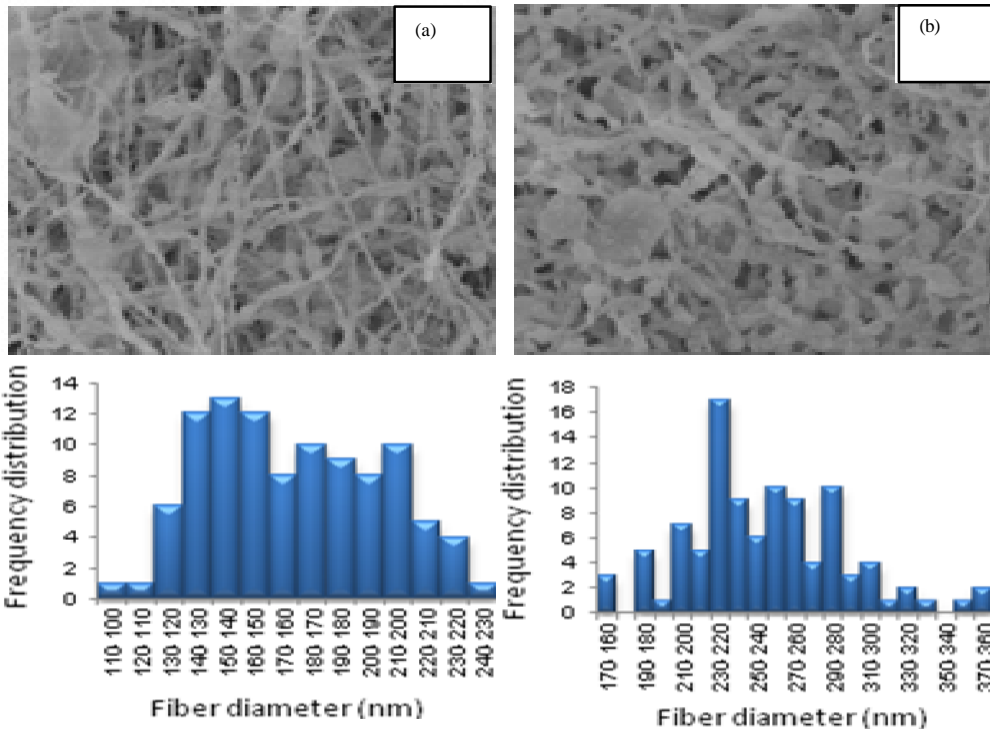


Fig. 7: SEM image of nanofibers Silica after calcining process in 5000c and nanoparticles with 90wt% of PVA and: a) 10; b) 12 wt% PVA and diameter distribution graph

There was a diameter decrease (17.2%) compared to the initial diameter.

**Calcination process at 900°C:** As it can be seen in Fig. 7A which shows the image of the nanofibers produced at

900 °C from composite nanofibers with the concentration of 10 wt% of polyvinyl alcohol and nanoparticles of 90 wt% of the auxiliary polymer, nanofibers are formed fairly appropriately and the diameter distribution graph enjoys less dispersion and the diameter distribution is placed

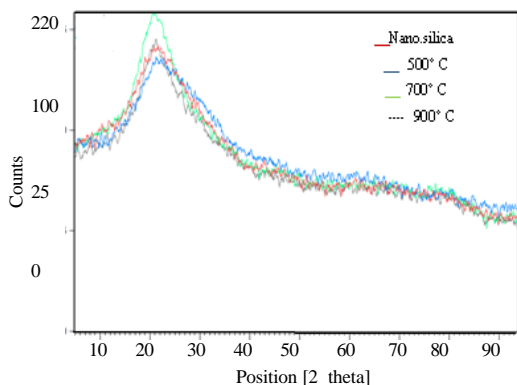


Fig. 8: Comparison XRD nanoparticles Silica with nanofibers Silica with 10wt% PVA and nanoparticles with 90wt% of PVA and calcined at 500, 700 and 900°C

between 100-240 nm with the highest frequency in 140-150 nm and the diameter mean of 168 nm. There was a diameter decrease (37.31%) compared to the initial diameter. Fig. 7B shows the SEM image and the diameter distribution graph of silica nanofibers produced from calcination process on the composite nanofibers with the concentration of 12 wt% of polyvinyl alcohol and nanoparticles of 90 wt% of the auxiliary polymer at 900°C. At this temperature and concentration, particles are aggregated and stuck. The diameter distribution is placed between 160-370 nm, with the highest frequency in 220-230 nm and the diameter mean of 249 nm. There was a diameter decrease (10.75%) compared to the initial diameter.

