## **CHAPTER** V

# PREPARATION AND CHARACTERIZATION OF SILVER NANOPARTICLES FILLED THE BLEND OF BACTERIAL CELLULOSE/POLY(VINYLIDENE FLUORIDE) NANOCOMPOSITE FILMS

## 5.1 Abstract

In this study, the effect of silver nanoparticles (AgNP) loading on blend film of BC/PVDF (2.5/97.5) was investigated on dielectric and optical properties based on the basis of flexible piezoelectric touch sensor. The dielectric enhancement was found at all amount of AgNP added. At 0.5wt% of AgNP, the dielectric constant increased about 3 times of non-AgNP added at 20 °C and 10 MHz. In addition, the dissipation factor tended to decrease from 0.25 to 0.15 at the same condition. Dynamic mechanical and thermal analysis indicated that there is no significant in improvement in storage modulus and degradation temperature. However, lower percentage of light transmittance was obtained in nanocomposite films with the presence of AgNP.

#### **5.2 Introduction**

Nowadays, the piezoelectric touchscreen sensor has been one of the most popular technologies. The use of this technology tended to be used as flexible screen, but the brittleness of the ceramic material which typically used in the piezoelectric sensor is the main problem for this application. To overcome this limitation, the use of polymeric material in the electronic devices becomes more interesting. Not only of its advantage of flexibility but also lightweight, chemical stability and processability. However, because low dielectric and piezoelectric properties was very low compare to the ceramic material, the enhancement in dielectric constant to the polymeric material was required.

There were few ways to enhance dielectric properties of polymeric material such as high amount of high dielectric fillers or small amount of conductive

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filler. High dielectric fillers, typically were ceramic material which can enhance the brittleness in the polymer composite while small amount of conductive filler (metal) has lower effect on the other properties. Metal particles are widely used as the conductive fillers in common electrical application due to their high electrical conductivity and processability (Kuang *et al.*, 2013). The conductivity of metal particles arise from the electron can move freely in electron sea which presence in the metallic structure. This mobile electron can move in all three-directions. Silver is an important metal to study due to its very high conductivity in the bulk and also for its attractive optical properties which depend on the nanoparticle shape and size (Zulkarnaim *et al.*, 2008). In advance, silver nanoparticles (AgNP) promises nanoscale filler. easily doped with natural fiber which can also create the antimicrobial property (Maneerung *et al.*, 2008). There was the report from (Kuang *et al.*, 2013) that silver nanoparticles can enhance the dielectric properties of the polymeric materials through the interfacial polarization and percolation threshold (Tee *et al.*, 2007).

The objective of this work is to increase the dielectric properties of BC2.5PVDF97.5 by incorporated of AgNP to the bio-based blend film. This AgNP-BC/PVDF nanocomposite films are mainly characterized the essential properties for piezoelectric touch sensor. The dielectric and optical properties were investigated. The crystalline phase, dynamic mechanical and thermal were also strongly discussed.

#### **5.3 Experimental Procedures**

5.3.1 Materials

Nata de coco was purchased from local food market. PVDF (SOLEF1008) was kindly supported by Solvay (Thailand) Co., Ltd. N,N-dimethylformamide (DMF) and sodium hydroxide (NaOH) were purchased from RCI Labscan Co.,Ltd. and Merck Ltd., respectively. Silver nitrate solution (AgNO<sub>3</sub>) and sodium borohydride solution (NaBH<sub>4</sub>) were purchased from Sigma-Aldrich Co., Ltd.

## 5.3.2 Extraction and Purification of Bacterial Cellulose

Nata de coco gel was firstly washed with water to remove some excess sugar. After that the washed nata de coco gel was grinded and treated with 0.1 M NaOH at 80 °C for 1 h to obtain the BC pellicles. The BC pellicles were then washed with hot deionized water until neutral pH is reached. The BC pellicles were dispersed in deionized water and kept in bottle. Density of BC pellicles would be determined after freeze-drying.

