INFLUENCE OF GLYOXAL IN PHYSICAL CHARACTERIZATION OF PVA NANOFIBRES

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ABSTRACT

The influence of solution composition is directly related with Poly (vinyl alcohol) PVA nanofibres properties. Electrospinning is a viable technique to develop PVA nanofibres. The presence of a crosslinking agent as glyoxal can produce variations not only in anti-water solubility effect but in the morphology of the electrodeposited fibres. The objective of this study was to characterize glyoxal influence on PVA nanofibres. Thus we studied fibre dimensions, weight of deposited fibres, fibre crystallinity. The relation between those properties and nanofibres web´ ones (colour, opacity and roughness) were studied. In this study we changed glyoxal concentration. SEM, DSC and AFM showed changes in fibres properties. We could observe how diameter fibre increased, collector surface was widely covered, and fibre crystallinity decreased. Regarding to web properties, roughness decreased and colour turned into more whiteness.

KEY WORDS

Nanofibres

Glyoxal

Poly (vinyl alcohol)

Electrospinning

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INTRODUCTION

Polymer fibres present diameters ranging from a few nanometres up to some microns. They can be used in conventional applications such as cloths, home textiles or technical ones, for example geotextiles [1], blood vessels [2], etc. Electrospinning is a common way to obtain nanofibres and it uses electrical forces to produce polymer fibres from polymer solution [2]. The polymer goes out from a syringe and a jet is produced, it travels towards a grounded collector [3]. A wide variety of polymers and chemical compounds can be used in electrospinning process: biodegradable polymers like collagen [2] and polylactic acid, water soluble polymers like PVA and polyethylenoxide (PEO), and other polymers like elastin, polyamide, polyurethane; carbon nanotubes [4], Zirconate-titane [5], and other metallic nanoparticles can be added like additives. Every polymer solution presents a characteristic viscosity that can change with concentration, temperature, and other parameters. Thus implies specific variables that should be controlled in polymer solution for spinning and of course the same as in electrospinning. Related with the process, apart from viscosity, conductivity, surface tension, electrostatic force, air friction, gravity and ambient conditions should be taken into account.[6-9]

Despite polyvinyl alcohol (PVA) is a non toxic polymer and has been commonly used in electrospinning either as PVA nanofibres [10] or as a polymer blend nanofibre [11,12] it is characterized by water solubility [13-16]. What makes no useful for so many applications as it can be easily solved by water.

Water solubility can be reduced by linear polymer crosslinking what implies more mechanical resistance in wet. It can be caused by three methods: new physical bonding, high radiation dose, and chemical reactions. New physical bonding can be produced by contact of PVA with methanol, it can be useful for three weeks more or less, and moreover it implies between 12

and 24 hours, what makes it no useful for industrial applications. High radiation dose treatment is made by gamma radiations and free radicals can be inserted such as breaking C-H. Because of radicals recombination from different PVA chains. Thus makes possible crosslinking. Chemical reactions are possible due to hydroxil group on PVA which reacts with a polyfunctional solution.

Glyoxal can be used combined with PVA as a crosslinking agent to cotton fibres. [17,18]. For their special properties, e.g. water-solubility, acid resistance, heat resistance, and good film character, the polyvinyl acetals, such as poly(vinyl formal), poly(vinyl butyral) and poly(vinyl glutaral), have shown utility in the manufacture of textiles, although biodegradability can decrease [19].

Recent developments in PVA nanofibres have heightened the need for developing more studies about water insolubility by means of crosslinking with dialdehyde [14,20] such as glyoxal [13]. Thus, different glyoxal concentration was used to prepare PVA fibres with diameter of 50-250 nm, and water insolubility was proved. However it is necessary to pay special attention to fibres surface and web nanofibres characterization.

This paper will focus on give an account of the fibres and the web nanofibres characteristics of PVA nanofibres compared with PVA nanofibres crosslinked with glyoxal. This paper begins by scanning electron microscopy (SEM) and fibre crystallinity in order to characterize developed nanofibres. Finally, the second section of this paper will examine the web properties by colour characterization and Atomic force microscopy (AFM).

