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Effect of PEG on Structure and Physical Properties of PVA/CMC Nanofiber

Karim Y. Nabat [a*], H. Farag [b], Mona .E. Ossman [c], M. Taha [b,d], and Nahla. A. Taha [e*]

[a]*Petrohemical Engineering Department, Faculty of Engineering, Pharos University, Alexandria 21544, Egypt.*
[b] *Chemical Engineering Department, Faculty of Engineering, Alexandria University, Alexandria 21544, Egypt.*
[c] *Environment and Natural Material Research Institute (ENMRI), City of Scientific Research and Technology Application (SRTACity), Alexandria 21544, Egypt.*
[d]*University of Science and Technology, Enviromental Engineering program, Zewail City of Science and Technology, 6th October city 21544, Egypt*
[e] *Modeling and simulation Research department ,Advanced Technology and New Materials Research Institute (ATNMRI), City of Scientific Research and Technology Application (SRTACity), Alexandria 21544, Egypt.*

Abstract

Nanofibers are a new class of material with unique features and a wide scope of uses. This research article studies the preparation of a novel polymer nanofiber composite from polyvinyl alcohol (PVA) (10%) blended with carboxymethyl cellulose (CMC) (2.5%) with different doses of Poly Ethylene Glycol (PEG) (0.25, 0.5, 0.75 g). Four different samples of PEG0, PEG1, PEG2 and PEG3 were prepared (6.25,7.5, 8.75 and 10 total % (w/v) respectively and The morphology and the average diameter of the electro spun fibers were observed using Scanning-electron microscopy (SEM). The SEM analysis was used also to help in optimizing the PEG concentration and the electrospinning condition (needle-to-collector distance, voltage, and flow rate), that can be used for the preparation of nanofiber membrane composed of PVA/CMC/PEG. The results showed that the nanofiber morphology and diameters of the nanofibers (PEG1, PEG2 and PEG3) were enhanced and the beads disappeared with increasing the PEG concentration and the optimum values for the electrospinning parameters to obtain PEG3 nanofibers with less average diameter are flow rate of 1ml/h, Voltage of 25 kV and Needle-to-collector distance of 25 cm with average diameter of electro spun nanofibers 362 nm. Also the thermal stability of the PVA/CMC/PEG (PEG3) nanofiber membrane was investigated by TGA. The results explained that the sample lost about 7 % at 115℃ in the first stage, and lost 93 % after reaching 550℃ as second stage of polymer degradation. The water contact angle (WCA) for PEG3 was measured to assess the water absorption and swelling properties of the membrane and the results showed the WCA was 45.35. This value was evidence that the membrane had hydrophilic properties. Treatment with cross linking is necessary if it will use in waste water applications.

1. Introduction

Nanofibers are a new chapter of material with outstanding characteristics (such as high surface area and little pore sizes), which make them reasonable for a wide extent of applications, for example, filtration [1], hydrophobic coating [2], energy storage [3], capacitors [4], drug delivery [5], sensors [6] and fuel cells [7]. The polymeric nanofibers applications have been extended since the late twentieth century on account of their significantly porous structure and the new era of material design [8]. The nanofibers can be delivered effectively utilizing electro spinning system considering its ability to use a wide scope of polymers, decreasing the fiber width in the size of nanometer, and credibility of scale-up production [9-10]. Electrospinning strategy was created during the most recent decade (from 2012) for the manufacture of fibers in submicron to nanometer scale extend. The benefit of electrospinning is that it doesn't require any functionalization procedure, it needs just a solvent that can break up the polymer. Electrospinning fundamentally relies upon the high electrostatic powers. factors that impact the electrospinning procedure are polymer concentration, viscosity of the

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solution, flowrate, electric field intensity and the distance between the needle and collector [11].

Despite the fact that electro spun nanofibers have been utilized in filtration applications for over twenty-five years, the vast majority conducted research has concentrated on engineered polymers [12]. Some other studied focused on electrospinning of natural polymers [13]. Studied on complex polysaccharides and plant sources polymers have not been sufficiently inspected for their potential to be electro spun into nanofibers as a result of the repulsive forces in solution among the poly-anions as well as the differences in chain configurations. So the efficiency and reproducibility of the electrospinning process and the fiber uniformity of natural polymers remains a challenge, thus limiting their practical application [14-15].

A strategy to beat this disadvantage is carried out by mixing the bio-polyelectrolytes with a non-harmful, water dissolvable, biocompatible, engineered polymer, which can decrease repulsive forces inside the charged biopolymer solutions and permit fiber electrospinning [16].