## 5.3.3 Fabrication of AgNP-BC/PVDF Nanocomposite Films

The nanocomposite films were firstly prepared by solvent casting technique. DMF was used as solvent to dissolve PVDF at 60°C and stirred until the homogeneous solution was obtained. The natural conductive fiber was firstly prepared by chemical reduction method. 500 mg of BC was soaked in 0.01 AgNO<sub>3</sub> solution at various amount of AgNO<sub>3</sub> to obtain (0.125 wt%, 0.25 wt% and 0.5 wt%) for 1 h. Then the Ag ion-saturated BC pellicles are reduced with 0.01 M of aqueous NaBH<sub>4</sub> 10 min and rinsed with a large amount of deionized water for 30 minutes to remove the excess chemical. Then, AgNP-BC pellicles and PVDF were mixed together at the desired weigh ratio 2.5/97.5. The mixtures were stirred at 60°C for 30 minutes to allow good distribution then poured on Petri dish glass. After the solvent was vaporized in an oven at 80°C for 48 h in order to remove all remaining solvent, the casted film of BC/PVDF were obtained. Then, hot-pressed technique was used to make a dense film. The casted nanocomposite films were cut into pellets, heated at 200°C for 20 min, followed by 10 tons of pressure for 15 min. Finally, the films were about 0.3-0.5 mm in thickness and named as shown in the Table 5.1, with A referred to AgNP, B referred to bacterial cellulose and P referred to PVDF. The number behind the samples' name shows weight percentage of AgNP and bacterial cellulose in PVDF hierarchical composite with fixed amount of PVDF at 97.5 wt%.

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Sample	Composition weight percentage (%wt)			
	AgNP	BC	PVDF	
BC2.5PVDF97.5	0.000	2.500	97.500	
ABP(0.125/2.375)	0.125	2.375	97.500	
ABP(0.25/2.25)	0.250	2.250	97.500	
ABP(0.5/2.0)	0.500	2.000	97.500	

## 5.3.4 Characterizations

The  $\beta$  and  $\alpha$  phase of crystalline formation of PVDF in the blends were investigated using Fourier transform infrared spectroscopy (Nicolet, NEXUS 640) via ATR mode. The crystalline phase behavior of the blends also confirmed Xray diffractometer (XRD) (Rigaku). The dielectric constant and dissipation factor were measured using a Network Analyzer (Agilent. E4991A) at the frequency ranging from 10 MHz to 1 GHz and temperature of -50 °C to 150 °C. The thermal transitions of each compositions T<sub>c</sub> and T<sub>m</sub> were investigated by DSC. Dynamic mechanical analysis was performed in tension mode using a GABO. EPLEXOR 100N at a constant frequency of 1 Hz to studies dynamic mechanical properties at temperature of -80 °C to 150 °C.

#### 5.4. Results and Discussion

## 5.4.1 Presence of AgNP in BC/PVDF Nanocomposite Films

SEM-EDS images of AgNP-BC/PVDF nanocomposite films at various amount of AgNP were used to confirm the presence of AgNP in nanocomposite by using the red spots to indicate the positions of AgNP in the nanocomposite films as shown in Figure 5.1. It was found that there were no red spots in the BC2.5PVDF97.5 blend film which was corresponded to the absence of the AgNP in this film. In addition, as higher amount of AgNP in the nanocomposite, the higher amount of red spots presence in the images. Moreover, with the higher amount of AgNP, the agglomeration of AgNP was formed.

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For nanocomposite films morphology, there was no change in the fracture surface even AgNP was added compared to BC/PVDF blend film. This may be because only small amount of AgNP was added which does not have the effect on reinforcing to the PVDF matrix.



Figure 5.1 SEM-EDS images of AgNP-BC/PVDF nanocomposite films

## 5.4.2 Surface Morphological Properties

Table 5.2 shows surface parameters of neat PVDF, its blend and its nanocomposite films. The average roughness parameters ( $R_a$ ) were in the range from 0.15 to 0.45 nm. BC and AgNP didn't affect to the sample roughness because BC and AgNP were presence in the sample, not the surface. However, even the roughness parameters were low, but they didn't achieve the surface roughness requirement which was lower than 5 nm (Choi et al., 2008).

