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# The effect of annealing on the structuer of alignmenet nanofiber conducting polymer (PAni.CSA/PEO)

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## ABSTRACT

Alignment nanofibers of PAni.CSA/PEO blends with different concentration of polyethylene oxide (PEO) have been prepared using the electrospinning technique. The details of the preparation method are well described. The method is relatively simple and is easily controlled. A systematic investigation on the effect of annealing temperature on the structure of PAni.CSA/PEO nanofibers was studied. Morphology and diameters of the as-spun and annealing nanofibers were studied by Atomic Force Microscope and found the annealing increases the nanofiber diameter and had clear effect on the morphology and orientation of nanofibers. The X-ray diffraction analysis shows the effect of annealing on the nanofibers PAni.CSA/PEO crystallization is unsystematic and depended on the concentration of PEO in the blend.


## KEYWORDS

Annealing;
Electrospinning;
Nanofibers;
PAni.CSA/PEO.

## INTRODACTION

Electrostatic fiber spinning or 'electrospinning' is a process for forming fibers with sub-micron scale diameters through the action of electrostatic forces. The collected fibers as continuous and alignment have some useful properties such as high surface area to mass ratio, and thus have great potentials in filtration, sensing applications[1].

The reduction of the diameter into the nanometer range gives rise to a set of favorable properties including the increase of the surface-to-volume ratio, modifications of the release rate or a strong decrease of the concentration of structural defects on the fiber surface which will enhance the strength of the fibers[2], low density, low specific mass and high pore volume which make them appropriate multiple extra functions into electrospun nanofibers to broaden their significances in applications[3]. Some applications above need to be subjected the nanofibers to heat, because that; in this work the samples were annealed to see the effect of this process on the nanofibes structure, as know the annealing, in materials science, is a heat treatment that alters a material to increase its ductility and to make it more workable. It involves heating material to specific temperature (depended on the type of material), maintaining a suitable temperature, and then cooling. Annealing can induce ductility, soften material, relieve in-

ternal stresses, refine the structure by making it homogeneous, and improve working properties[4].

This study describes electrospinning of nanofibers from polyethylene oxide (PEO) and its blend with polyaniline (PAni.CSA). The focus is on the effect of annealing on the structure properties of PAni.CSA/PEO nanofibers, like alignment, diameters, crystalline etc.

