



## CHAPTER V

### FABRICATION OF $\alpha$ -CHITIN WHISKER-REINFORCED POLY(VINYL ALCOHOL) NANOCOMPOSITE NANOFIBERS BY ELECTROSPINNING

#### 5.1 ABSTRACT

The present contribution reports, for the first time, the successful fabrication of  $\alpha$ -chitin whisker-reinforced poly(vinyl alcohol) (PVA) nanocomposite nanofibers by electrospinning. The  $\alpha$ -chitin whiskers were prepared from  $\alpha$ -chitin flakes from shrimp shells by acid hydrolysis. The as-prepared chitin whiskers exhibited the length in the range of 231 to 969 nm and the width in the range of 12 to 65 nm, with the average length and width being about 549 and 31 nm, respectively. Successful incorporation of the chitin whiskers within the as-spun PVA/chitin whiskers nanocomposite nanofibers was verified by infrared spectroscopic and thermogravimetric methods. The incorporation of chitin whiskers within the as-spun nanocomposite fiber mats increased the Young's modulus by about 4 to 8 times over that of the neat as-spun PVA fiber mat.

#### 5.2 INTRODUCTION

Nanocomposites are a class of composites with at least one phase having a dimension in the neighborhood of 1-1000 nm. As most of the present-day nanofillers used to prepare nanocomposites with synthetic polymeric materials are inorganic [1], their processability, biocompatibility, and biodegradability are much more limited than those of naturally organic ones. In nature, a large number of animals and plants synthesize extra cellular high-performance skeletal biocomposites consisting of a matrix reinforced by fibrous biopolymers [2]. Cellulose is a classical example where the reinforcing elements exist as whisker-like microfibrils that are biosynthesized and deposited in a continuous manner [3].

Favier et al. [4] was the first to prepare cellulose whiskers from tunic of tunicate *Microcosmus fulcatus* by acid hydrolysis (i.e. tunicin whiskers) and use

them as reinforcing nanofillers in a terpolymer consisting mainly of styrene and butyl acrylate (i.e. poly(S-co-BuA) latex). Since then, tunicin whiskers have been used as reinforcing fillers in poly( $\beta$ -hydroxyoctanoate; PHO) [5], poly(hydroxyalkanoate; PHA) [6], plasticized maize starch [7], and poly(oxyethylene) [8]. Whiskers from other polysaccharides, such as starch [9] and chitin [10], can also be prepared. Unlike tunicin whiskers which can only be prepared by hydrolysis in a strong sulfuric acid ( $H_2SO_4$ ) solution [4-8, 10], both starch and chitin whiskers can be prepared by hydrolysis in a hydrochloric acid (HCl) solution [9, 10].

Chitin is a high molecular weight biopolymer predominantly found in exoskeleton shells of arthropods as well as the internal flexible backbone of cephalopods. Chemically, chitin molecules consist of *N*-acetyl-*D*-glucosamine units. Chitin is non-toxic, odorless, biocompatible with living tissues, and biodegradable [11]. Chitin whiskers have been successfully prepared from crab shells [10, 12, 13], squid pens [14], and tubes of *Riftia pachyptila* tube worms [15]. Regardless of the chitin sources, the commonly-used hydrolytic condition to arrive at chitin whiskers is 3 N HCl at the boil for 90 min under vigorous stirring [10, 11, 15]. Recently, we reported successful preparation and characterization of nanocomposite films of poly(vinyl alcohol) (PVA)/ $\alpha$ -chitin whiskers and chitosan/ $\alpha$ -chitin whiskers with or without heat treatment [16]. The whisker content ranged between 0 and 29.6 wt.%.

In recent years, electrostatic spinning or electrospinning has been viewed as a simple and versatile method for fabricating ultrafine fibers with diameters in the nanometer to sub-micrometer range [17]. The kernel of the technique is the use of a high electrostatic potential applying to a spinning liquid across a charged nozzle and a grounded screen collector. At right conditions, a charged stream of the spinning liquid was ejected and ultimately a mat of non-woven fibers is collected on the collector [17]. Various materials have successfully been electrospun into ultrafine fibers. Among these, there have been increasing reports about the preparation of nanocomposite electrospun fibers as novel functional materials. Some of the nanomaterials that were incorporated into electrospun polymeric matrices are layered nanosilicate [18], calcium carbonate, hydroxyapatite, or silver nanoparticles [19], cadmium sulfide nanorods [20], and graphite nanoplatelets [21].

Apparently, these nanomaterials are inorganic in nature. It is therefore the main objective of the present contribution to fabricate nanocomposite electrospun fibers based on the PVA/ $\alpha$ -chitin whiskers system. PVA was chosen as the matrix due mainly to its excellent film-forming and electrospinning ability [16, 22]. The as-electrospun nanocomposite PVA/ $\alpha$ -chitin whiskers fiber mats were characterized for their chemical, thermal, and mechanical integrity. The results were also compared with those obtained for the corresponding as-cast nanocomposite PVA/ $\alpha$ -chitin whiskers films.

### 5.3 EXPERIMENTAL

#### 5.3.1 Preparation of $\alpha$ -chitin whiskers

$\alpha$ -Chitin whiskers were prepared from chitin flakes derived from shells of *Penaeus merguensis* shrimps [Seafresh Co., Ltd. (Thailand)]. Chitin flakes were first hydrolyzed with 3 N hydrochloric acid (HCl) under vigorous stirring with refluxing at 120°C for 6 hrs. The ratio of HCl to chitin flakes was 30 cm<sup>3</sup>·g<sup>-1</sup>. After acid hydrolysis treatment, the chitin whisker suspension was diluted with distilled water, followed by centrifugation at 10,000 rpm for 10 min. The suspension was then transferred to a dialysis bag and dialyzed in distilled water for 2 days. The solid content of the as-prepared chitin whisker suspension was measured gravimetrically to be about 5.1 wt.%.

#### 5.3.2 Preparation of nanocomposite fiber mats and films

Poly(vinyl alcohol) (PVA) powder ( $M_w = 75,000$  Da and degree of hydrolysis (DH) = 98%; Fluka, USA) was first dissolved in warm distilled water (85°C) under slight stirring for 3 hrs to obtain a PVA solution at a fixed concentration of 10 wt.%. Varying amount of the as-prepared chitin whisker suspension (between 1 and 10 g) was well mixed with 20 g of the as-prepared PVA solution. Distilled water may be added into the mixture so that the total weight of the mixture was fixed at about 30 g. Table 5.1 summarizes the composition of each of the as-prepared mixtures. The final concentrations of both PVA and chitin whiskers

in the mixtures were calculated and are also summarized in Table 1. The weight content of chitin whiskers with respect to the weight of PVA (i.e. the CW/PVA ratio) ranged between 0 and 25.4%. The PVA/chitin whiskers mixtures were continuously stirred over night prior to further fabrication.