6	<b>Roughness parameters</b>				
Sample	R <sub>q</sub> (nm)	R <sub>a</sub> (nm)	R <sub>z</sub> (nm)		
neat PVDF	28	26	85		
BC2.5PVDF97.5	57	49	152		
ABP(0.125/2.375)	19	17	60		

 Table 5.2 Results of the AFM analysis of PVDF. its blend and its nanocomposite films

### 5.4.3 <u>Crystalline Phase Behavior</u>

The absorption bands characteristic of BC/PVDF blend film and AgNP-BC/PVDF nanocomposite films showed two common polymorphs referred as the  $\alpha$  and  $\beta$  phases. The molecules in the  $\alpha$  phase show vibration bands at 612 cm<sup>-1</sup>, 763 cm<sup>-1</sup> (CF<sub>2</sub> bending and skeletal bending), and 795 cm<sup>-1</sup> (CF<sub>2</sub> rocking). The formation of the  $\beta$ -phase, all-trans or planar zigzag conformations. is observed by the presence of specific absorption bands at 840 cm<sup>-1</sup> (CH<sub>2</sub> rocking) (Salimi *et al.*, 2003; Dahan *et al.*, 2012). Moreover, there is an absence of a peak in the 1630 – 1780 cm<sup>-1</sup> range which refers to C=O stretching in the DMF molecules. So, this can be concluded that both BC/PVDF blend film and AgNP-BC/PVDF nanocomposite films can be considered as solvent free.

The  $\beta$  phase content, F( $\beta$ ), of the nanocomposite films were calculated by using equation 3.1 and summarized in Table 5.3. The highest  $\beta$ -phase content (F( $\beta$ )=0.64) was observed in BC2.5PVDF97.5 blend film. With the presence of AgNP. F( $\beta$ ) become decreasing (F( $\beta$ )=0.36-0.37). This occurred because of the agglomeration of AgNP which may disrupt the PVDF chains not to form trans conformation ( $\beta$ -phase) easily, while the  $\alpha$ -phase was formed due to its thermodynamic favored phase.



Figure 5.2 FT-IR spectra of compressed AgNP-BC/PVDF nanocomposite film.

Sample	<b>F</b> (β)
BC2.5PVDF97.5	0.64
ABP(0.125/2.375)	0.36
ABP(0.25/2.25)	0.37
ABP(0.5/2.0)	0.36

**Table 5.3**  $\beta$ -phase contents. F( $\beta$ ) of AgNP-BC/PVDF nanocomposite films

Figure 5.3 shows XRD patterns of crystalline phase of AgNP-BC/PVDF blends at 0.125, 0.25, and 0.5 wt% of AgNP. The peaks at 20.4° refer to  $\beta$ -crystalline phase which has orthorhombic unit cell while the monoclinic  $\alpha$ crystalline phase can be found at of 18.6° and 27° (Chang *et al.*, 2008). When the amount of AgNP was increased, there was no significant change in height of those characteristic peaks because only small amount of AgNP added does not affect to the PVDF chains orientation according to FT-IR spectra. There were small peaks observations at 44.3°, 64.4° and 78.0°. These 20 peaks indicated to the AgNP planar

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(200), (220) and (311), which can confirm that AgNP was successfully synthesized (Kuang *et al.*, 2013).



**Figure 5.3** X-ray diffraction patterns of AgNP-BC/PVDF nanocomposite films compared with BC2.5PVDF97.5 blend film.

### 5.4.4 Thermal Properties

Figure 5.4 shows the second-heating curves of AgNP-BC/PVDF nanocomposite films with different contents of AgNP. The DSC parameters are also shown in Table 5.3. From the result, there was a transition temperature observed around 171-174 °C which corresponding to  $T_m$  of PVDF. In this blend film, only PVDF part was melted while BC cannot. There was no significant change of  $T_m$  in nanocomposite films compared to blend film because only small amount of AgNP was added which was observed in SEM-EDS images.