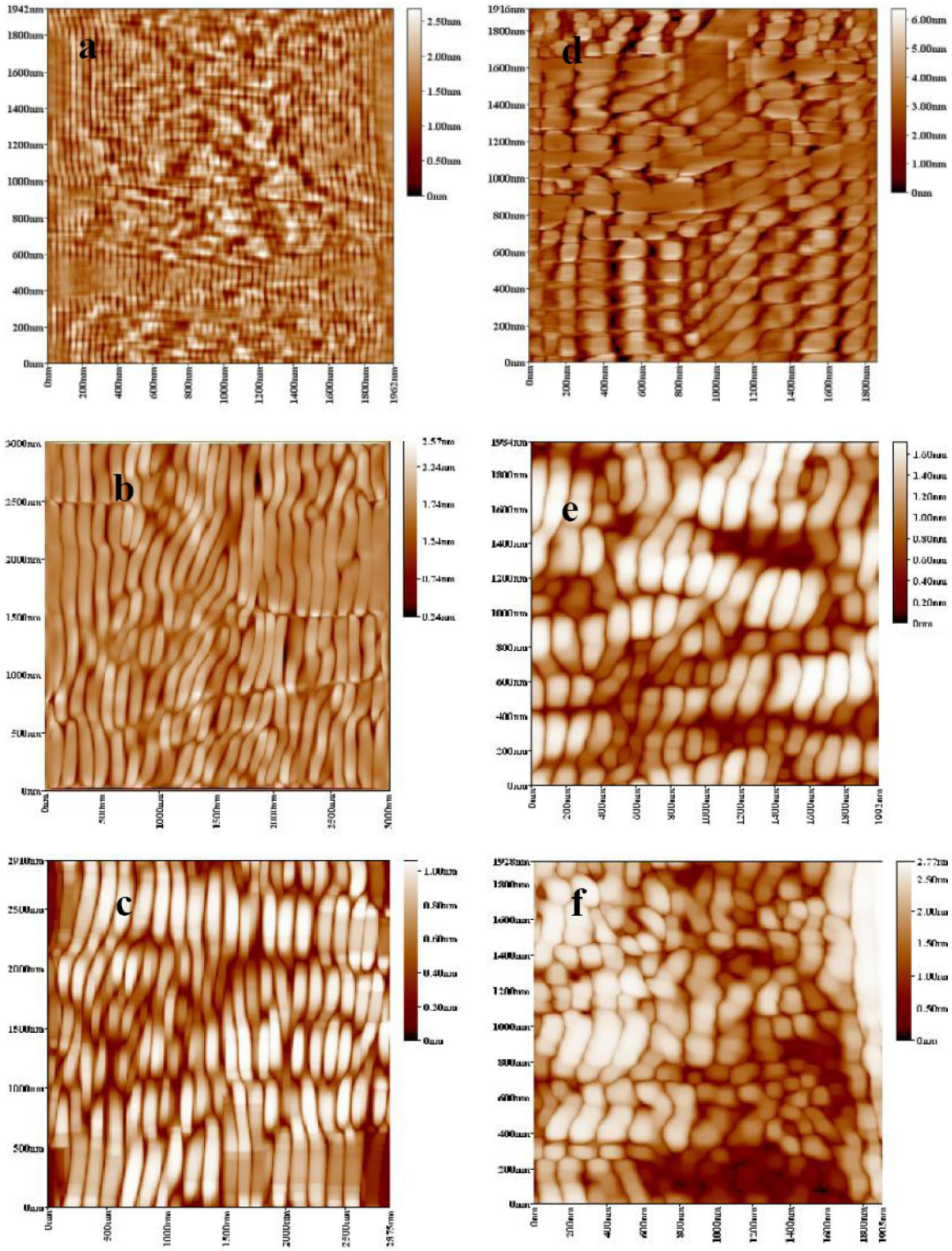


**Figure 1 : AFM morphology for as-spun nanofibers of PAni.CSA/PEO Blend (a) 8 wt% PEO (b) 30 wt% PEO (c) Pure PEO, for annealing sample (d) 8 wt% PEO (e) 30wt% PEO (f) Pure PEO .**

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## EXPERMENTAL

Polyaniline emeraldine base was synthesized by the oxidation polymerization of aniline in acidic media. Using a method similar to the reported by[5,6]. 100 mg of PAni emeraldine base was mixture with 129 mg of camphorsulfonic acid (CSA) provided by (Fluka) and dissolved in 10 ml $CHC1_3$ provided by (Labort) put under stirring for 8-9 hours. The resulting deep green solution was filtered. PEO ($M_w$ 200.000 provided by (Alpha chemika) was added to assist in fiber formation with different weight ratios concentrations (8 wt%, 30 wt %) for each 10 ml from PAni.CSA solution. Polymer nanofibers were obtained using the electrospinning technique.

About 1 ml of the solution blends was placed in hypodermic syringe, the syringe was connected to a metal needle, and in horizontally state for moving collector (Rotating a cylinder aluminum collector), the tip of the needle and moving collector was held at a adjustable DC power supply (as anode and cathode) respectively. The processing conditions of the different solutions concentrations were as blew: the voltage electrospun is 10 KV, the distance between needle and collector is 20 cm, After electrospinning, the collcted fibers samples were kept in an oven at room temperature for a couple of days for drying.

Following that these nanofibers were annealing at 60 °C in atmosphere for 30 minute.

Nanofibers polymers blends have been investigated employing atomic force microscopy (AFM) (AA3000 Angstrom advanced Inc.), this investigate the nanofibers diameters and alignment as well as the defects or the damaging in the nanofibers.The structural characteristics of PAni.CAS/PEO nanofibers films determine by X–ray diffraction (XRD). The measurement has been done by using Philips X– ray diffractometer that having the following features: source Cu, current was (30 mA), voltage was (40 kV), and the wavelength(λ) =1.5405A°. The characterization bonds for the functional gropes measured by Fourier Transform Infrared (FT-IR) spectra (Shimadzu FTIR-8400S), all spectra were recorded between 4000 and 400 $cm^{-1}$.

## RESULT AND DISCUSSION

Figure 1(a, b, c) It was obviously observed that the PAni.CSA/PEO nanofibers exhibited uniaxially aligned nanofibers were obtained in all blends (8 wt%, 30 wt% PEO, and Pure PEO) under the same conditions. However, the 8 wt% concentration appears to have the best alignment and the average diameter was 40.49 nm, 92.35 nm, and 111.37 nm for 8 wt%, 30 wt% and pure PEO concentration respectively. It can be also seen from these image that there are fibers which are free from defects such as beads, relatively smooth with a generally uniform thickness along the fiber especially at low concentration.