To prepare nanocomposite fiber mats, each of the as-prepared PVA/chitin whiskers mixtures was placed in a 50-ml plastic syringe. A blunt-end stainless-steel gauge 20 needle (i.e. outside diameter = 0.91 mm) was used as the nozzle. The tilt angle of the syringe and the needle was 45° from a horizontal baseline to ascertain a constant presence of a liquid droplet at the tip of the nozzle. A sheet of aluminum foil on a plastic backing was used as the screen collector. A Gamma High Voltage Research DES30PN/M692 power supply was used to generate a high DC potential across the needle (connected to the positive emitting electrode) and the screen collector (connected to the grounding electrode). The applied potential was fixed at 15 kV over a fixed collection distance between the tip of the nozzle and the screen collector of 15 cm. The feed rate of the solutions for most experiments was controlled by means of a Kd Scientific syringe pump at 3 ml/hr. The collection time was also fixed at 12 hrs. The as-spun fiber mats were kept in room condition for one day prior to further characterization.

To prepare nanocomposite films, each of the as-prepared PVA/chitin whiskers mixtures was poured onto a glass petridish by fixing the volume of the mixture at about 15 ml. The sample was let dry in room condition for 3 days prior to further characterization.

### 5.3.3 Characterization

Morphological appearance of the as-prepared  $\alpha$ -chitin whiskers was observed using a JEOL JEM-200CX transmission electron microscope (TEM). Samples of the chitin whiskers were prepared from a drop of a dilute chitin whisker suspension which was deposited and let dry on a formvar grid. The average dimension of the whiskers was determined from TEM images, from which at least 120 whiskers were measured for their length and width using image-analytical software (SemAfore 4.0). The viscosity and the conductivity of the PVA/chitin

whiskers mixtures were measured at room condition using a Brookfield DV-III programmable viscometer and a Orion 160 conductivity meter, respectively. Morphological appearance of the as-spun fiber mats was observed using a JEOL JSM-5200 scanning electron microscope (SEM), while the average diameter of the fibers within the mats was determined from SEM images, from which at least 300 fibers were measured for their diameters using the same image-analytical software.

Both the as-prepared neat PVA and PVA/chitin whiskers nanocomposite fiber mats and films were characterized for their chemical, thermal, and mechanical integrity. A Thermo Nicolet Nexus 671 Fourier-transformed infrared spectroscope (FT-IR) was used to investigate the chemical functionalities of the as-prepared fiber mat and film samples. The measurements were carried out at 32 scans and a resolution of  $4\text{ cm}^{-1}$ . A TGA 2950 DuPont thermogravimetric analyzer (TGA) was used to investigate the thermal stability of the samples and to verify the existence of chitin whiskers within the nanocomposite samples. A sample of about 3 to 10 mg was placed in a platinum pan. The scanning range was 30 to  $600^{\circ}\text{C}$  using a heating rate of  $10^{\circ}\text{C}\cdot\text{min}^{-1}$  in nitrogen atmosphere. A Lloyd LRX universal tester was used to determine the tensile strength, modulus, and elongation at break of the samples, according to ASTM D882 standard test method. The dimension of the samples was  $10 \times 120\text{ mm}^2$ , with the thickness ranging between 45 and  $215\text{ }\mu\text{m}$ . The gauge length and the crosshead speed were 50 mm and  $20\text{ mm}\cdot\text{min}^{-1}$ , respectively. The reported values were averages from 5 measurements.

## 5.4 RESULTS AND DISCUSSION

### 5.4.1 Morphological appearance and sizes of $\alpha$ -chitin whiskers

Selected TEM images of chitin whiskers prepared from a dilute suspension of chitin whiskers by acid hydrolysis of chitin flakes are shown in Figure 5.1. The as-prepared chitin whisker suspension exhibited a colloidal behavior, due to the presence of the positive charges ( $\text{NH}_3^+$ ) on the surface of whiskers which resulted from the protonation of amino groups of chitin in acidic conditions [10]. The suspension contained chitin fragments consisting of both individual microcrystals

and aggregated microcrystals. The chitin fragments consisted of slender rods that exhibited broad distribution in both of their length and width (see Figure 5.2). The width of these chitin fragments ranged from 12 to 65 nm, while the length ranged from 231 to 969 nm. Statistic evaluation of the results suggested the average width and length of these whiskers to be about 31 and 549 nm, respectively. The aspect ratio (i.e. average length to width ratio;  $L/D$ ) of these whiskers was calculated to be about 17.7. These dimensions are in line with the reported values for chitin whiskers obtained from crab shells ( $L = 50\text{--}300$  nm and  $D = 6\text{--}8$  nm [10];  $L = 100\text{--}600$  nm and  $D = 4\text{--}40$  nm [12]; and  $L = 100\text{--}650$  nm and  $D = 10\text{--}80$  nm [13]), squid pens ( $L = 50\text{--}300$  nm and  $D = \sim 10$  nm [14]), and shrimp shells ( $L = 150\text{--}800$  nm and  $D = 5\text{--}70$  nm [16]), but are much shorter in length when compared with the chitin whiskers from *Riftia* tubes ( $L = 500$  nm to 10 mm and  $D = \sim 18$  nm [15]). The solid content of the as-prepared chitin whisker suspension was about 5.1 wt.%.

#### 5.4.2 Characterization of PVA/chitin whiskers mixtures and resulting nanocomposite fiber mats and films

Prior to electrospinning, the as-prepared PVA/chitin whiskers mixtures were measured for their viscosity and conductivity. The results are shown in Figure 5.3. The viscosity of the mixtures increased with the addition of an increasing amount of chitin whiskers (i.e. from about 180 cP for neat PVA solution to about 250 cP for the mixture containing 10 g of the chitin whisker suspension), while the conductivity of the mixtures ranged between about 850 and 910  $\text{mS}\cdot\text{cm}^{-1}$ .

Electrospinning of the as-prepared PVA/chitin whiskers mixtures was rather easy. Figure 5.4 shows selected SEM images of the as-spun fibers from neat PVA solution and PVA/chitin whiskers mixtures (see Table 5.1 for detailed information about the composition of the spinning dopes). All of the as-spun fibers were cross-sectionally round; however, a slight difference between the fibers obtained from neat PVA solution and PVA/chitin whiskers mixtures is evident. Clearly, the as-spun fibers from neat PVA solution were smooth, while those from PVA/chitin whiskers mixtures exhibited a combination of smooth and bead-on-string morphology. Statistical analysis of the fiber diameters (see Figure 5.5) revealed that the average diameter of fibers from neat PVA solution was about 175 nm. Upon a

small addition of the chitin whiskers, the average fiber diameter increased to about 185 and 210 nm for fibers from the mixtures containing 1 and 2 g of the chitin whisker suspension and leveled off with the average fiber diameter ranging between 213 and 218 nm with further increase in the chitin whisker suspension content.