In addition, AgNP has slightly effect on the PVDF crystallinity ( $X_C$ ). The result shows that increasing of AgNP content could decrease the  $X_C$ , this occurred because AgNP may agglomerate and disrupt the PVDF chains not to crystallize easily. Crystallization temperature was also observed and showed in Figure 5.5 and Table 5.4. It was found that higher amount AgNP in nanocomposite films can slightly enhance  $T_c$  because of the agglomeration of AgNP which can disrupt the PVDF crystalline formation. In advance, the  $X_c$  values from heating and cooling step were almost the same which can indicate that PVDF can completely melt in this system.



Figure 5.4 DSC second-heating curves of AgNP-BC/PVDF nanocomposite tilms.



Figure 5.5 DSC first-cooling curves of AgNP-BC/PVDF nanocomposite films.

**Table 5.4** DSC parameters of AgNP-BC/PVDF nanocomposites films compare withBC2.5PVDF97.5 blend film

Sample	$\Delta H_m$ (J/g)	$T_m(^{\circ}C)$	X <sub>c</sub> (%)	$ \begin{array}{c} \Delta H_c \\ (J/g) \end{array} $	$T_{c}(^{\circ}C)$	X <sub>c</sub> (%)
BC2.5PVDF97.5	38.55	171.8	42.79	38.61	146.6	43.90
ABP(0.125/2.375)	34.73	172.0	38.55	36.24	147.8	41.21
ABP(0.25/2.25)	33.43	173.8	37.11	33.08	146.9	37.61
ABP(0.5/2.0)	32.05	171.9	35.57	33.52	147.9	38.11

\*Heat of fusion value for 100% crystalline PVDF,  $\Delta H_m^{\circ} = 92.4$  J/g and Heat of crystallization value for 100% crystalline PVDF,  $\Delta H_c^{\circ} = 90.2$  J/g

Thermal degradation behavior was observed by using TGA. Figure 5.6 shows TGA thermograms of the AgNP-BC/PVDF nanocomposite films. In this experiment, BC2.5PVDF97.5 was provided for comparison. It was found that the

thermal degradation behavior of the BC2.5PVDF97.5 was divided into two steps. The first step was started from 300 °C to 350 °C, which corresponded to the degradation of BC (George *et al.*, 2011). Whereas, the second step, the degradation started from 420 °C to 520 °C, which corresponded to the degradation of PVDF (Chae *et al.*, 2007). However, it was found that in all Ag-BC/PVDF nanocomposites, the degradation temperature did not change compared to BC2.5PVDF97.5 blend film, due to the fact that only small amount of AgNP added (<0.5wt%) that doesn't have the effect on the thermal properties. Furthermore, it was also found that these AgNP-BC/PVDF nanocomposite films were free from DMF due to the absence of thermal degradation at 150 °C, which was the boiling point of DMF.



**Figure 5.6** TG-DTA thermograms of AgNP-BC/PVDF nanocomposite films in N<sub>2</sub> atmosphere.

## 5.4.5 Dynamic Mechanical Properties

Storage modulus (E') of various amounts of AgNP in BC/PVDF blend films compared to BC2.5PVDF97.5 is shown in Figure 5.7. It can be noted that the storage modulus decreased dramatically with increasing of temperature. This phenomena occurred due to the fact that polymer chains had energy to vibrate and move, so the mechanical property of nanocomposites become worse. No significant change of E' even different amount of AgNP added due to very small amount of AgNP filled.