Polyvinyl Alcohol (PVA), is a synthetic hydrophilic polymer, promptly makes into film because of the way that it contains a lot of – OH groups, which give a stage to hydrogen bond development with water particles. In the view of the amazing properties of PVA, much enthusiasm for research has been given to its electrospinning for usage in various applications such as biosensors [17, 18], filtration membranes [19-21], pharmaceutical and therapeutic devices due to its non-poisonous quality, great chemical and thermal stability, high bio compatibility and its great fiber shaping [22-24]. To redesign mechanical properties, various polymers are routinely added to the PVA arrangement during the electrospinning procedure [25].

Cellulose is natural polysaccharide known as biodegradable material that found in nature [26]. Sodium carboxymethyl cellulose (CMC) is a cellulose subordinate which made by the carboxymethylation of cellulose and has carboxymethyl groups (- CH2-COOH) bound to a portion of the hydroxyl groups of the glucopyranose monomers that build up the cellulose structure [27-28]. PVA mixed with polysaccharides, for example, CMC can reduce repulsive forces within the charged biopolymer solutions and allow fibers spinning and make the mixture alluring as a direct result of simplicity of modification, and great biocompatibility [29]. Polyethylene Glycol (PEG) is a thermoplastic polymer with flawless crystalline and great water soluble. Various studied showed that the addition of poly (ethylene glycol) (PEG) was effective for the fabrication of fiber mats blended with natural polymers [30, 31].

In this work, the PVA/CMC was blended with PEG in different ratios and study the characterization of produced nanofibers by using the electrospinning technique.

2. Materials and Methods

2.1. Materials

Polyvinyl alcohol (PVA, 98% hydrolysis, Mw = 72,000 g mol^{-1} purchased from Merck Schuchardt OHG, Germany), Sodium carboxymethyl cellulose (CMC, Mw = 90,000 g mol^{-1}, purchased from Sigma Aldrich, Germany), and Poly Ethylene Glycol (PEG, Mw = 6,000 g mol^{-1}, purchased from Alfa Aesar, UK).

2.2. Preparation of PVA/CMC solution

1 g of PVA powder was dissolved in 10 mL of distilled water (DW) and warming at 90° C for 3 h with delicate blending by a magnetic stirrer equipped with heater and sensor to maintain constant temperature in a closed container to set up a PVA solution (10% w/v). PVA solution was left to chill off to room temperature, and stored in closed jug till use after complete dissolving. Along these lines, CMC solution (2.5% w/v) was set up by adding 0.25 g of CMC to 10 mL of distilled water (DW) with delicate mixing by a magnetic stirrer at room temperature for 30 min, at that point the CMC solution was left and stored in closed container till use. PVA/CMC (PEG0) solution was prepared by combining the recently arranged solutions at 60°C for 1 h with blending to guarantee homogeneity in the prepared solution.

2.3. Preparation of blended PVA/CMC/PEG solutions.

CMC/PEG solution with different concentration (5, 7.5, 10 % w/v) were prepared by adding different doses of PEG (0.25, 0.5, 0.75 g) to the CMC solution.

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Then the CMC/PEG solutions with different concentrations were added to the PVA solution (10% w/v) at 70° C and blended for 1 h to get a homogeneous solutions of PVA/CMC/PEG with different concentrations (7.5, 8.75, 10 % w/v) (PEG1, PEG2 and PEG3) separately. The total volume of the blend solution was always maintained at 20 ml. Table (1) lists the concentrations and volumes of the blend solutions.

Table1. Concentrations and volumes of blend solutions of PVA/CMC/PEG.

Sample code	Concentration (%) (w/v)		Total (%) (w/v)	Total volume of blend solution (ml)
	PVA	CMC/ PEG		
PEG0	10	2.5	6.25	20
PEG1	10	5	7.5	20
PEG2	10	7	8.75	20
PEG3	10	10	10	20

2.4. Electrospinning of nanofibers

Electrospinning was carried out using a 3 mL syringe. The feeding rates of the solutions were 0.5-1.5 mL/h with applying a voltage range of 10-40 kV. The electro spun fibers were collected on an aluminum foil and the collecting distance between the needle tip and the collector was 10-25 cm.