Attempts to reveal the incorporation of chitin whiskers within the as-spun fibers from PVA/chitin whiskers mixtures by TEM were unsuccessful, due mainly to the difficulty in the sample preparation. FT-IR and TGA were alternative methods in order to verify the successful incorporation of chitin whiskers within the as-spun fibers from PVA/chitin whiskers mixtures. Figure 5.6 shows FT-IR spectra of as-spun fibers from neat PVA solution and PVA/chitin whiskers mixtures as well as those of corresponding as-cast films. Evidently, the absorption peaks at  $1440\text{ cm}^{-1}$  ( $\text{CH}_2$  bending) and  $858\text{ cm}^{-1}$  ( $\text{CH}_2$  rocking) characteristic to PVA were obvious in all of the spectra of the as-spun fiber mats and the as-cast films [16], while the characteristic amide I peak at  $1655\text{ cm}^{-1}$  ( $-\text{CONH}-$ ) was apparent in all of the spectra of the as-spun fiber mats and the as-cast films from PVA/chitin whiskers mixtures [16]. It is evident that the relative intensity of these characteristic peaks increased with an increase in the content of the corresponding component and that no additional peaks other than those characteristic to either PVA or chitin were observed in these spectra.

Thermogravimetric analysis for all of the as-spun fiber mats and the as-cast films was used as an alternative means for verifying the existence of chitin whiskers within the as-spun fiber mats and the as-cast films from PVA/chitin whiskers mixtures. Figure 5.7 shows the derivative curves of the TGA thermograms for all of the as-spun fiber mats and the as-cast films as well as the as-prepared chitin whiskers. It should be noted that the moisture content for all of the as-spun fiber mats and the as-cast films was within 3 to 5 wt.%, as suggested by their respective TGA thermogram. According to Figure 5.7, both neat as-spun PVA fiber mat and as-cast PVA film exhibited two steps in the loss of their mass at about  $320$  and  $440^\circ\text{C}$ , respectively, while the as-prepared chitin whiskers exhibited only one step in the loss of their mass at about  $390^\circ\text{C}$ . Clearly, all of the as-spun fiber mats and the as-cast films from PVA/chitin whiskers mixtures exhibited an extra step in the loss of

their mass at about 360 to 380°C from that of the neat PVA, which confirmed the presence of chitin whiskers within the samples. Evidently, the weight loss associated with the thermal degradation of chitin whiskers increased with increasing amount of the chitin whisker suspension that was added in the PVA/chitin whiskers mixtures.

#### 5.4.3 Mechanical properties of nanocomposite fiber mats and films

Both the FT-IR and TGA techniques confirmed the incorporation of chitin whiskers within the as-spun PVA/chitin whiskers fiber mats. These mats were subsequently tested for their mechanical integrity in terms of the tensile strength, Young's modulus, and the elongation at break by comparing with that of the corresponding as-cast films.

Figures 5.8 and 5.9 show mechanical properties as well as thickness of the as-spun fiber mats and the as-cast films, respectively. As shown in Table 5.1, the weight content of chitin whiskers with respect to the weight of PVA (hereafter, the CW/PVA ratio) ranged between 0 and 25.4%. The thickness of the as-spun fiber mats ranged between about 100 and 215  $\mu\text{m}$ . The tensile strength of the as-spun fiber mats increased from that of the neat PVA fiber mat (i.e.  $4.3 \pm 0.7$  MPa) with initial addition of the chitin whiskers to reach a maximum value (i.e.  $5.7 \pm 0.6$  MPa) at the CW/PVA ratio of about 5.1% and decreased with further increase in the chitin whisker content. Interestingly, incorporation of the chitin whiskers increased the modulus of the nanocomposite fiber mats from that of the neat PVA fiber mat appreciably (i.e. about 4 to 8 times), at the expense of the elongation at break in which it decreased rather monotonously with increasing chitin whisker content. Specifically, the Young's modulus increased from that of the neat PVA fiber mat (i.e.  $35.3 \pm 4.8$  MPa) to reach a maximum value (i.e.  $278 \pm 41$  MPa) at the CW/PVA ratio of about 15.2% and decreased thereafter.

According to Figure 5.9, the thickness of the as-cast films ranged between about 45 and 70  $\mu\text{m}$ . The tensile strength of the as-cast films increased from that of the neat PVA film (i.e.  $20.1 \pm 2.0$  MPa) with the addition of the chitin whiskers to reach a maximum value (i.e.  $45.2 \pm 3.6$  MPa) at the CW/PVA ratio of about 17.7%. On the contrary to the tensile strength, the elongation at break of the



nanocomposite films decreased rather monotonously from that of the neat PVA film with increasing chitin whisker content. Similar to the nanocomposite fiber mats, incorporation of the chitin whiskers increased the modulus of the nanocomposite films from that of the neat PVA film. Specifically, the Young's modulus increased monotonously from that of the neat PVA fiber mat (i.e.  $48.4 \pm 11.6$  MPa) to reach a maximum value (i.e.  $1\ 500 \pm 450$  MPa) at the maximum CW/PVA ratio of about 25.4% investigated in this work. Based on the results observed for both the as-spun and the as-cast nanocomposite samples, it can be concluded that chitin whiskers are an effective reinforcing biological material.

The observed increase in the tensile strength of both the nanocomposite fiber mats and films with increasing chitin whisker content could be due to the interaction between PVA and chitin whiskers via hydrogen bonding. However, such interaction caused the nanocomposites to become more rigid with increasing whisker content, resulting in the observed decrease in the elongation at break. Moreover, the different behavior in the Young's modulus for the nanocomposite fiber mats and films in that, in the case of the nanocomposite fiber mats, the property value initially increased with the addition of chitin whiskers, reached a maximum at the CW/PVA ratio of about 15.2%, and decreased with further increase in the chitin whisker content, while, in the case of the nanocomposite films, the property value increased monotonously with the addition of the chitin whiskers and increasing chitin whisker content, could be due to the difference in the dimensionality of the two sample types. For the nanocomposite fiber mats, the observed decrease in the Young's modulus at the CW/PVA ratio of greater than about 15.2% could be due to the aggregation of chitin whiskers due to the confinement effect. On the other hand, such a confinement effect was not the factor for the nanocomposite films, unless the thickness of the films approaching the sizes of the nanosized reinforcing fillers.

The bulk density of the as-spun fiber mats and the as-cast films was calculated from both the mass and the volume of the samples. The bulk density of neat PVA fiber mat was about  $0.279 \pm 0.001$  g·cm<sup>-3</sup>, while that of neat PVA film was about  $1.006 \pm 0.006$  g·cm<sup>-3</sup>. Furthermore, the bulk density of all the as-spun

nanocomposite fiber mats ranged between 0.195 and 0.308 g·cm<sup>-3</sup>, while that of all the as-cast nanocomposite films ranged between 1.005 and 1.173 g·cm<sup>-3</sup>. In an engineering point of view, comparison of the mechanical properties of different materials is usually made by normalizing their property values with their respective density, i.e. the specific property values. Figure 8 shows the specific tensile strength and the specific Young's modulus for the as-spun fiber mats and the as-cast films. Clearly, normalization of the property values with the respective density of the samples did not affect the general conclusion of the obtained results, but it shows that the specific property values for both the as-spun fiber mats and the as-cast films now fell within the same range (especially in the case of the specific Young's modulus; see Figure 5.10b).