Damping factor (Tan  $\delta$ ) of various amounts of AgNP in BC/PVDF blend compared to BC2.5PVDF97.5 is shown in Figure 5.11. As observed, there were three relaxation peaks in BC2.5PVDF97.5. The first relaxation was  $\beta$  relaxation or dynamic glass transition temperature (Tg) which was found at -40 °C indicate to the-molecular movement in amorphous regions (Rekik et al., 2013). The 2<sup>nd</sup> relaxation which was not observed in polymer nanocomposite films, was observed at 25 °C corresponding to the  $\beta$ ' relaxation. This relaxation indicated to the folded of polymeric chains in amorphous region due to the compressed-force. The 3<sup>rd</sup> relaxation is  $\alpha$  relaxation that referred to the segmental motion in crystalline regions, exhibited near the melting temperature (O-Rak, 2013). Only two relaxation peaks were observed in nanocomposites films. For  $\beta$  relaxation mechanism, the incorporated of AgNP lower the dynamic T<sub>g</sub> because of the energy dissipation in the form of heat by AgNP to the surrounded polymeric chains, so PVDF chains in the amorphous regions can move easily. The  $\beta$ ' relaxation seems to disappear due to the AgNP may obstruct the PVDF chains in amorphous region not to create fold part. In addition, at any amount of AgNP in nanocomposites films had higher dissipation factor than the blend film, the reason why this can occurred is the nature of AgNP which is good thermal conductor, so the AgNP can dissipate heat energy inside the nanocomposites films better than blend film.



**Figure 5.7** Storage tensile modulus, E` vs temperature of various amount of AgNP in BC/PVDF blend films compared with BC2.5PVDF97.5 blend film.



Figure 5.8 Damping factor (Tan  $\delta$ ) vs temperature of various amounts of AgNP in BC/PVDF blend films compared with BC2.5PVDF97.5 blend film.

Sample	Transition Temperature (°C)			Storage Modulus, E' (Mpa)		
	β	β'	α	-50 °C	0 °C	50 °C
BC2.5PVDF97.5	-38.6	25.2	91.9	5404	2757	1769
ABP(0.125/2.375)	-40.9	-	93.4	6018	2782	1616
ABP(0.25/2.25)	-40.2	-	99.2	5768	2702	1695
ABP(0.5/2.0)	-41.2	-	101.5	5308	2611	1592

 Table 5.5
 Summary of transition temperature and storage modulus of AgNP 

 BC/PVDF nanocomposite films at various amount of AgNP from DMA technique

## 5.4.6 Optical Properties of AgNP-BC/PVDF Nanocomposite Films Compared to BC2.5PVDF97.5 Blend Film.

Figure 5.9 shows the appearances of nanocomposite films with various amounts of AgNP. The presence of AgNP in nanocomposite films made the films more opaque. This result can be confirmed by UV/Vis spectra of nanocomposite films which is shown Figure 5.10. The UV/Vis spectra indicated that even small amount of AgNP was added, the percentage of transmittance dramatically decrease. This can be explained by the fact that AgNP was in the nano-size which causing high surface-to-volume ratio. In addition, AgNP has neutral charge, so their particles didn't repel each other. According to these reasons, AgNP tended to segregate and form a micro-size aggregation.



Figure 5.9 AgNP-BC/PVDF sample appearances.



Figure 5.10 UV/Vis spectra of AgNP-BC/PVDF nanocomposite films.

### 5.4.7 Dielectric Properties of AgNP-BC/PVDF Nanocomposite Films

Figure 5.11 and 5.13a show the effect of AgNP in nanocomposite films on the dielectric constant. As can be seen, the dielectric constant of nanocomposite films was higher than that of BC/PVDF blend film at all range of temperature and frequency. Considering at frequency of 10 MHz and room temperature, the highest dielectric constant was obtained at 0.5wt% of AgNP which was 13.6, 4 times higher than blend film at the same condition. The enhancement of dielectric constant was due to the fact that AgNP is a conductive filler which can provide higher polarization. Moreover, due to the presence of AgNP in the nanocomposite films, higher the dipolar or orientation polarization was obtained, resulting to increase the dielectric constant. Figure 5.12 and 5.13 (b) show the effect of AgNP in nanocomposite films on the dissipation factor. Not only dielectric constant was increased, but also the dissipation factor when higher amount of AgNP was occurred.