2.5 Characterization of nanofibers

2.5.1. Scanning Electron Microscope (SEM).

The surface morphologies of the PVA/CMC nanofibers and PVA/CMC/PEG nanofibers were evaluated by Scanning Electron Microscope (SEM, JEOL JSM-6360LA. Japan). Sampled surfaces were covered with a slim layer of gold before testing. The fiber diameter of the electro spun nanofibers were estimated using SMILE-VIEW program from the SEM images in original amplification. In any event 100 separated nanofibers were arbitrarily chosen, and their diameters were mea¬sured and the average diameter was estimated.

2.5.2. Fourier-transform infra-red spectra (FT-IR).

FT-IR of nanofibers were tested by a Fourier Transform infrared spectrometer (FTIR, Shimadzu FTIR-8400 S, Japan) FTIR spectra. The trials were completed in the scope of 400 to 4000 cm-1. The infrared spectra were estimated in the transmission mode utilizing thick tangles of electro spun nanofibers.

2.5.3. Thermo-gravimetric analysis (TGA).

Thermal stability of electrospinning nanofi¬bers was examined utilizing Thermo-Gravimet¬ric Analyzer (Shimadzu Thermal Gravimetric Analysis (TGA)—50, Japan). All recorders were performed under a nitrogen environment with a flow rate of 10 ml/min by increasing the material temperature from 25 0C to 600 0C at a heating rate of 10 0C min-1

2.5.4. Water contact angle (WCA).

The static contact angle (θ) of a water droplet with the Nano fibrous mats (NFs) was measured using a Drop Shape Analysis System (Ramé-Hart Instrument Goniometer, model 500-F1, France). The NFs samples (1 cm x 1 cm) were positioned horizontally on a glass slide. Deionized water was dropped onto the fixed sample at ambient temperature (~ 26 oC). The contact angles were calculated using the software by analyzing the drop shape. Data for the contact angle are the mean of three measurements. 'Automatically update document styles' to make the styles available. You will in this case have to adjust the margins of the document, using one of the first two options from the 'Document formatting' menu.

Alternatively, you may insert the whole text or parts you previously prepared by using on the 'Insert' menu of Word® the option 'File…'. In that case take care to retain the above mentioned section breaks. After the file is inserted you can style it by placing the cursor in each paragraph and clicking the required style on the drop-down menus.

3. Results and discussions
3.1. Study of the surface morphology using SEM:

The morphology and the average diameter of the electro spun fibers were observed using Scanning-electron microscopy (SEM). SEM analysis was used also to help in optimizing the PEG concentration and the electrospinning condition (needle-to-collector

distance, voltage, and flow rate), that can be used for the preparation of nanofiber membrane composed of PVA/CMC/PEG.The SEM figures of PVA/CMC (PEG0) nanofibers were shown in Figure (1) with magnification factor of 7000 and 10000. The f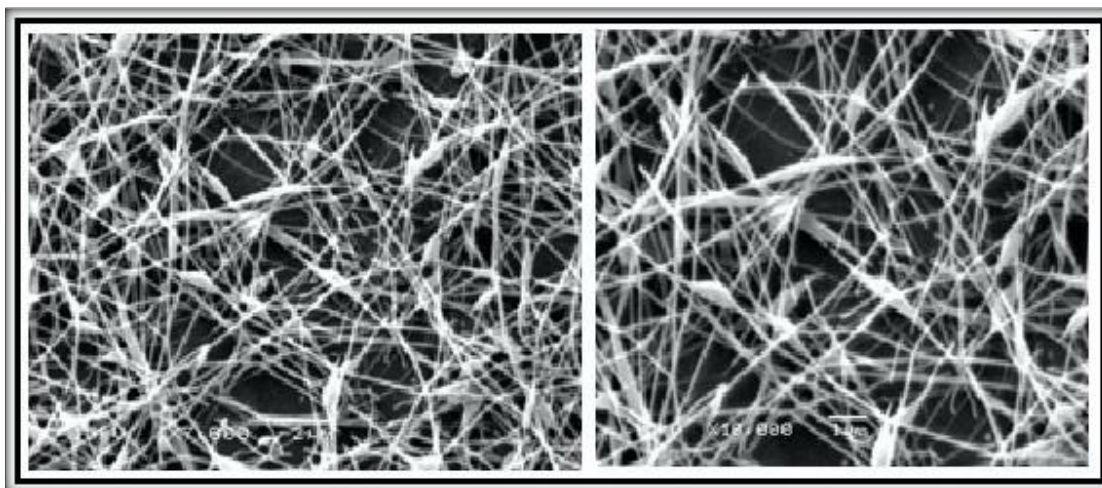igure showed a ribbon shaped fibers with a wide diameter distribution in the Nano-micro scale and the morphology of the fibers had beads and not smooth. To reach our research goal, the nanofiber was modified by adding different concentration of PEG to enhance the surface morphology of the Nano fiber mat.