## 5.5 CONCLUSION

Chitin whisker-reinforced nanocomposite nanofibers were fabricated by electrospinning a mixture between an aqueous solution of poly(vinyl alcohol) (PVA) and a suspension of  $\alpha$ -chitin whiskers from chitin flakes from shells of *Penaeus merguensis* shrimps. The as-prepared  $\alpha$ -chitin whiskers consisted of slender rods having a broad distribution in both of their length and width. The average length and width of these whiskers were about 549 and 31 nm, respectively, with the average aspect ratio between the length and the width being about 18. Electrospinning of the PVA/chitin whiskers mixtures was straightforward. The average diameter of the as-spun nanocomposite nanofibers ranged between 185 and 218 nm, regardless of the whisker content. Incorporation of the chitin whiskers within the as-spun nanocomposite nanofibers was confirmed by infrared spectroscopic and thermogravimetric analyses. The tensile strength of the as-spun PVA/chitin whiskers nanocomposite fiber mats increased from that of the neat as-spun fiber mat (i.e.  $4.3 \pm 0.7$  MPa) with initial addition of the chitin whiskers to reach a maximum value (i.e.  $5.7 \pm 0.6$  MPa) at the chitin whiskers to PVA ratio of about 5.1% and decreased with further increasing the whisker content. Lastly, the presence of chitin whiskers within

the as-spun nanocomposite fiber mats increased the Young's modulus by about 4 to 8 times over that of the neat as-spun fiber mat.

## 5.6 ACKNOWLEDGEMENTS

The authors acknowledge partial support received from 1) the National Research Council of Thailand (NRCT), 2) Chulalongkorn University (through invention and research grants from the Ratchadapesek Somphot Endowment Fund), 3) the Petroleum and Petrochemical Technology Consortium [through a Thai governmental loan from Asian Development Bank (ADB)], and 4) the Petroleum and Petrochemical College (PPC), Chulalongkorn University. J. Junkasem acknowledges a doctoral scholarship received from the Royal Golden Jubilee PhD Program, the Thailand Research Fund (TRF).

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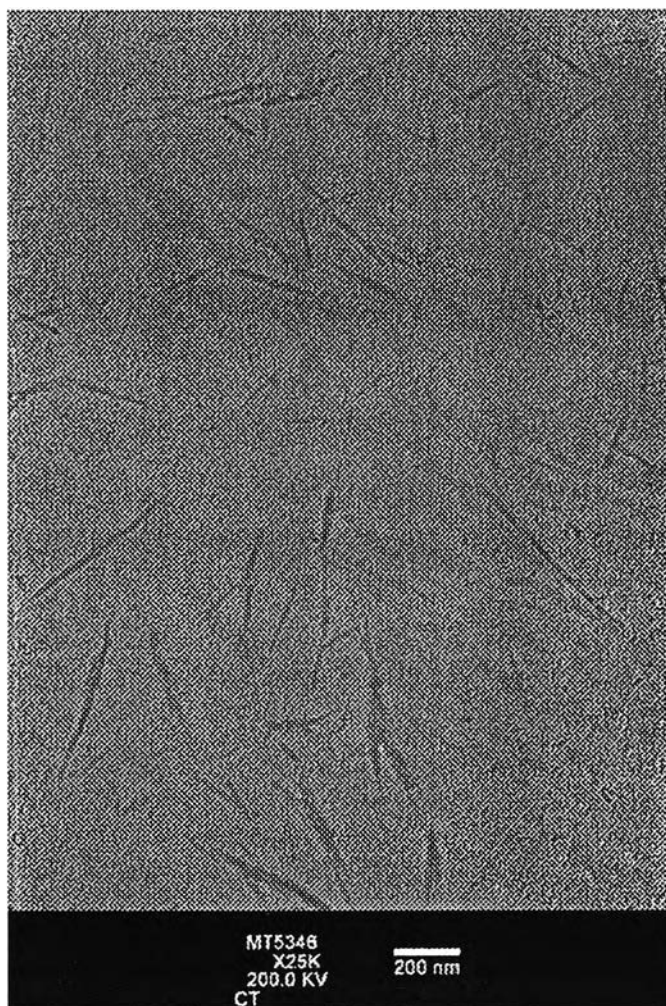
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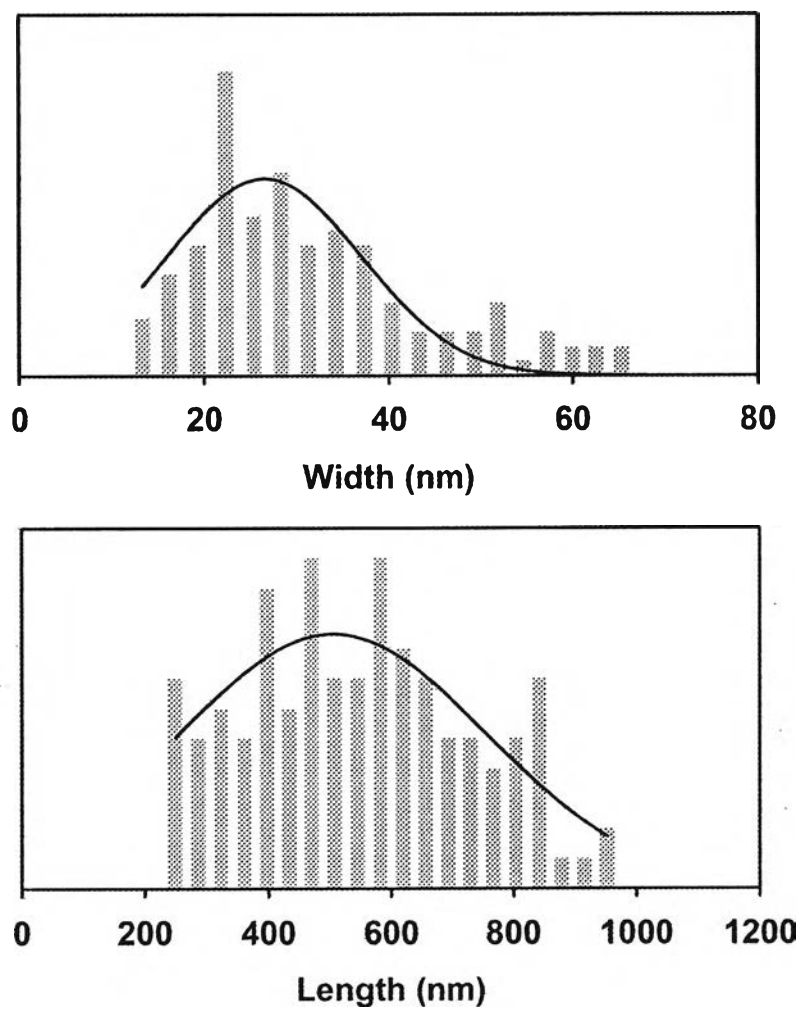
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**Table 5.1** Composition of PVA solution and PVA/chitin whiskers mixtures