Figure 1(d) shows the AFM images of the PAni.CSA/PEO (8 %wt) nanofibers after annealing at 60°C. first not the a some number of inter-fiber welds between fiber, there was increase in fiber diameter from 40.49 nm for as-spun to 63.76 nm for annealing sample, also the uniform thickness of as-spun well be change after annealing and see like waists along the fiber, in addition some unsystematic in the orientation of nanofibers.

At Pure PEO and high PEO concentration 30 %wt (Figure 1. e, f ) the effect of annealing well be as bigger than 8 % wt, but without the waists, and the fibers will be shorter and in some zone be random and unorganized, in other zone the welds between fibers loss it the fibers state, in addition the increasing in fiber diameter (see TABLE 1). The creation of welds at low PEO contents and the unacceptable transformation of the nanofibers morphology at high PEO concentrations were attributed to the low melting point (65°C) of PEO in fiber state[7].

Figure 2(a b and c) showed X-ray diffraction (XRD) patterns from the as-spun and the annealing

**TABLE 1 : The effect of annealingn on the structure parameters of Pure and PAni.CSA/PEO nanofibers blends**

| PEO wt% | Ava. Nanofibers diameters As -spun | Ava. Nanofibers Diameters Annealing | Grin Size (A°) As-spun | Grin Size (A°) Annealing |
|---|---|---|---|---|
| 8 wt% | 40.49 nm | 63.76 nm | 66.28 | 85.70 |
| 30 wt% | 92.35 nm | 98 | 38.92 | 38.89 |
| Peru PEO | 111.37 nm | 119 | 35.38 | 17.67 |