EXPERIMENTAL

Materials

PVA Solviol®R 67,000 M_w aqueous solution (16% wt/v) was obtained from Novacke Chemicke Zavody, a.s. Novaky, Slovak Republic. Glyoxal and phosphoric acid were supplied by Aldrich

company. These chemicals were used without further purification. Distilled water was used as solvent.

As collector to retain nanofibres, a 100% polypropylene spunbonded commercial grade Pegatex®R (Pegas Nonwovens, s.r.o., Znojmo, Czech Republic) was used. It is characterized by 16.5 g/m^2, green colour and antistatic finishing with surface resistivity of 3.81●10^{11} Ω, and vertical resistivity about 2.78 ●10^{11} Ω, as the guidelines of UNE EN 1149-1:2007 and UNE EN 1149-2:1998 says.

PVA solution preparation

PVA solution was prepared from PVA Solviol aqueous solution, distilled water, glyoxal (if needed) and phosphoric acid at ambient temperature with constant stirring. PVA glyoxal concentrations are shown in Table 1. Conductivity value of PVA solution was controlled within 2-5 mS/cm by adding phosphoric acid. Solution viscosity was measured with a with a Brookfield LVDV I+ rotational viscometer at a temperature of 25ºC ± 2ºC . Results are shown in table 1 too.

Insert Table 1 about here

Electrospinning

The electrospinning equipment was a Nanospider^{TM} – NS Lab supplied by Elmarco (Elmarco s.r.o., Liberec-Ruzodol, Czech Republic). Voltage used was about 57 KV, the distance from tip to collector was 11 cm. The process was carried out at a temperature of 16º C and 37% of relative humidity.

Crosslinking of PVA fibres was induced by putting PVA fibres in an oven and cured by crosslinking at 130º C for 5 minutes. Conditions previously reported as the optimal ones [13].

Scanning Electron Mycrocspy (SEM).

A FEI Quanta 200 scanning electron microscope (SEM) was used for surface observation. Each sample was fixed on a standard sample holder and sputter coated with gold. It was then examined with by the SEM with suitable acceleration voltage (10 kV) and magnification. Images

with magnification at 5000 were studied with software XT Microscope Server and XT Docu (Analysis Image Processing) which allows measurement of fibre diameter.

Atomic Force Microscopy (AFM)

The surface morphology of the irradiated samples was analyzed by an Atomic force Microscope (AFM) Digital Instruments NanoScope IIIa and Digital Instruments MultiMode made by Veeco metrology Group. Scanning was carried out in contact mode. All samples were scanned at room temperature in atmosphere. The scanning size was 5µm x 5 µm.

Differential Scanning Calorimetry (DSC)

Samples were prepared as required by the equipment. DSC curves were obtained using a DSC Mettler-Toledo 821 (Mettler Toledo Inc., Schwarzenbach, Suiza) at 10 °C min^{-1} heating rate in a nitrogen atmosphere (2 Kg/cm^2). The pan type used are standard Aluminium crucibles with a 40 µl volume with capacity of sealed to avoid losses of material. The weight of samples was approximately 3 mg.

We studied if treatment could induce some effect on polymer melting temperature (Tm) or in the degree of crystallinity of PVA nanofibres (X_c). It was obtained from DSC results; degree of crystallinity is obtained as equation 1 express:

$$X_c (\%)= \Delta H_c/ \Delta H_{100} \quad (1)$$

Where ΔH_c represents the crystallization enthalpy and ΔH_{100} is the heat of crystallization for a 100% crystalline PVA, ΔH_{100}=138.6 J/g (from literature) [21,22].

Change in colour.