Fig.1. SEM for PEG0 nanofiber with different magnification factor (5000-10000), electrospinning condition: Flow rate= 0.5 ml/h, Voltage = 40 kV, needle-to-collector distance = 20 cm

By adding different concentration of PEG to PEG0 to obtain PEG1, PEG2 and PEG3 with (concentration 7.5, 8.75, 10 % w/v respectively), the results showed that the nanofiber morphology and diameters of the nanofibers (PEG1, PEG2 and PEG3) were enhanced and the beads disappeared with increasing the PEG concentration to reach uniform, regular and smooth nanofiber at (10% w/v) concentration of PEG (PEG3) as shown in Figure (2). On other hand it was found that adding PEG to PVA/CMC blend leads to fiber diameter reduction (PEG3 nanofibers diameter < PEG2 nanofibers diameter < PEG nanofibers diameter). For enhancing the PVA/CMC/ PEG nanofiber, the effect of changing the electrospinning parameters were studied to reach smooth and uniform nanofiber mat. Figure (3) presented the SEM images for the effect of changing needle-to-collector distance from 10 to 25 cm. It was cleared from Figure (3) that the prepared PEG3 nanofibers exhibited randomly oriented, beads-free, smooth and uniform nanofibers with diameter of 400 nm at 25 cm. By studying the effect of changing the voltage value from 15 to 30KV, the obtained SEM images appeared in Figure (4) showed that the nanofibers diameter enhanced to reach 362 nm with smooth and uniform fibers produced at 20 kV. Figure (5) showed the effect of changing the flow rate of solution from electrospinning needle ranged from 0.75 to 1.5 ml/h, the SEM images revealed that the best PEG3 nanofibers were at 1 ml/h as the nanofibers had diameter of 362 nm with uniform fibers. Table (2) shows the effect of different electrospinning parameters on the obtained PEG3 nanofiber diameter. From Table (2), the optimum values for the electrospinning parameters to obtain PEG3 nanofibers with less average diameter are flow rate of 1ml/h, Voltage of 25 kV and Needle-to-collector distance of 25 cm.
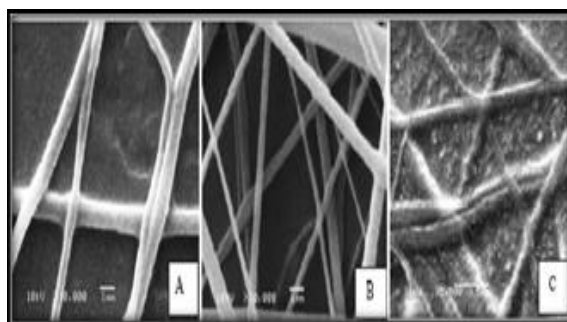

Fig.2. SEM for PVA/CMC/PEG nanofibers with magnification factor 10000 with electrospinning condition: flow rate= 0.5 ml/h, Voltage = 25 kV, needle-to-collector distance = 20 cm. (A): PEG1, (B): PEG2, (C) PEG3
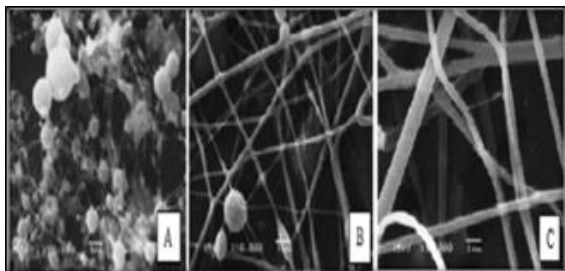
Fig.3. Effect of changing the needle-to-collector distance with flow rate= 1 ml/h, Voltage = 25 kV, On the morphology of PEG3. A): 10 cm, B) 15 cm, C) 20 cm, D) 25 cm
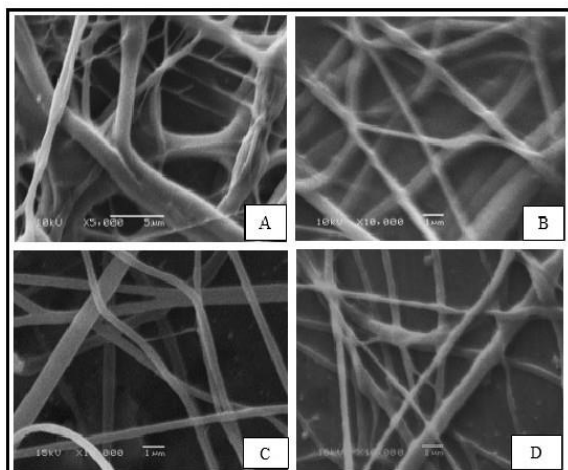