• Figure 5.11 shows the dielectric constant of AgNP-BC/PVDF nanocomposite films compare with BC2.5PVDF97.6 blend film as a function of temperature at 10 MHz. 100 MHz and 1 GHz. The result shows the highest dielectric

constant of all sample was found at high temperature because of PVDF chains has high energy enough to move freely, then dipole orientations of AgNP, BC and PVDF were easily to formed and allowed them to keep up with the changing of electric field. At lower temperature, the segment motion of the chains were frozen which stopped the three components not to orient the dipole and create the polarization further.

For the frequency dependence, similar to previous chapter, at lower frequency (10 MHz), the dipoles of polymeric chains had sufficient time to align with the electrical field direction causing to the higher dielectric constant ( $\epsilon$  of 0.5wt% of AgNP = 16 at the frequency of 10 MHz). With higher frequency, the dipoles had shorter time to align with the electrical field direction, so the lower dielectric constant obtained ( $\epsilon$  of 0.5wt% of AgNP = 10 at the frequency of 1 GHz). This result was also corresponded to the results in Figure 5.13a. This can be concluded that the presence of AgNP, nanocomposite films still had the same temperature and frequency dependence on the dielectric behavior as the blend film.

For the dielectric relaxation behavior, Figure 5.12 shows the relaxation peaks at various frequencies. Similar to dynamic mechanical analysis technique, the dynamic glass transition temperature  $(T_g)$  also can be measured by the dielectric properties measurement. The relaxation behavior was referred to the change from rubbery state to glassy state of the PVDF chains. At lower frequency, the relaxation behavior was found at low temperature, while occurred at high temperature with higher frequency. This happened because at low frequency, the PVDF chains in amorphous region have sufficient time to relax the energy, but insufficient time for PVDF chains in amorphous region to relax at high frequency. Therefore. PVDF chains in amorphous region needed to have high energy enough to relax. The dynamic T<sub>2</sub> was shifted to higher temperature. In contrast, it was found that at this dynamic T<sub>g</sub>, the dielectric constant was increased significantly due to the PVDF chains in amorphous region were free to move and more amount of dipolar polarization was formed. In advance, it was found that the dynamic T<sub>g</sub> was lower when AgNP was incorporated. At 10 MHz, dynamic Tg was shifted from 30 °C to 20 °C and 90 °C to 60°C for frequency of 100 MHz. The reason why this happened due to AgNP was the thermal conductor which can dissipate heat to surrounding well.

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 $\mathsf{PVDF}$  chains in amorphous region can obtained enough energy to relax faster than the absence of AgNP.





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**Figure 5.11** Dielectric constant of AgNP-BC/PVDF nanocomposite films as a function of temperature at 10 MHz, 100 MHz and 1 GHz of frequency.



**Figure 5.12** Dissipation factor of AgNP-BC/PVDF nanocomposite films as a function of temperature at 10 MHz, 100 MHz and 1 GHz of frequency.

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**Figure 5.13** Frequency dependence of (a) Dielectric constant (b) Dissipation factor of loaded AgNP-BC/PVDF nanocomposite films at 20 °C

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## 5.5 Conclusions

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The silver nanoparticles filled BC/PVDF (2.5/97.5) nanocomposite films were prepared using solution casting followed by compression technique. The crystalline phase behavior of the blend composed of mixed crystalline phases ( $\alpha$  and  $\beta$ ) as investigated by FT-IR spectra and XRD patterns. There was not significantly change in thermal properties which was confirmed by both DSC and TG-DTA techniques. In addition, storage modulus was not changed from the blend even AgNP was added. Those phenomena may be explained due to the very small amount of AgNP added. However, even if the small amount added, the optical and dielectric properties were significantly changed. Due to the agglomeration of the AgNP in BC, the percentage of visible transmittance becomes worse. For dielectric properties, the higher amount of AgNP, the higher the dielectric constant. The dissipation factor was also increased when the amount of AgNP increased. On the basis of these results, these bio-composite films were still far from reality for using as flexible piezoelectric touchscreen sensor. More and more improvements are required to develop this film for using in this application in reality.

## 5.6 Acknowledgement

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