One of the colour systems on which most colorimetric is based on is the CIE L*a*b*system [23,24]. To check if nanofibres cover more collector surface or not, some measurements in a reflexion spectrofotometer were made. We determine colorimethric values as ΔEab too, and

opacity and its differences between samples. Tests were developed in agreement with the standard guidelines for textile fabrics [25-27]

RESULTS AND DISCUSION

PVA solubility is well known, and some studies have been developed to demonstrate the crosslinking effect of dialdehyde compounds [13,14], and how water insolubility can be obtained. In this study we analyze the influence of the concentration of crosslinking agents in nanofibres web characteristics.

The representative images of PVA fibres for solutions of different concentrations of glyoxal are shown in figure 1. There are some similarities between all images in figure 1, all of them resulted uniform nanofibres which. We can observe collector fibres for every figure too. Figure 1a is different from figure 1b, 1c, and 1d in a number of respects. To begin with, figure 1a represents nanofibres without glyoxal (PVA 0), whereas the rest contain it on PVA solution. It seems that figure 1a presents less density of fibres than the rest of images in figure 1. Nanofibres with high concentrations of glyoxal (figure 1d) tend to have greater diameters than fibres without it (figure 1a) or with low concentrations (0.1% figure 1b and 0.4% figure 1c). Diameters values are shown in table 2.

Insert figure 1 about here

Previous studies have reported that solution concentration has a significant effect on the final size of nanofibres [13,14]. The solution viscosity and surface tension play an important role in the electrospinning process to obtain continuous fibres. Moreover electrode distance, applied field strength and deposition time can induce different sizes too [13, 28]. In our research we only varied glyoxal concentration so that we could assign changes to glyoxal presence. Table 1 shows results about fibre size related to the concentration of glyoxal. We can observe that solution without glyoxal present a vast range of diameters whereas its presence reduces the range. Nevertheless the majority of diameters without the crosslinking agent are in the range of 300 – 350 nm. The most interesting finding was that when glyoxal is introduced at 0.1% wt, we can observe that diameters reduce their values, and when 0.4% wt is used the diameters are similar to the nanofibres without glyoxal. Moreover, it can be observed that there was a

significant difference in size between samples with different concentration of crosslinking agent. When glyoxal concentration in PVA solution was increased from 0.1% to 0.7% fibre diameter increased about 33.3%.

To check the quantity of web we obtain, the web weight in square meter (g/m^2) was calculated. As the collector presents a constant value of 16.5 g/m^2, we could obtain the web one as difference between the total and the collector. Table 2 shows those results and we can observe the presence of glyoxal produces an increase in the weight of the sample. It is interesting to note that they are not big differences, because we are working in nanometric scale. Thus little differences can be considered significant. We can conclude that the higher glyoxal concentrations on PVA solution, the higher mass will we deposited on the collector surface. Thus occurs because glyoxal solution increases solution viscosity, what makes more heavy the same volume.

In order to investigate thermal behaviour, such as melting, and modifications in crystalline structure, we performed DSC measurements. Pure PVA melting temperature (T_m) is about 220-225° C as literature reports [13,14,21,22]. Table 2 provides the results obtained from the preliminary analysis of our samples. Surprisingly PVA nanofibres webs present a lower T_m for pure PVA (184.69° C). Preliminary work on PVA electrospun nanofibres was undertaken by Tong Lin [14]. They produced PVA nanofibres form 16% PVA solution, and PVA nanofibres crystallinity reached about 28.8% [14]. There was no significant difference between their result and our study. The web from PVA 0 presents a crystallinity of 26.4 %. As this one does not contain glyoxal, we considered they present similar values. Table 2 illustrates that when glyoxal is introduced in PVA solution T_m is more or less the same for every sample and we can´t suggest there is a variation as very little changes can be observed. Previous studies have reported that, crystallinity is reduced as glyoxal content increases [14,29], and they concluded that the crosslinking reaction reduced PVA crystallinity due to change of crystalline morphology induced by the crosslinker. Thus fits with our analysis for glyoxal samples when concentration is higher than 0.4 g/L. The most striking result to emerge from the data is that for concentrations in the range from 0 to 0.4 g/L changes in crystallinity are not significant. This findings further support the idea that form concentrations higher than 0,4 g/L, the higher concentration of

crosslinking agent the less crystallinity. Furthermore, crystallinity can be influenced by some additives and several web post-treatments [30]. It is also important to remark the importance of the distance between the electrodes [31]. Further studies, which take these variables into account, will need to be undertaken.