Fig.4. Effect of changing the voltage with flow rate= 1 ml/h, distance = 25 cm, On the morphology of PEG3. A): 15 kV, B) 20 kV, C) 25 kV, D) 30 kV
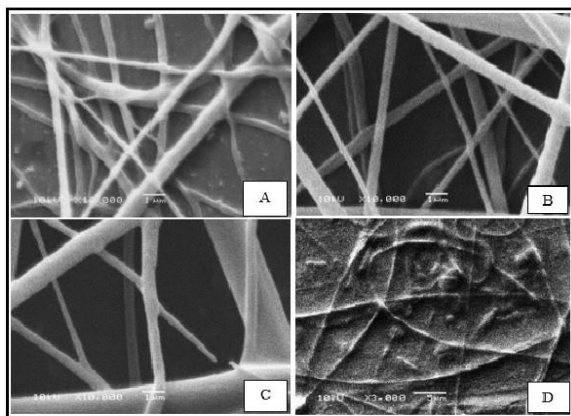

Fig.5. Effect of changing the flow rate with voltage= 25 kV, distance = 25 cm, On the morphology of PEG3. A): 0.75 ml/h, B) 1 ml/h, C) 1.5 ml/h

3.2. Study of the thermal stability of PEG3 membrane using TGA:

The thermal stability of the PVA/CMC/PEG (PEG3) nanofiber membrane was investigated by TGA and the results are presented in Figure (6). The sample was tested in the temperature range from ambient temperature up to 600oC under Nitrogen flow gas. The main phenomenon of the degradation pattern of

the sample follow two stages of weight losses, at first stage the loosely bond water goes out and the PEG3 polymeric chain mainly degraded in second stage. The degradation pattern summarize that the sample lost about 7 % at 115 ℃ in the first stage, and lost 93 % after reaching 550 ℃ as second stage of polymer degradation.

Table2. The effect of different electrospinning parameters on the obtained PEG3 nanofiber diameter.

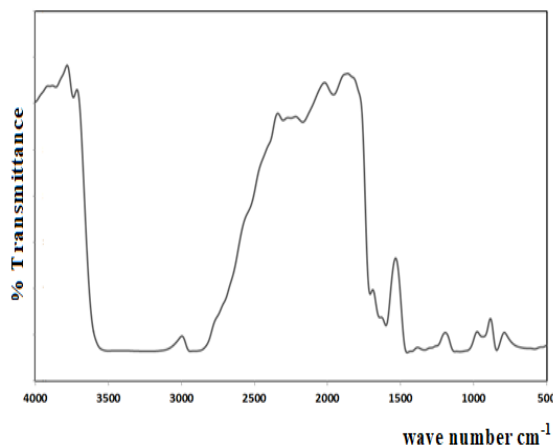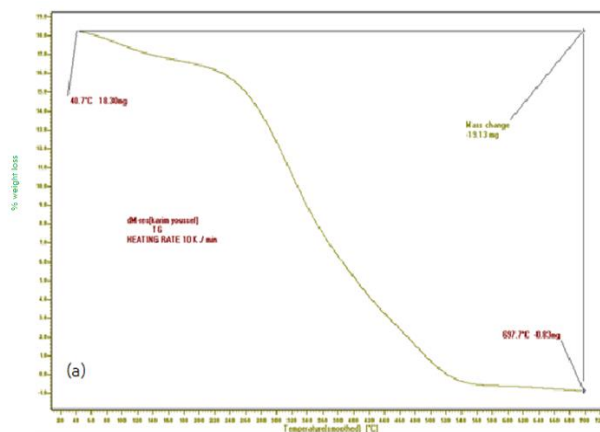| Flow rate (ml/h)	Voltage (kV)	Needle-to-collector distance (cm)	Fiber Diameter (nm)
1	15	10	685
1	15	15	415
1	15	20	460
1	15	25	400
1	20	25	502
1	25	25	362
1	30	25	702
0.75	25	25	520
1.5	25	25	810