As mentioned above, the common feature of all web produced after calcining process is frangibility, lightness, fragility and lack of flexibility and strength and this fragility increased with increasing calcining temperature. Also, due to the removal of the auxiliary polymer nanofibers in the process, the nanofiber diameter reduced and it led to shrinking the web obtained compared to the first web. The results showed that the calcining temperature had an effect on the formation of nanofibers. However, the silica nanofibers at temperatures close to evaporation temperatures of 500°C and 700°C have better shaped and the structure is malformed at 900°C due to melting and approaching the adjacent nanofibers. The highest percentage of diameter reduction was related to the concentration of 10 wt% of polyvinyl alcohol and nanoparticles of 90 wt% of the auxiliary polymer at 700°C. This claim can be proved by Fig. 8 which shows the XRD testing on the calcined nanofibers at 500°C, 700°C and 900°C with an initial concentration of 10 wt% of polyvinyl alcohol and nanoparticles of 90 wt% of the auxiliary polymer and silica nanoparticles. To study the crystallinity, the XRD spectrum shows that the pattern

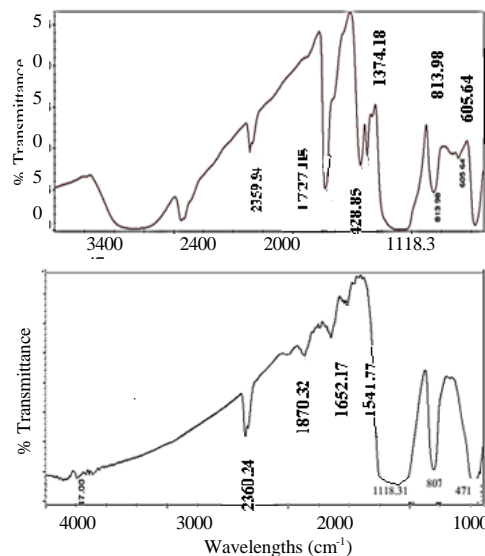


Fig. 9: FTIR: a) nanofibers composite PVA/Nano SiO<sub>2</sub>, b) nanofibers Silica after calcining process

was similar for all four charts and there is a sharp peak near  $2\theta = 20$  (SiO<sub>2</sub> peak). The area under the chart shows the level of crystallinity of the composed nanofibers and as it is evident it has had greater area at 700°C with a higher and sharper peak in  $2\theta = 20^\circ$  compared to other samples. Fourier Transfer Infrared spectroscopy analysis (FTIR) was performed for composite nanofibers PVA/nano SiO<sub>2</sub> and silica nanofibers prepared after calcining process at 500°C, 700°C and 900°C and their results is shown in Fig. 9ab. Comparing the graphs shows that the calcining process is well performed and the auxiliary polymer is completely removed from the nanofiber structure and graphs for three temperatures had a similar range.

The broad peak in the range of 3000-3650 cm<sup>-1</sup> is due to vibration of O-H bond and the peak in 2490 cm<sup>-1</sup> is because of C-H bond vibrations and peak in the range of 1430 cm<sup>-1</sup> is relevant to CH-O-CH bond vibrations in polyvinyl alcohol. And the peak in the range of 471 and 1118 cm<sup>-1</sup> is because of vibrations caused by the Si-O-Si bond which is related to the silica.

Effect of calcination duration on the morphology of nanofibers: According to the results obtained, silica nanofiber obtained from the composite nanofibers with initial concentration of 10 wt% of polyvinyl alcohol and 90 wt% of the auxiliary polymer calcined and employed at 700 ° C was chosen as the nanofiber with the optimal concentration and temperature. Then, the nanofiber with the optimal concentration and temperature was used to evaluate the effect of calcination process duration and the sample was placed in the furnace for 7 h.

Figure 10 shows the image of the silica nanofibers produced at 700 ° C for 7 hours from composite nanofibers



