Nanostructure and Surface Characteristic of Electrospun Carbon Black/PVDF Copolymer Nanocomposite

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Abstract

Carbon black/PVDF copolymer (CB/PVDF copolymer) nanofiber composite has been synthesized by electrospinning process. Nanostructure and surface property of CB/PVDF copolymer were studied by using Scanning Electron Microscopy (SEM), Fourier Transform Infrared Spectroscopy (FTIR), X-ray Diffraction (XRD) and water contact angle (WCA). Nanofiber composite has smooth surface morphology without bead on nanofiber string. Increase of CB content in nanofiber composite decreased diameter of nanofiber size. Range of average nanofiber diameter was from 405.2 and 421.3 nm where it depended on CB content in nanofiber composite. FTIR and XRD analysis showed that structure of PVDF copolymer in nanofiber composite had a dominant β phase crystal structure. The β phase was showed and confirmed by vibration band peak at 838.55 cm^{-1} of IR spectrum and $2\theta = 20.44^\circ$ of XRD. Presence of CB in nanofiber composite also influenced WCA and water spreading on nanofiber composite. WCA of pristine PVDF copolymer, CB/PVDF copolymer nanofiber with 1% CB, and CB/PVDF copolymer with 5% CB were $113.8, 105.2, 117.3^\circ$. From this result showed that nanostructure and surface characteristic can be adjusted and controlled by presence of CB in nanofiber composite. This result has potential application for air filtration to solve air pollution issued.

Keywords: CB/PVDF copolymer nanofiber composite, carbon black, nanostructure, electrospinning, water contact angle of nanofiber composite

Kulcsszavak: CB/PVDF kopolimer nanoszál kompozit, szén fekete, nanoszerkezet, elektro-szállékpézés, víz nedvesítési szöge nanoszál kompoziton

1. Introduction

Nanofiber, fabricated by electrospinning, has great potential to be used in many environmental applications. The main advantage of this process is the relative quick and simple way to fabricate a variety of materials into nanofiber structure [1]. Besides, the high surface-to-volume ratio, large porosity (up to over 80%) and adjustable functionality are more effective than conventional non-woven and polymeric membranes in particulate separation and liquid filtration [2].

Poly(vinylidene fluoride) (PVDF) nanofiber and poly(vinylidene) fluoride copolymer are the polymer materials which easily synthesized by electrospinning process. Excellent resistance towards harsh chemicals, piezoelectricity, flexibility, durability, lightweight, and relatively low acoustic impedance properties [3-5] considered PVDF nanofiber to be used in some applications such as membrane separator of secondary Li-ion battery [6-8], supercapacitor [9-10], sensing element [11-13] and air filtration [14-16].

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PVDF has dipole moments due to the polar C-F bond which distribute along the fiber axis. It causes the electrostatic attractive forces integrate along the length of the fiber segments. This integration strengthens the electric field projected from the fibers [15, 17]. The electric charge which takes place between filter media and dust particles makes PVDF nanofiber is capable for capturing particles (charged and uncharged aerosols) effectively in air filtration application [14-16, 18-19]. The fibers with small diameter enhance the van der Waals attractive force between nanofiber and particle [14] then trap particles among the polymer matrix [16].

The unique electrospinning process offers opportunities to fine tune the surface functionality through polymer chemistry,

blending and nanofiller incorporation during the process [2]. New approaches in a modification of nanofiber were reported by many researchers which have been incorporating nanoparticle in solution polymer system for fabricating nanofiber composite.

It has been over one decade that carbon black (CB) nanoparticle became one of the interesting materials to modify polymer nanofiber due to small particle size (high surface area), aggregation behavior, low cost and conductive property [20-21]. Those properties make CB has usability as an adsorbent material [22]. Islam et al [23] reported that CB is the most effective adsorbent material than extruded activated carbon and granular activated carbon. In nanofiber system, the presence of CB in polyurethane (PU) [20] and polyaniline (PANI) [24] nanofiber composite increased the fiber web strength and electrical conductivity. However, it decreased significantly when CB incorporated in PEO system [25].

As much as we know that no other researchers reported the effect of incorporating CB in pristine PVDF copolymer solution to synthesize nanofiber using electrospinning. In this work, we used some variation of the amount of CB which incorporated into PVDF copolymer to produce nanofiber composite. We concerned in basic concept including effect of the filler content on the fiber morphology, the bond structure of the nanofiber composite, the crystallinity and the hydrophilicity. These results will support usability of CB/PVDF copolymer nanofiber composite to improve PVDF nanofiber function as air filtration. We suggested this nanofiber composite not only trap particulate matter, but also adsorb chemical pollutant in the air. Increasing the web strength and electrical conductivity of CB/PVDF copolymer nanofiber composite are expected can improve their ability to trap particulate matter more easily.