PVA films on different surfaces do not present great differences of colour ($\Delta E \approx$ 1-4) [15,16]. The results obtained from the colour measurement are shown in table 3. First of all we studied chromatic values and we calculated differences from the sample without glyoxal (PVA 0). We could find some variations in lightness/darkness (ΔL^*). When glyoxal is added lightness is increased in about 5 units what means that it could be whiter. On the other hand when chromatic values are analyzed, significant differences could be notice. On the values for redness/greenness (a^*) some changes can be appreciated towards more red colours. What is interesting in this data is that sample losses green colour. We should remember that collector substrate is green and PVA nanofibres produce a white web on the substrate. As a^* moves towards higher values it actually means that green colour is lower. The Δa values differs from Δb ones. The results obtained from the preliminary analysis of b^*, show colour modification on yellowness/blueness but not as high as for redness/greenness. The b^* values moves to a downward trend and indicates yellowness is reduced to more blueness. Despite those results, Δb differences are not higher than 2.5 units, so that it could be noticeable but not so much than variations in redness/greenness. Variations in the parameters of colour present no logical relation with glyoxal concentration. However, despite this, we could confirm that glyoxal presence makes samples less green and whiter. When colour differences are studied the webs on the collector which contain glyoxal, present great differences respect the web of PVA without glyoxal (ΔE >14). Some of the issues emerging from this finding relate specifically to results on fibre diameter and g/m^2. As we mentioned above, glyoxal presence produces fibres with higher diameter and more weigh in the same surface. Thus, fits with colour changes because more density of PVA fibres and more width cover more collector surface. As collector surface is green and PVA nanofibres are white, more opacity is shown because of glyoxal presence.

Because of that evidence, we studied opacity for each sample. PVA films show low values for opacity [15, 16], nevertheless if we study results from table 3, we can observe it increases when

glyoxal is introduced in PVA solution. This study produces results which corroborate the findings of fibre diameter, weight in surface unit, and colour.

In order to characterize web surface topography AFM studies were developed. Figure 2 presents the 3D image of the nanofibres analyzed. It can be seen from the AFM images that the electrospun fibres have uniform smooth surfaces with cylindrical structure. They present diameters discussed above and shown in table 2.

Insert figure 2 about here.

Table 2 shows the root-mean-squared roughness (R_{rms}) values determined by AFM of the PVA nanofibres with different concentration of glyoxal. These values change towards lower values when glyoxal concentration increases. As can be observed in figure 3, this fact is due to the width of the peaks that decreases as a consequence of the glyoxal concentration presence. The presence of glyoxal makes roughness to decrease. At first PVA samples were more roughness because nanofibres were completely defined. On the other hand, when glyoxal was introduced in PVA solution, fibres overlap one to another and it produces fibres with higher diameters but not so defined limits as melting joints can be observed. They are caused because when glyoxal is introduced on PVA solution, more time is required to obtain the fibre and when a new one crosses on; solved polymer is mixed with the polymer of the previous one.

Insert figure 3 about here

CONCLUSIONS

Electrospun fibres´ properties depend on a vast amount of variables. Some of them are directly related with the electrospinning conditions, on the other hand collector plays an important role in the final product properties and moreover, polymer solution is important too. Polymer solution can vary of course with polymer nature. In our study we selected PVA as it is an important

polymer in electrospinning field. Furthermore, pH values, polymer concentration, additives chemical structure, additives concentration, temperature, etc. can influence final result.

This study was focused on the influence of glyoxal presence on PVA solution and produced results which corroborate the findings of a great deal of the previous work in this field. Concisely we stated changes in fibre size depend on the product concentration in solution and we demonstrated that crosslinking agent influences too. The weight in surface unit (g/m^2) increased as well. The higher concentration of glyoxal on PVA solution the higher diameters we reached.