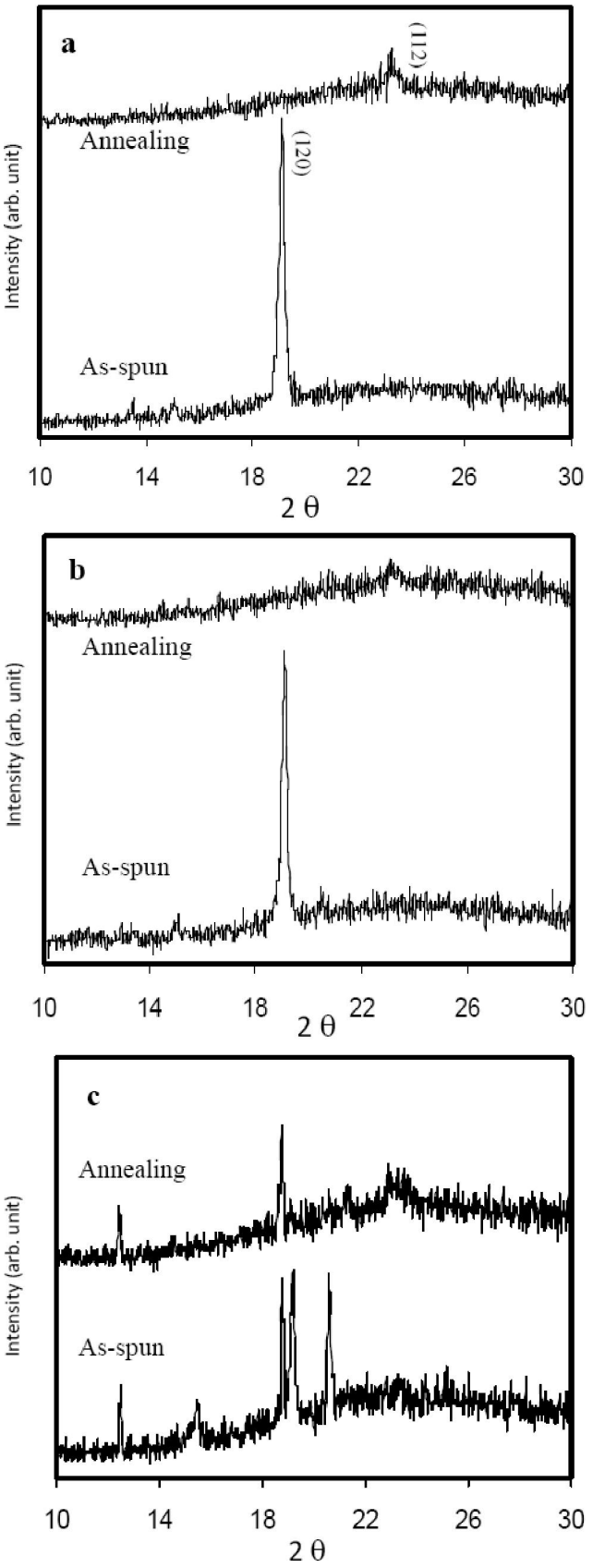


**Figure 2 : The XRD spectra of (a) Powder PEO (b) PAni.CSA/ PEO 8% wt (c) PAni.CSA/PEO 30% wt**

nanofibers for Pure and different concentration of PEO. The diffraction pattern of as- spun pure PEO with 30 wt % concentration showed only one peak characteristic of crystalline PEO at 2θ=19.13 is assigned as (120) reflections[8,9], this peak is disappearance after annealing as show in the diffraction pattern of annealing nanofibers Figure (2 a), but can be found that the growth new week peak at 2θ=23.24, also the annealing change of crystalline orientation and this confirms by shift of the position of the scattering peak from 2θ=19.13 to 23.24 that corresponds (120) and (112) reflections respectively, same as this behavior see in PAni.CSA/PEO nanofiber at 30 wt% PEO concentration Figure (2 b), this is meaning that the amorphousity of sample is increasing with annealing at high PEO concentration (both of pure PEO and with PAni.CSA), and this behavior attribute to the aforementioned reason above about the low melting point (65° C) of PEO in nanofiber state.

In contrast, the annealing PAni.CSA/PEO nanofiber with 8 wt% PEO concentration Figure 2(c) showed decreased in numbers of peaks with annealing, while see only two diffraction strong peaks indexed with values of 2θ =12.44 and 2θ =18.12 comparing by five peaks in as-spun nanofiber, as well as the grain size increasing from 66.28 nm for as spun to 85.70 nm for annealing nanofiber. This main the annealing can dramatically increase the amount of crystallinity in sample and enhance the structure of PAni.CSA/PEO nanofiber, and this agreement with[10], Form the other hand the effect of annealing on the state of crystalline in PAni.CSA/PEO depended on the ratio concentration of PEO in blend.

Figure 3 show the compares of FT-IR spectra for as-spun and annealing PAni.CSA/PEO nanofiber. The spectra of PAni.CSA/PEO in as-spun samples was seen as strong IR absorption peaks at a wave number of 3450 $cm^{-1}$, these refer to presence of OH bonding because of hydrogen bonding in the NH stretch between (PEO) and (PAni.CSA), from this Fig notice that the spectrum of a thermal treatment presents similar features to those of a as-spun, observed a small change in intensity of the bands in the annealing spectrum. Nevertheless, there are no additional vibrational bands as compared to spectrum of the as-spun sample. Therefore the annealing of the PAni.CSA/PEO nanofibers does not effect on the bonds structure and the agree-

ment with Sujith et. al.[11]

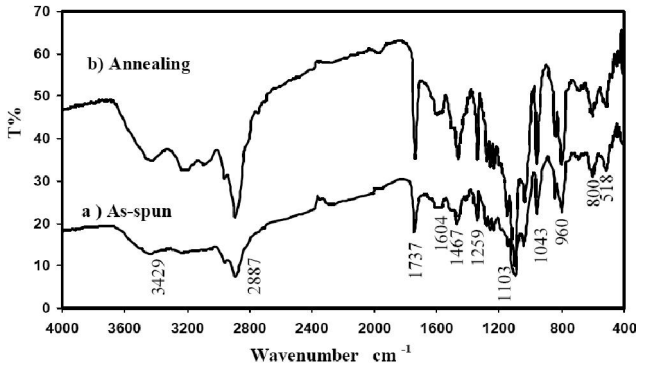


**Figure 3 : The FT-IR spectra of PAni.CSA/PEO blend at 8 wt % PEO a) as-spun b) annealing at 60°C**

## CONCLUSIONS

In the present work the nanofibers PAni.CSA/PEO blends have been successfully produced using the electrospinning techniques. The effect of annealing temperature on the morphology, nonfibers diameters, crystallinity, and functional gropes of electrospun PAni.CSA/PEO nanofibers were investigated. It was found that the annealing had unsystematic effect on the morphology, fiber uniform as well as the crystallinity state of samples where it effect had major dependent on the PEO concentration in samples. While the nanofiber diameters increasing with increase the annealing temperature of nanofibers. Also see the annealing does not effect on the bonds structure of the PAni.CSA/PEO nanofibers blands.

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