Sample	Weight (g)			Concentration (wt.%)		CW/PVA ratio (%)
	PVA solution	Chitin whisker suspension	Distilled water	PVA	Chitin whiskers	
PVA	20.12	-	10.13	6.65	-	-
PVA + 1g CW	20.01	1.01	9.01	6.66	0.17	2.55
PVA + 2g CW	20.02	2.01	8.04	6.66	0.34	5.11
PVA + 3g CW	19.97	3.00	7.01	6.66	0.51	7.66
PVA + 4g CW	20.03	4.00	6.17	6.63	0.67	10.11
PVA + 5g CW	20.01	5.04	5.01	6.66	0.85	12.76
PVA + 6g CW	20.01	6.00	4.03	6.66	1.01	15.17
PVA + 7g CW	20.01	7.01	3.08	6.65	1.18	17.74
PVA + 10g CW	20.01	10.01	-	6.66	1.69	25.38

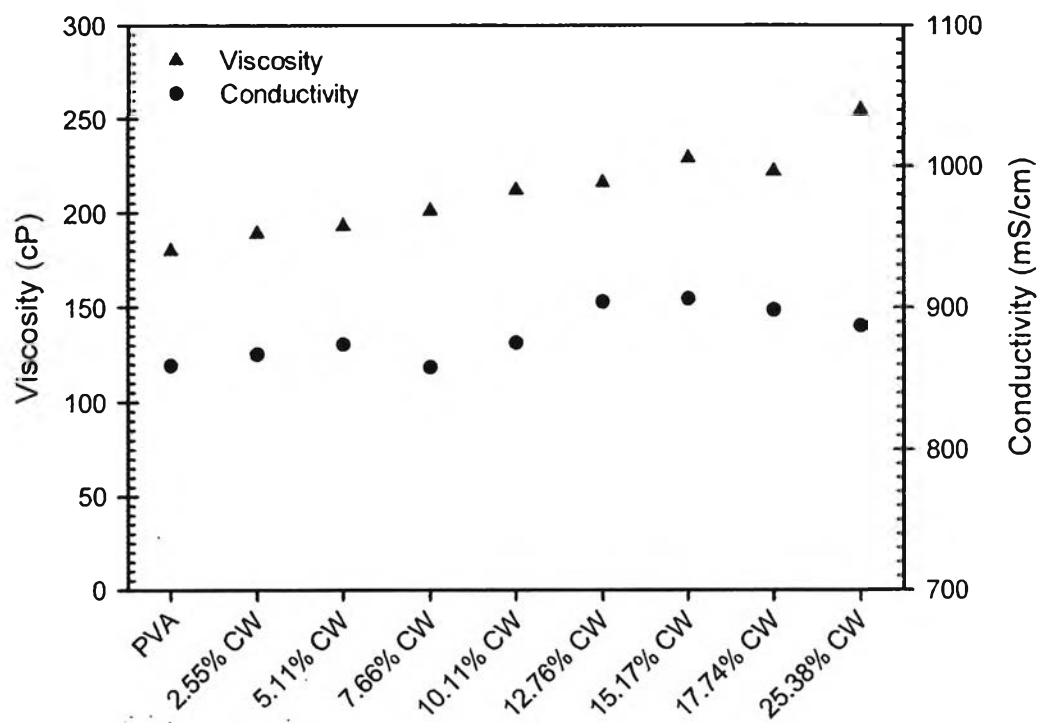


**Figure 5.1** Selected TEM images of chitin whiskers from dilute suspension of chitin whiskers prepared by acid hydrolysis of chitin flakes from shells of *Penaeus merguensis* shrimps.