Thus is directly related with colour changes as collector was not white but coloured one, treated samples were whiter and presented more surface covered when glyoxal concentration was higher. In contrast to earlier findings, however, results show that low concentrations of glyoxal (lower 0.4) do not cause significant variations in cristallinity, and crystallinity decreases with crosslinking agent when concentration is higher than 0.4 g/L. Thus in the range of concentrations studied (0.1-0.7 % w/v) implies the nanofibres properties were influenced by glyoxal presence, not only in anti-water effect as demonstrated previously, but in fibre and web properties.

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FIGURE CAPTIONS

Figure 1.- SEM of PVA fibres as a function of glyoxal concentration. a) PVA 0 = without glyoxal. b) PVA G1= with 0.1% w/v of glyoxal. c) PVA G1= with 0.4% w/v of glyoxal. d) PVA G1= with 0.7% w/v of glyoxal.

Figure 2.- AFM 3D representation of the surface topography (5µm x 5µm) of PVA fibres as a function of glyoxal concentration. a) PVA 0 = without glyoxal. b) PVA G1= with 0.1% w/v of glyoxal. c) PVA G1= with 0.4% w/v of glyoxal. d) PVA G1= with 0.7% w/v of glyoxal.

Figure 3.- Roughness profiles of the surface topography (5µm x 5µm) of PVA fibres as a function of glyoxal concentration. a) PVA 0 = without glyoxal. b) PVA G1= with 0.1% w/v of glyoxal. c) PVA G1= with 0.4% w/v of glyoxal. d) PVA G1= with 0.7% w/v of glyoxal.
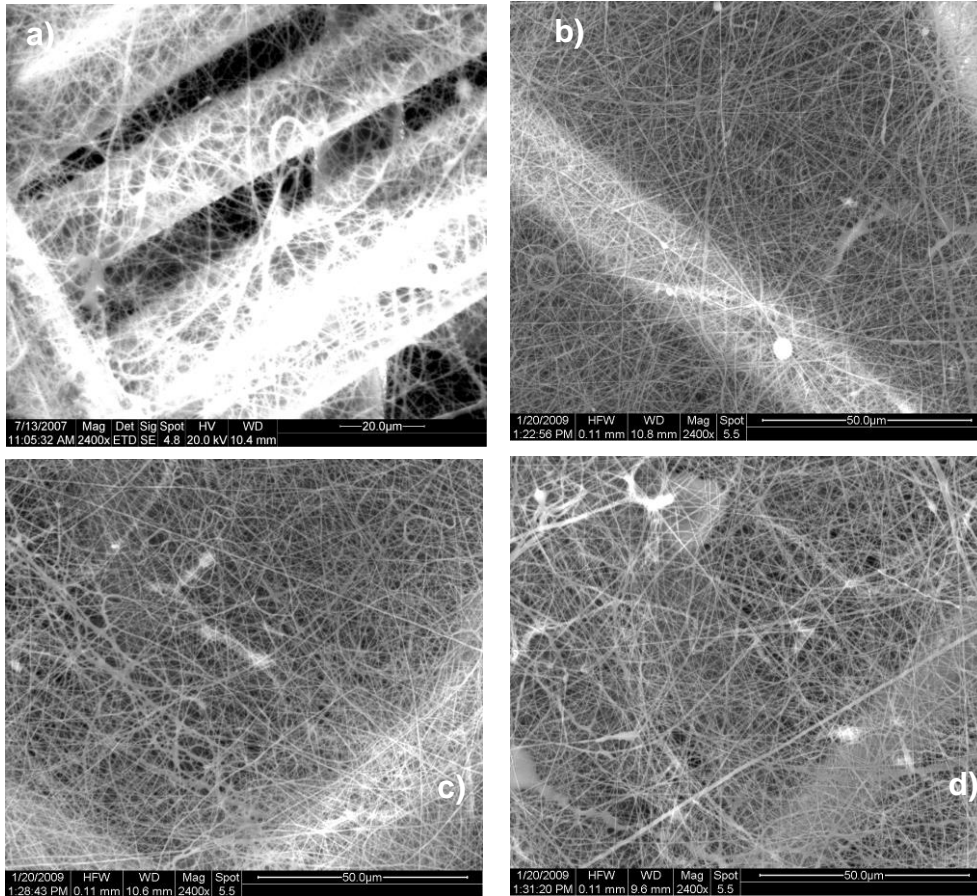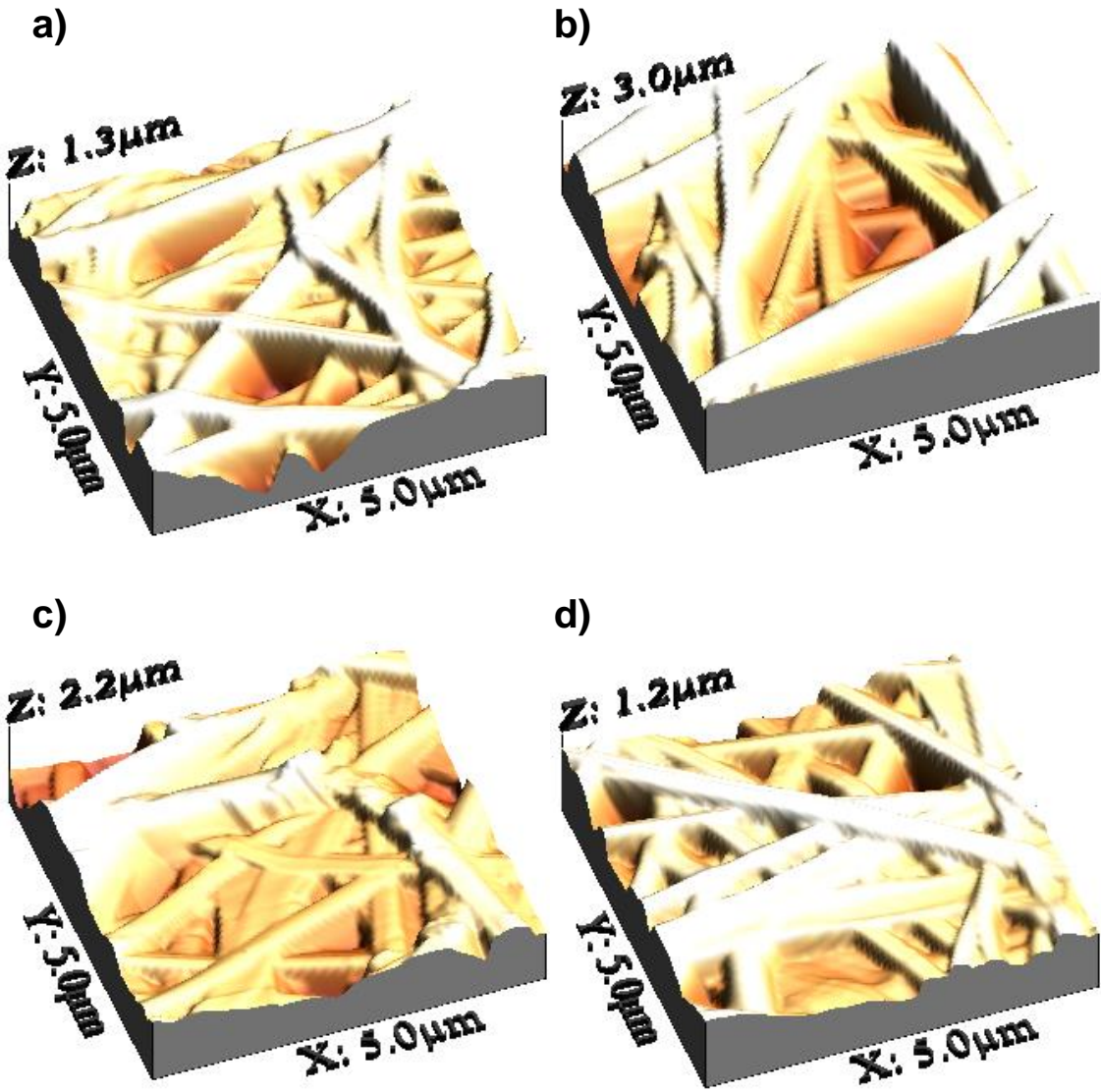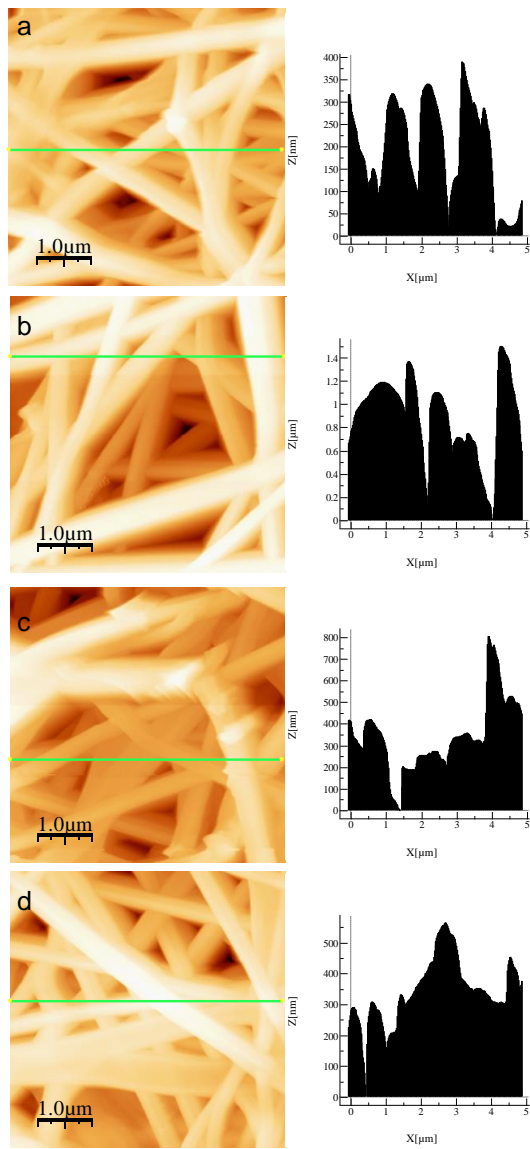
FIGURES