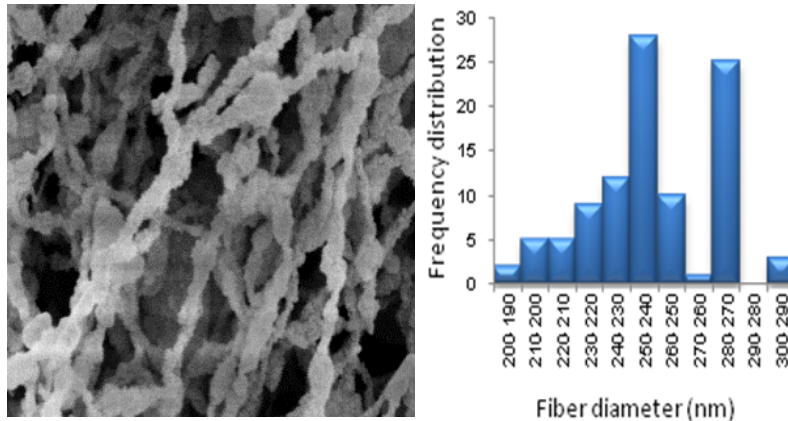


Fig. 10: SEM image of nanofibers silica after calcining process (with 10wt% PVA and nanoparticles with 90wt% of PVA) in 7000c and in 7 hours and diameter distribution graph

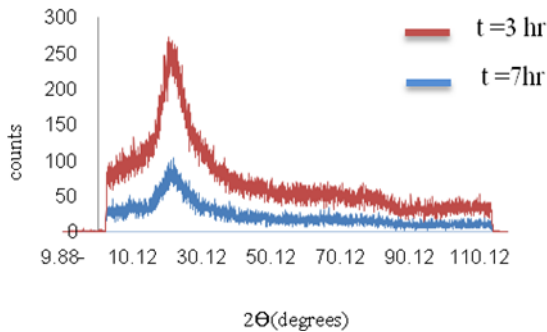


Fig. 11: Comparison XRD nanofibers Silica with 10wt% PVA and nanoparticles with 90wt% of PVA and calcined at 7000c in 3 and 7 h

with the concentration of 10 wt% of polyvinyl alcohol and nanoparticles of 90 wt% of the auxiliary polymer as well as the diameter distribution graph. The diameter distribution of nanofibers is placed between 190-300 nm, with the highest frequency in 240-250 nm and the diameter mean of March 15, 2017248 nm. There was a diameter decrease (7.46%) compared to the initial diameter.

Figure 11 compares the XRD spectra of the calcined nanofibers in 3 and 7 h. As it is shown, the peak is shorter than that of the optimal sample. As a result, the area is less and this indicates that the sample crystallinity has decreased. The results of SEM and XRD on the composite nanofibers with initial concentration of 10 wt% of PVA and 90 wt% of the auxiliary polymer calcined and employed at 700°C for 7 hours revealed the effect of increased calcining process on nanoparticle's loss and skewness from their fibrous directions. Therefore, the diameter and dispersion of nanofibers increased. This also resulted in the reduction of nanofibers diameter compared

to the pre-calcined sample. This can be due to the fact that nanofibers are exposed to a temperature below their melting temperature for a long time. This results in nanoparticles instability due to lower crystallinity of the nanofibers structure and in their loss. Moreover, the web obtained in this part was of great fragility. All these factors are the results of the reduced the crystallinity of created nanofibers and XRD testing proves it.

According to the results obtained from nanofibres as a result of the calcination process for 3 and 7 h, it can be said that duration of calcining process has an effect on the quality of the nanofibers formation as well as the amount of crystallinity so that increasing the duration of the process leads to instability of the nanoparticles as well as their decreased crystallinity. In fewer hours (3 h, because the auxiliary polymer is recently removed and nanoparticle have no enough time to disorganize the structure and fall, they can keep track of their fibrous direction. Therefore, crystallinity of the nanoparticles increases because of longer duration.

## CONCLUSION

First, composite nanofibers were produced by auxiliary polymer with a concentration of 8 wt% and nanoparticles by to 50, 70, 90 and 100 wt% of the auxiliary polymer. With the increased percentage of nanoparticles, the produced web is crispy and less flexibility. According to the SEM results, the concentration of 8 wt% of polyvinyl alcohol and nanoparticles of 90 wt% of the auxiliary polymer was selected as the optimum percentage. Because the auxiliary polymer evaporation occurs at a temperature less than 494°C, to investigate the effect of temperature, composite nanofibers were calcined at three

different temperatures of 500, 700 and 900°C for 3 h. According to results, silica nanofibers with the concentration of 10 wt% of PVA and 90 wt% of the auxiliary polymer have a better structure than other samples. According to the XRD findings, it is observed that nanofibers with the mentioned concentration enjoy greater crystallinity at 700°C. To check the duration of calcination, the composite nanofibers produced in optimal temperature and concentration were placed in the furnace for 3 and 7 h. in the calcining process for 3 h because the auxiliary polymer is recently removed and nanoparticle have no enough time to disorganize the structure and fall, they can keep track of their fibrous direction. Therefore, crystallinity of the nanoparticles increases because of longer duration.

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