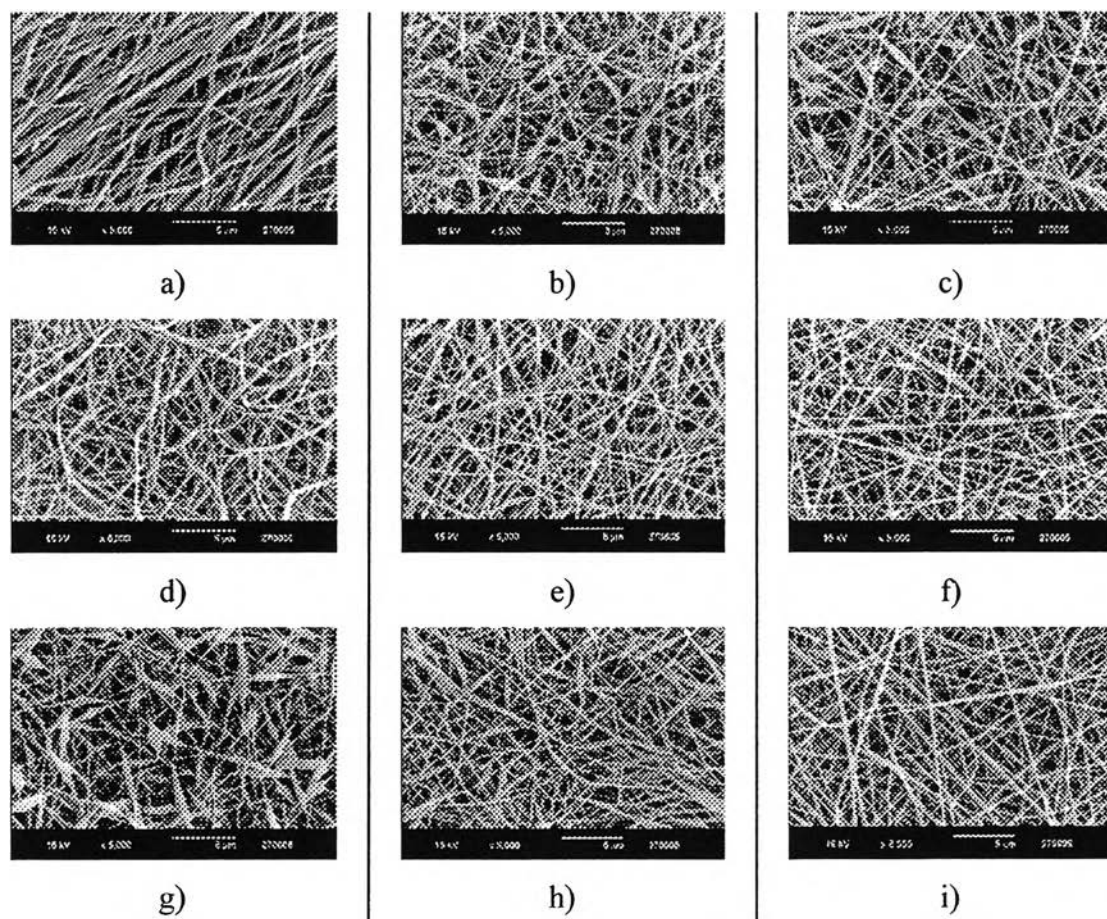


**Figure 5.2** The distribution of length and width of chitin whiskers from TEM.

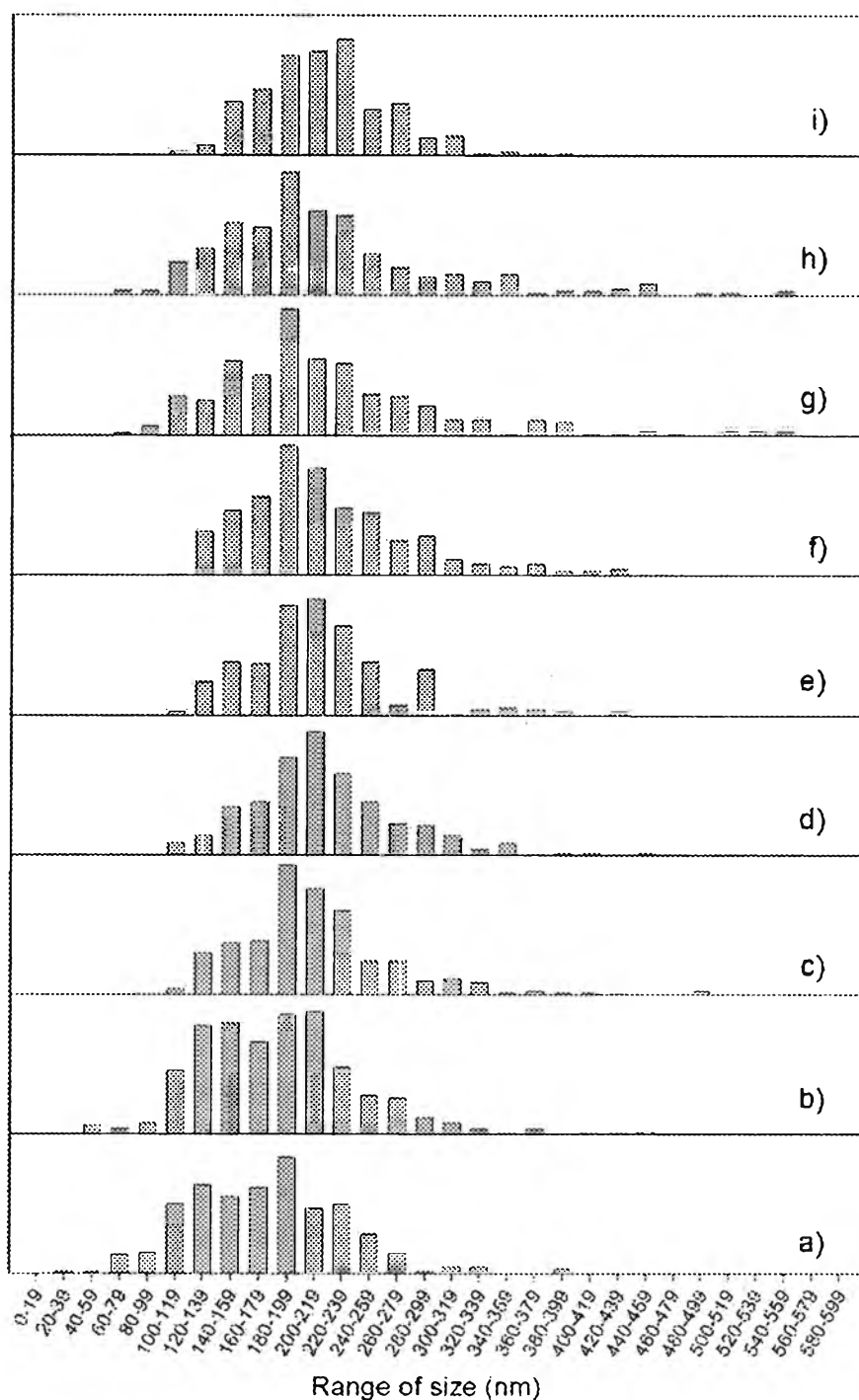




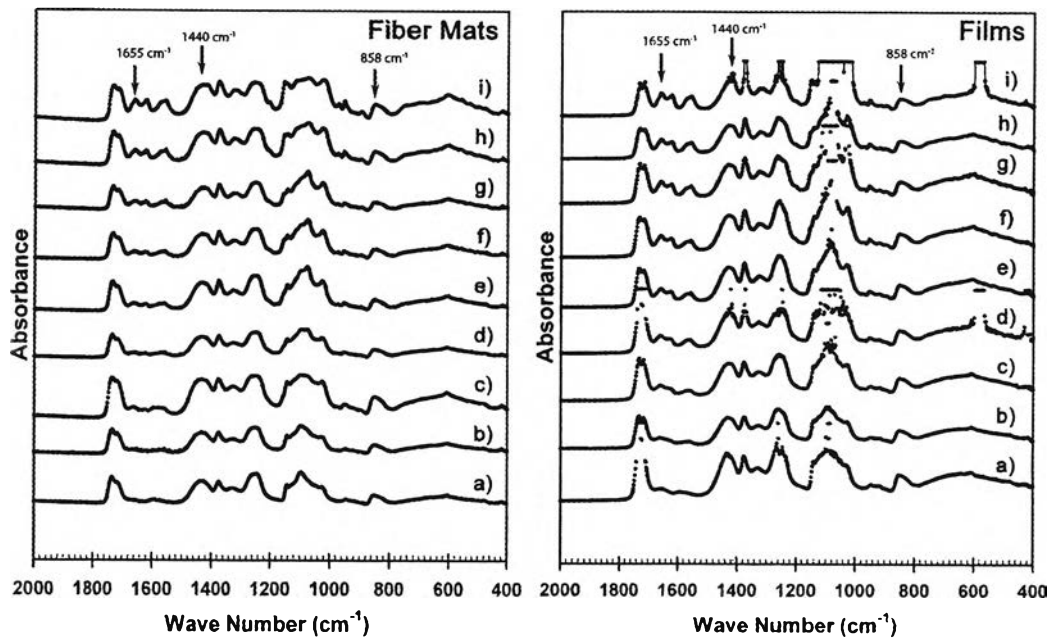
**Figure 5.3** Viscosity and conductivity of neat PVA solution and PVA/chitin whiskers mixtures.