2. Experimental

2.1 Materials

Poly(vinylidene fluoride) (PVDF) copolymer which was used in this work is poly(vinylidene fluoride-co-hexafluoropropene) (PVDF-HFP). It was purchased from Arkema Inc. U.S.A. The *N,N*-dimethylacetamide (DMAc) from Merck was used as solvent for preparation of polymer solution. Carbon black (Dashblack HAF N330) was obtained from OCI Company Ltd. and used as nanoparticle which incorporated into polymer solution.

2.2 Nanofiber preparation

A 24% (w/v) cPVDF solution was prepared by dissolving 2.4 g cPVDF powder in 10 mL DMAc. It was stirred at room temperature for 5 h. CB was added to the cPVDF solution with variation concentration 0, 1, and 5% (w/v) for pristine PVDF, CB1/PVDF copolymer and CB5/PVDF copolymer, respectively, then stirred for 12 h. The composite solution was spun into nanofiber using electrospinning with parameters 22 kV, 13 cm, and 0.004 mL/min for 2 h.

2.3 Characterization

Surface morphology of nanofiber was analyzed by Scanning Electron Microscopy (SEM) SEMSU3500. Fourier Transform

Infrared Spectroscopy (FTIR) Nicolet iS5 ATR iD5 Thermo Scientific was used for analysis of interaction between PVDF and carbon black in nanofiber composite. Crystallinity between pristine PVDF nanofiber and its composite was analyzed by XRD Shimadzu 7000. Water contact angle was measured by drop shape method and calculated by the equation:

$$\frac{\theta}{2} = \tan^{-1} \left(\frac{h}{d} \right) \text{ with } \theta = \text{contact angle, } d = \text{diameter, } h = \text{height.}$$

3. Results and discussion

3.1 Morphological characteristics and fiber diameters

Incorporated CB into PVDF copolymer nanofiber system gives effect onto morphology and diameter of nanofiber. The amount of CB was added into polymer solution is 0, 1, and 5% (w/v). Fig. 1 shows that pristine and composite of nanofibers have smooth morphology without bead on nanofiber string. Increase of CB content in CB/PVDF copolymer nanofiber composite results decreasing diameter of nanofiber. Diameter of pristine PVDF copolymer nanofiber is 599.5 nm then changes to 405.2 and 421.3 nm after adding carbon black 1 and 5% (w/v), respectively. The diameter of CB/PVDF copolymer nanofiber composite tends to decrease was caused by the tendency of CB to absorb the solvent. Evaporation of solvent in composite polymer solution can be slower than pristine polymer during the fiber formation [20, 26]. Besides, the conductivity of CB in composite polymer solution also caused diameter of CB/PVDF copolymer nanofiber is smaller than pristine PVDF copolymer [27-28]. In CB5/PVDF copolymer composite nanofiber, the diameter is larger than CB1/PVDF copolymer. It suggested the amount of CB 5% (w/v) attributed to form agglomeration of nanoparticle. The agglomeration of CB is apparent on the fiber surface clearly, see Fig. 1.c.

3.2 Infrared spectrum

The bond structure and interaction between CB and PVDF copolymer in nanofiber composite is shown by Fig. 2.

In the IR spectrum of PVDF copolymer, crystalline β phase peak at 839.23 cm^{-1} indicated rocking vibration of CH_2 group. The presence of CB in composite nanofiber caused shifting peak at 838.55 and 834.62 cm^{-1} for CB1/PVDF copolymer and CB5/PVDF copolymer, respectively. The CF_2 symmetrical stretching was shown at 1400.27, 1175.09, and 1072.76 cm^{-1} . Incorporated CB in CB/PVDF copolymer nanofiber composite caused shifting peak at CF_2 function group such as 1399.30, 1171, and 1071.99 cm^{-1} for CB1/PVDF copolymer and 1396.61, 1164.68, and 1062.16 cm^{-1} for CB5/PVDF copolymer. The 1274.58 cm^{-1} is a peak for CF stretching vibration. Decreasing band intensity at about 1274 cm^{-1} in CB/PVDF copolymer can be caused by interaction between function group of CB and CF of PVDF. The 876.81 cm^{-1} is identical for CH_2 of PVDF. It shifted to 875.71 and 871.17 after added CB 1 and 5% (w/v), respectively. Band peaks at around 1160-900 cm^{-1} and 800-600 cm^{-1} of CB/PVDF copolymer nanofiber composite are broader than pristine PVDF copolymer nanofiber due to dominant effect from increasing amount of CB in nanofiber composite. The $\text{C}\equiv\text{C}$ bonding and $\text{CH}_3\text{-O-}$ at 2114.04 and 993.36 cm^{-1} are characteristics of CB function groups.