FIGURE 1

FIGURE 2

a)

b)

c)

d)

FIGURE 3

Table 1.- Glyoxal concentrations for PVA solutions

SAMPLE	PVA 0	PVA G1	PVA G2	PVA G3
PVA (g/L)	14	14	14	14
GLYOXAL (% w/v)	0	0,1	0,4	0,7
Viscosity (cps)	*887,3*	*1213*	*1216*	*1445*

TABLE 2

Table 2.- Fiber diameter, density, T_m, crystallinity and roughness.

SAMPLE	Fiber diameter (nm)	Net density (g/m^2)	T_m (º C)	Cris (%)	Rrms (5µx5µ) (Nm)
PVA 0	250 – 350	0,130	184.69	26, 4	0,26
PVA G1	200 – 300	0,201	184.78	28,6	0,18
PVA G2	300 – 350	0,217	186.22	26,1	0,13
PVA G3	350 – 400	0,224	185.31	17,1	0,10

TABLE 3

Table 3.- Colour measurement

SAMPLE	ΔL	Δa	Δb	ΔEab	Opacidad	Δ opacidad
PVA 0					46,2	
PVA G1	4,3087	13,5586	-1,7765	14,3373	58,92	12,72
PVA G2	6,0936	16,2637	-2,3397	17,5247	62,52	16,32
PVA G3	5,0875	16,342	-1,9802	17,2298	63,34	17,14