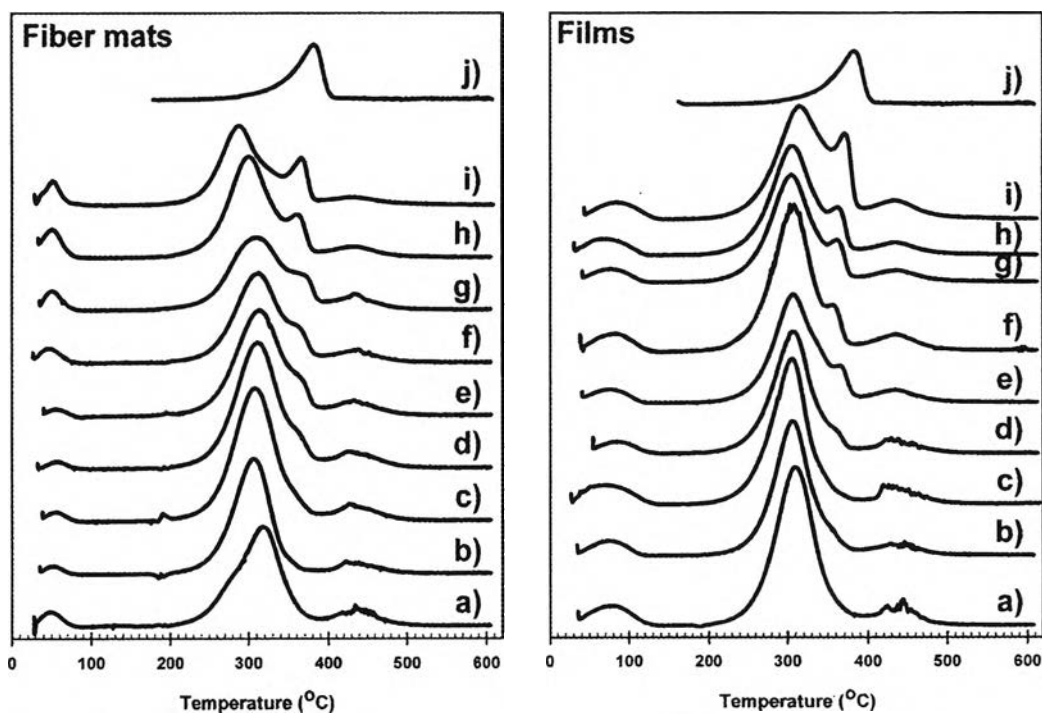
**Figure 5.4** Selected SEM images of electrospun fibers from a) neat PVA solution and PVA/chitin whiskers mixtures containing b) 2.55%, c) 5.11%, d) 7.66%, e) 10.11%, f) 12.76%, g) 15.17%, h) 17.74%, and i) 25.38% of chitin whiskers. The scale bar represents 5  $\mu\text{m}$ . The average fiber diameter of these fiber mats was 175, 185, 210, 216, 213, 216, 218, 216, and 214 nm, respectively.



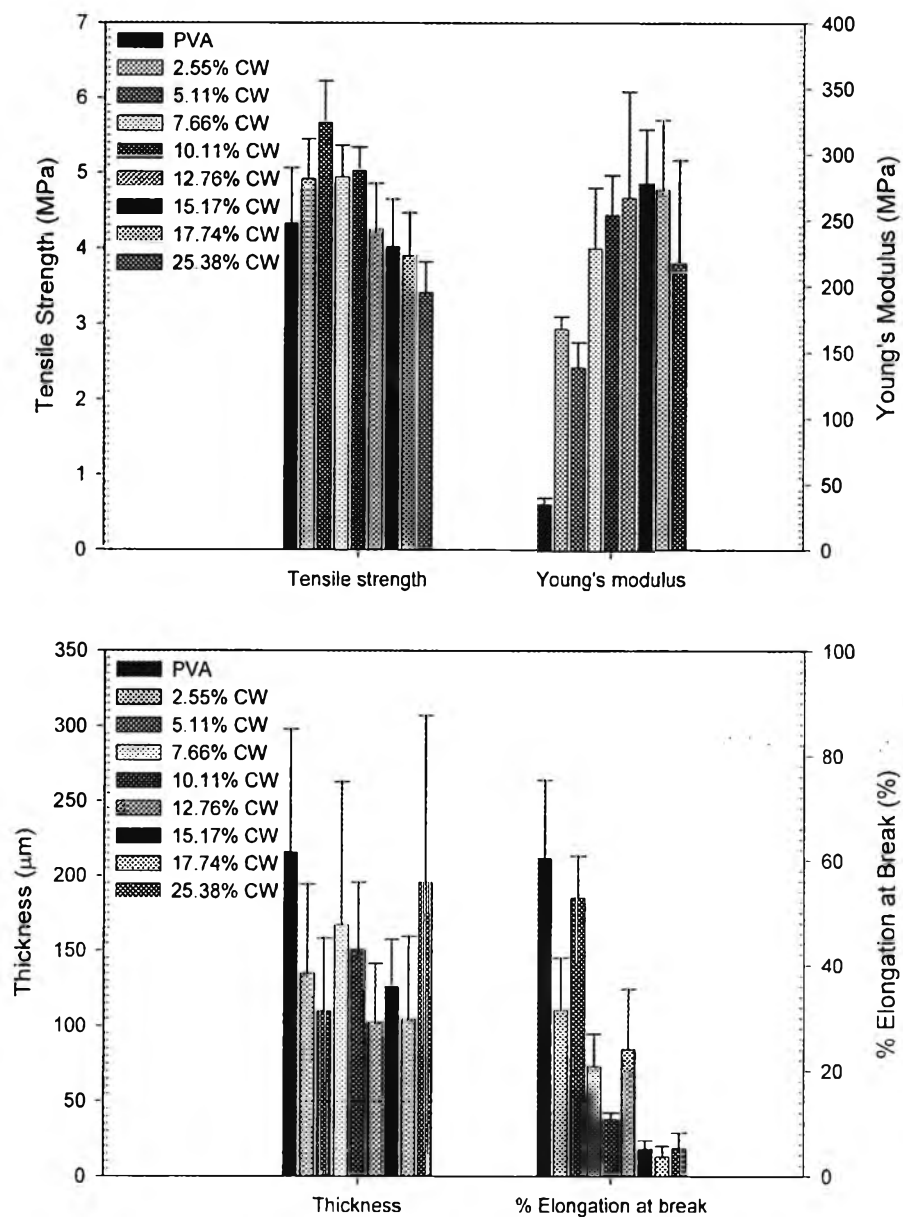
**Figure 5.5** The statistical analysis of diameter of as-spun nanofibers from a) neat PVA solution and PVA/chitin whiskers mixtures containing b) 2.55%, c) 5.11%, d) 7.66%, e) 10.11%, f) 12.76%, g) 15.17%, h) 17.74%, and I) 25.38% of chitin whiskers.