Fig. 1 SEM images of CB/PVDF copolymer nanofiber with various amount of CB (a) pristine PVDF copolymer, (b) 1% (w/v) and (c) 5% (w/v)

1. ábra Elektronmikroszkópos felvételek; CB/PVDF kopolimer nanoszál különböző CB adagolással (a) kiindulási PVDF kopolimer, (b) 1% (w/v) és (c) 5% (w/v)

3.3 Crystallinity

X-ray diffraction (XRD) pattern confirms the existence and effect of CB in nanofiber composite. Fig. 3 shows characteristic peak at $2\theta = 20.44^\circ$ for pristine PVDF copolymer nanofiber, 20.06° for CB1/PVDF copolymer and 20.88° for CB5/PVDF copolymer. Those peaks show strong peak of piezoelectric crystal β -phase structure of PVDF copolymer. The CB which incorporated in PVDF copolymer nanofiber gives effect on crystallinity of composite. CB acts as the nucleation agent in polymer matrix which affecting the crystallization process in the formation of PVDF copolymer nanofiber [29].

Incorporating CB in PVDF copolymer nanofiber decreases the crystallinity. The pristine PVDF copolymer nanofiber has 49.84% and decreases to 42.73 and 48.49% for adding CB 1 and 5% (w/v), respectively. The crystalline structure of the CB/PVDF copolymer may arise from the cooperative orientation of the CB and PVDF copolymer chains along the fiber axis to facilitate the nucleation of highly oriented chains crystallites at the interface [30]. The amount and type of nanoparticle can result difference crystallinity [29-31].

Fig. 2 FTIR spectrum of CB/PVDF copolymer nanofiber composite (a) CB nanoparticle, (b) pristine PVDF copolymer, (c) CB1/PVDF copolymer, and (d) CB5/PVDF copolymer

2. ábra FTIR spektrumok; CB/PVDF kopolimer nanoszál kompozit (a) CB nanorészecske, (b) kiindulási PVDF kopolimer, (c) CB1/PVDF kopolimer, és (d) CB5/PVDF kopolimer

Fig. 3 XRD spectrum of nanofiber composite (a) pristine PVDF copolymer, (b) CB1/PVDF copolymer, and (c) CB5/PVDF copolymer

3. ábra Kompozit nanoszál röntgendiffraktogramok (a) kiindulási PVDF kopolimer, (b) CB1/PVDF kopolimer, és (c) CB5/PVDF kopolimer

3.4 Hydrophobicity

The hydrophobicity of the nanofiber is analyzed by its value of water contact angle. The amount of CB which incorporated in CB/PVDF copolymer nanofiber composite gives effect to contact angle value. Pristine PVDF copolymer nanofiber has 113.8° then decreases to 105.2° for CB1/PVDF copolymer, but increases to 117.3° for CB5/PVDF copolymer.

Many pores of CB have ability to absorb liquid matter. In Figs. 1 and 5, CB incorporated well inside of CB1/PVDF copolymer nanofiber. When water droplet contacted with surface nanofiber, it is not only interacts with C-F of PVDF copolymer but also with function groups and pores of CB. It suggested contact angle value of water droplet is smaller than pristine PVDF copolymer nanofiber. Unlike CB5/PVDF copolymer, CB made formation of agglomeration and apparent on surface of nanofiber. In this case, the surface roughness plays a role in the formation of higher contact angle.

Fig. 4 shows the spreading time of water droplet on CB/PVDF copolymer composite nanofiber. After 130 and 240 minutes, water droplet on CB5/PVDF copolymer composite nanofiber is faster to spread than pristine PVDF copolymer and CB1/PVDF copolymer regularly. In that time, water droplet has contacted with CB surface then CB can easily absorb water.

Fig. 4 The spreading time of water droplet on CB/PVDF copolymer nanofiber composite

4. ábra Vízecpp terjedési ideje CB/PVDF kopolimer nanoszál kompoziton

Fig. 5 Illustration of CB incorporated in CB/PVDF copolymer nanofiber composite

5. ábra CB elegyedésének illusztrációja CB/PVDF kopolimer nanoszál kompozittal

4. Conclusions

PVDF copolymer nanofiber composite was successfully synthesized by electrospinning which incorporating CB into solution system. These results clearly demonstrate that CB has significant effect on nanostructure and surface characteristic. Their morphology showed that the presence of CB in PVDF copolymer nanofiber system decreases diameter of nanofiber

nanocomposite. In 5% (w/v) of CB in nanofiber composite, the agglomeration of nanoparticle is apparent in surface of nanofiber. Interaction between CB and PVDF was showed by shifting peak at CF_2 and CH_2 function groups and making band be broader than pristine PVDF copolymer. IR and XRD data confirm a dominant of β phase crystal structure of PVDF copolymer. The presence of CB in nanofiber composite also decreases their crystallinity. Besides, the hydrophobicity of CB/PVDF copolymer nanofiber composite changes to 105.2° and 117.3° for adding 1 and 5% (w/v) CB content, respectively, from 113.8° of pristine PVDF copolymer. After 130 and 240 minutes, CB5/PVDF copolymer nanofiber composite is faster to spread than pristine PVDF copolymer and CB1/PVDF copolymer.

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