Fig.6. (a): TGA curve for PEG3 nanofiber membrane at 600 ℃
(b): DTG curve for PEG3 nanofiber membrane

3.3. FTIR spectroscopy of Nano fiber membrane:

A good additional information about the structure of nanofiber membrane was found by FTIR spectra. Figure (7) showed PEG3 nanofiber spectra with scanning range 4000-500 cm-1. The broad band at 3510 cm-1 observed for O-H stretching vibration, intermolecular H- bonding from PVA & CMC & PEG. At 2938-2903 cm-1 was the CH symmetric stretching of methyl and propyl group (CH2- CH3). 2304 cm-1was for C-O bond and the peak appeared at 2251-2169 cm-1 was for C=C stretching of alkenes molecules, while C-H at 1959 cm-1. The C=O stretching band for the sample appeared at 1708cm-1 , at 1600 -1642 cm-1 C-O stretching band observed .The peak at 1456 cm-1 correspond to the C-H stretching band in the aliphatic structure of the polymer. A weak C-O stretching band of polyvinyl alcohol at 1129 cm-1. The stretching vibration of R-O-R group appears at 1093 cm-1 for PEG O-H band observed at 900-950 cm-1, and at 842 cm-1 the CH2 broad band appeared.
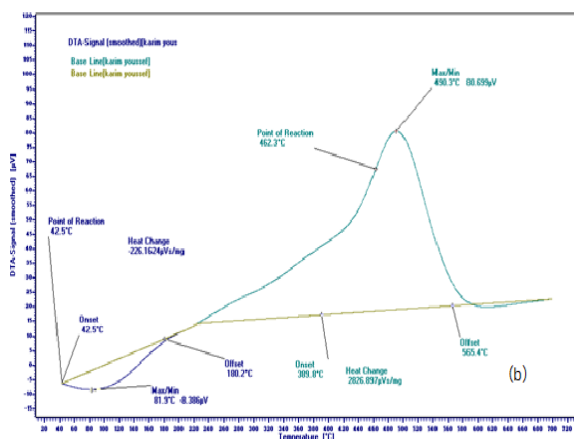

Fig.7. FTIR spectra of PEG3 nanofiber membrane

3.4. Hydrophobicity Analysis:

One of the important measurement for polymeric membranes is Water contact angle (WCA) to assess the water absorption and swelling properties of the membrane. Figure 8 showed the PVA/CMC/PEG nanofiber membranes contact angle between the electro spun membrane and the deionized water to determine the hydrophobicity of the nanofiber membrane. The WCA was 45.35. This value was evidence that the membrane had hydrophilic properties. Treatment with cross linking is necessary if it will use in waste water applications.
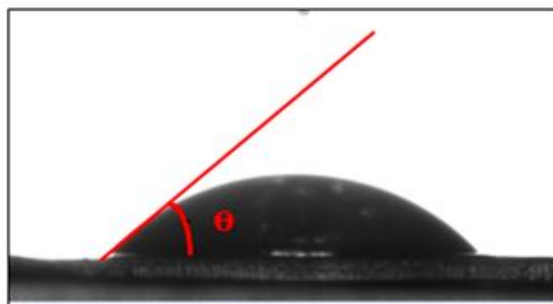

Figure (8): Contact angle of PEG3 nanofiber membrane

4. Conclusion

The electrospinning of PVA blended with CMC with different doses of PEG were carried out using distilled water as a solvent. The electro spun of PVA/CMC (PEG0) solution without any addition of PEG resulted in a nanofiber with beads formation. The additions of PEG with different concentration 7.5, 8.75, 10 % w/v respectively, (PEG1, PEG2 and PEG3) were tested to produce free beads nanofibers. The PEG1 and PEG2 gave nanofibers with beads formation, while the PEG3 gave uniform, regular and smooth nanofibers. The optimum condition for producing randomly oriented, bead-free, smooth and uniform nanofibers found to be 25 kV, 25 cm and 1 ml/h with average diameter of electro spun nanofibers 362 nm. The TGA analysis of the PVA/CMC/PEG (PEG3) nanofiber membrane was investigated and the results explained that the sample lost about 7 % at 115℃ in the first stage, and lost 93 % after reaching 550℃ as second stage of polymer degradation. The Water Contact Angle test was carried out to assess the water absorption and swelling properties of the fibers and it found to be 45.35. This value was evidence that the fibers had hydrophilic properties. Treatment with cross linking is necessary if the fibers need to be used in waste water applications.

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