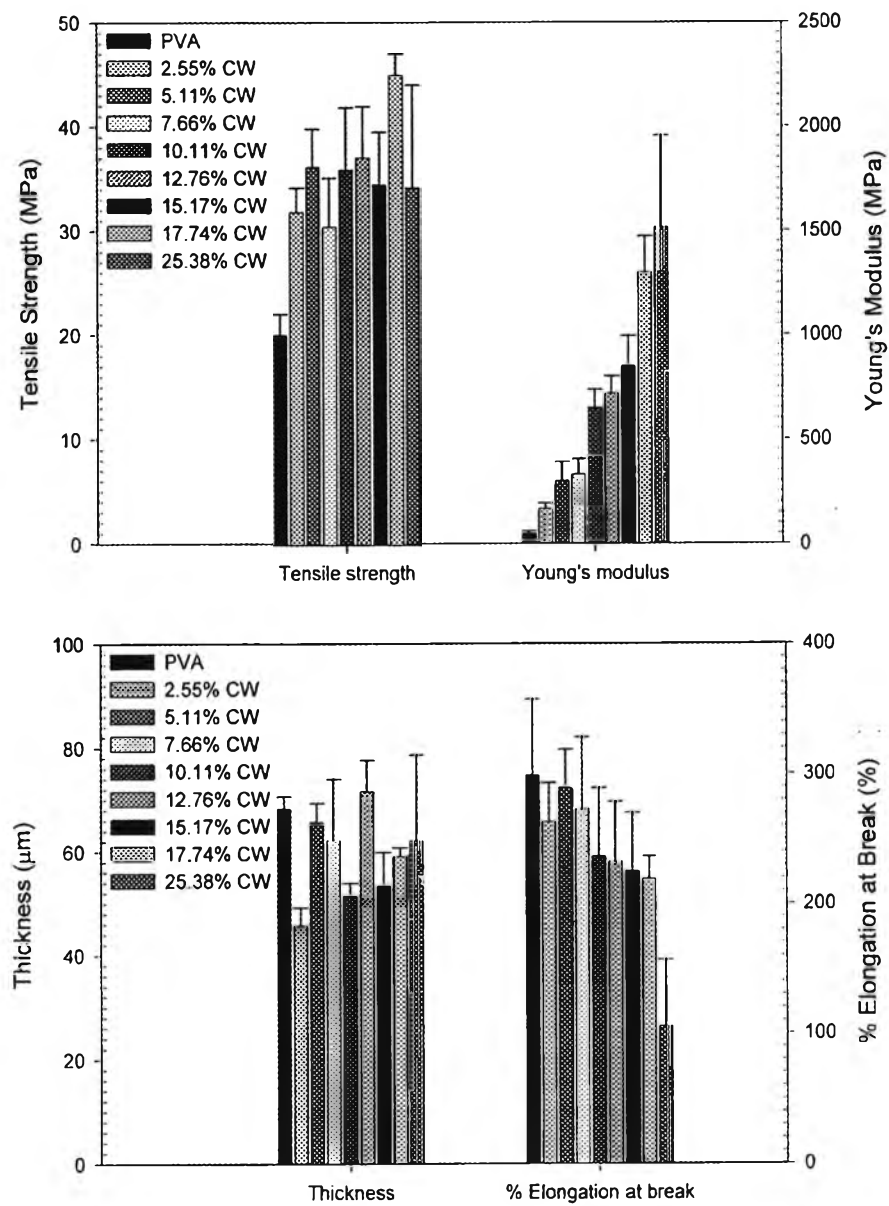
**Figure 5.6** FT-IR spectra of electrospun fiber mats and solution-cast films from a) neat PVA solution and PVA/chitin whiskers mixtures containing b) 2.55%, c) 5.11%, d) 7.66%, e) 10.11%, f) 12.76%, g) 15.17%, h) 17.74%, and I) 25.38% of chitin whisker.



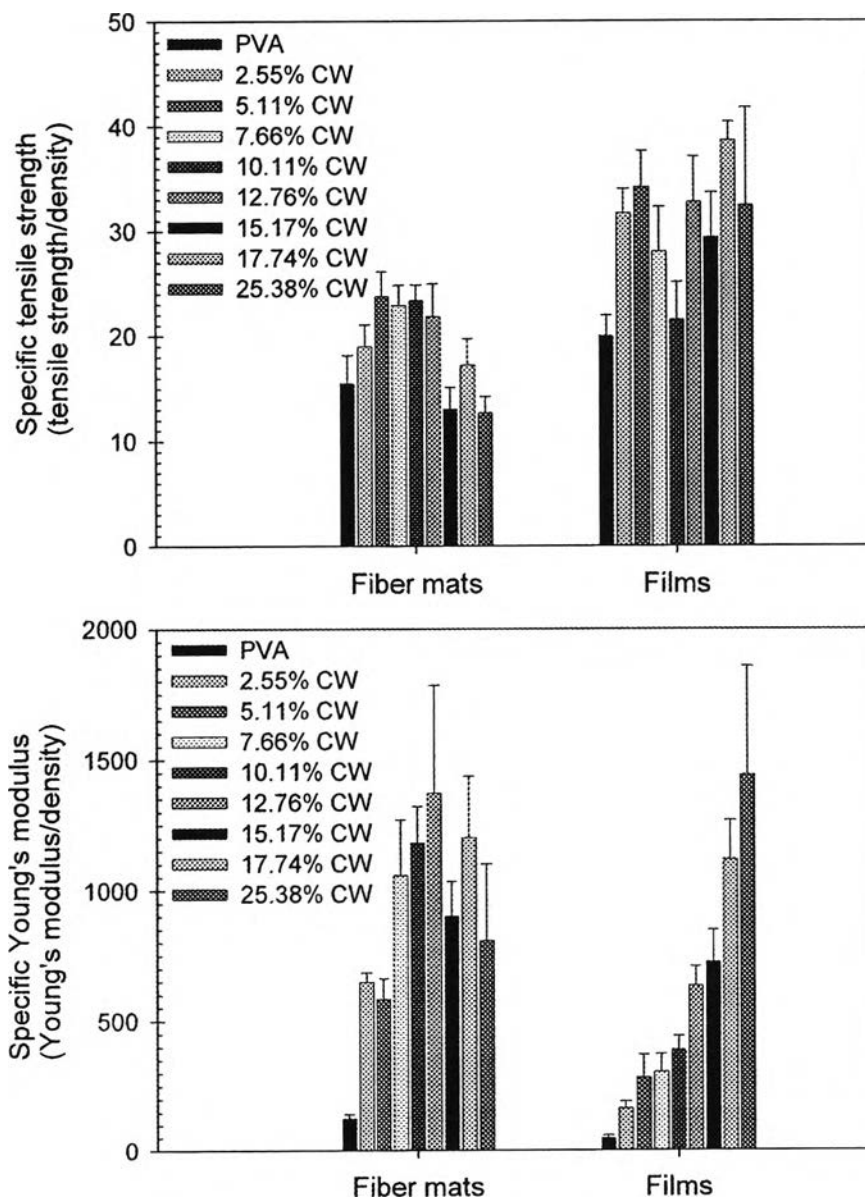
**Figure 5.7** Derivative TGA thermograms of electrospun fiber mats and solution-cast films from a) neat PVA solution and PVA/chitin whiskers mixtures containing b) 2.55%, c) 5.11%, d) 7.66%, e) 10.11%, f) 12.76%, g) 15.17%, h) 17.74%, and I) 25.38% of chitin whisker as well as j) as-prepared chitin whiskers.



**Figure 5.8** Tensile strength, Young's modulus, elongation at break, and thickness of electrospun fiber mats from neat PVA solution and PVA/chitin whiskers mixtures containing 2.55% to 25.38% of chitin whisker.



**Figure 5.9** Tensile strength, Young's modulus, elongation at break, and thickness of solution-cast films from neat PVA solution and PVA/chitin whiskers mixtures containing 2.55% to 25.38% chitin whisker.



**Figure 5.10** Specific tensile strength and specific Young's modulus of electrospun fiber mats and solution-cast films from neat PVA solution and PVA/chitin whiskers mixtures containing 2.55% to 25.38% of chitin whisker.