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# Fabrication and Properties of Rubber Nanofiber from Electrospinning

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## Abstract

Electrospun nanofibers of rubber blending with poly(vinyl chloride) (PVC) were prepared by electrospinning. Various concentrations of rubber solution were added to the PVC solution for several purposes of the electrospun nanofibers. The pristine and rubber plasticized nanofibers were characterized by field emission scanning electron microscopy (FE-SEM), differential thermal analysis (DTA), and thermogravimetry analysis (TGA), respectively. Thermal properties from DTA and TGA results indicated that the addition of rubber decreased glass transition temperature ( $T_g$ ) of PVC and increased heat resistance of PVC nanofiber. The dielectric constant of plasticized nanofibers measured using precision LCR meter indicated that the membranes were dense.

**Keywords:** PVC, rubber, electrospinning, nanofiber

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## 1. Introduction

### 1.1. Elastomeric materials

Elastomers are important polymers having various applications in daily life. Elastomers can be classified by application purposes such as general and special. Elastomers are often mixed together to achieve superior properties which arise from the individual systems. Blending is macroscopically homogeneous mixture of two or more different species of polymers which is carried out for several purposes [1], such as improving the physical and mechanical properties of the first elastomer and obtaining good processing characteristics of rubber compound. The most important characteristic of a blending of two (or more) polymers is the phase behavior. Polymer blends (like low-molecular-weight solvents) can exhibit miscibility or phase separation and various levels of mixing in between the extremes and polymer blends as a mixture

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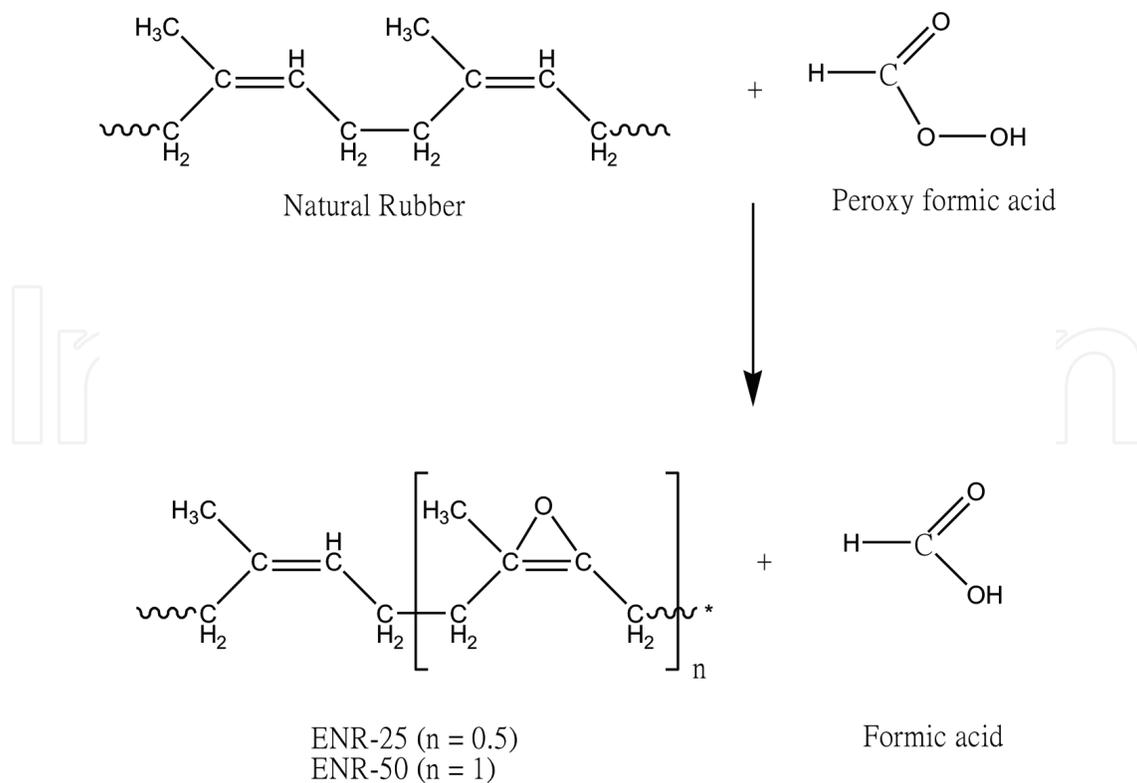
of at least two macromolecular substances, polymer, or copolymer, in which the ingredient content is above 2 wt% [2].

**Epoxidized natural rubber (ENR)** is a modified molecule of natural rubber with the chemical name cis-1,4polyisoprene. There structuring using chemicals such as peroxy acid will react with double bonds of the natural rubber molecules to form ring oxygen that is replaced; the epoxidation reaction of natural rubber is shown in **Figure 1**. In the beginning, formic acid reacts with hydrogen peroxide to form peroxy formic acid [3, 4]. Then, the peroxy formic acid reacts with double bond of natural rubber is arranged as a ring epoxide and formic acid is a by-product as shown in **Figure 1**.

However, only three types of ENR are considered as commercial standard. These are ENR-10, ENR-25, and ENR-50 where the integers designate 10, 25, and 50 mole% of epoxide incorporated into the natural rubber chain, respectively [4, 5].

Besides the proposed commercial uses shown, ENR has the potential to be further exploited for its usage as advanced materials such as in blends, additives, and fuel cells applications [6]. ENR had been blended with various polymers such as natural rubber [7, 8], styrene-butadiene rubber (SBR) [9], and poly(vinyl chloride) (PVC) [10–12].

**Polyurethane (PU)** is formed by reacting an isocyanate with a polyol. Both the isocyanates and polyols used to make polyurethanes contain on average two or more functional groups per molecule. Most polyurethanes are thermosetting polymers that do not melt when heated.



**Figure 1.** The reaction epoxidation of natural rubber.

Polyurethane has the flexibility and strength of rubber and the hardness of plastic. Many commercially available PU can be used to make good electrospinning solutions.

## 1.2. Electrospinning

Electrostatic spinning or electrospinning is a unique process to prepare electrospun fibers with diameters in the range of micrometers to nanometers that depends on the type of polymer and processing conditions [13, 14].

Electrospinning is the process that occurred over 60 years. In the year 1934, Formhals was a patented process and used as electrical instruments in synthetic fibers [15]. In the year 1969, Taylor published work describing the phenomena occurring at the time. Polymer solution is ejected from a metal needle at high-voltage power supply. As the electrostatic charge on the droplet overcomes the surface tension, the low-molecular-weight fluid breaks up into small droplets, named a Taylor cone [16].

The basic principles of electrospinning are very simple. There are mainly three components to fulfill the process as follows: a high-voltage power supply, a syringe with a metal needle, and a collector. The polymer solution with surface tension is encapsulated within a syringe, forming a hemispherical drop at the end of the capillary tip which is connected to an anode that connects to a high-voltage power supply, usually between 5 and 30 kV, which supports the ejection of a liquid jet followed by solvent evaporation as the jet travels through the air leaving behind ultrafine polymer fibers collected on a grounded cathode-connected metallic collector. The interaction between the liquid jet surface and external electric field is the cause of bending or a spiral track of the liquid jet which not only resulted in the electrospinning jet being elongated up to ultrafine fibers but also randomly deposited nonwoven electrospun mats [17–19]. Surface area and pore size of electrospun mats are larger and smaller, respectively, than commercial nonwoven fabrics. So, they are used in a wide variety of applications of electrospun mats, for example, tissue engineering, drug delivery system [20], wound-dressing [21] membrane [22], and conductive fiber [23].

It is well known that the morphology and size of electrospun fibers depends on various processing parameters such as the solution properties, controlled variables, ambient conditions, viscosity, and surface tension. With the control of these parameters, optimal nanofibers can be prepared. Electrospinning parameters and their effects on fiber morphology are as follows:

## 1.3. Solvents used for electrospinning

The polymer solution, used for electrospinning, usually was prepared by dissolving in different solvents. The solvent type is important on its spinnability both of first and foremost step of electrospinning process. The solvents should have some proper properties which are suitable with polymer types such as surface tension, vapor pressure, and relative density. Thus, the selection of an appropriate solvent system is indispensable which cause successful electrospinning process [24]. It is well known that different solvents may contribute to different surface tensions. The solution viscosity is determined by the concentration of the polymer,

but the value of surface tension depends on both the polymer and the solvent [25, 26]. **Table 1** shows physical properties of different solvents.

The ability of the volatility in the solvent such as reported that study PVC/PU electrospinning by solutions for electrospinning were prepared in a mixed solvent of tetrahydrofuran (THF) and *N,N*-dimethylformamide (DMF). It was found that THF has the ability of a good volatility and DMF has the ability of a poor volatility. It is found that when THF is used, 100% of the fibers have high porosity, and when DMF is used 100% of the fibers are smooth and microtexture fiber [27].

Solvents	CAS number	Surface tension at 20°C (mN/m)	Vapor pressure (hPa at 20°C)	Relative density at 20°C (g/dm <sup>3</sup> )
Acetone	67-64-1	25.2	233	0.79
Acetic acid	64-19-7	27.4	15.4	1.05
Chloroform	67-66-3	27.5	211	1.48
Dichloromethane	75-09-2	26.5	475	1.33
<i>N,N</i> -Dimethylformamide	68-12-2	37.1	3.77	0.94
Ethanol	64-17-5	22.1	59	0.80
Formic acid	64-18-6	37.6	42	1.22
Hexafluoroisopropanol	920-66-1	16.1	269	1.62
Methanol	67-56-1	22.7	128	0.79
Tetrahydrofuran (THF)	109-99-9	26.4	173	0.89
Trifluoroacetic acid	76-05-1	21.9	141	1.48
Water	7732-18-5	72.8	23.39	0.998

**Table 1.** Physical properties of some common liquids.

## 2. Fabrication of polymer nanofibers

There are a wide range of polymers that are used in electrospinning and are able to form fine nanofibers within the submicron range and used for varied applications. Electrospun nanofibers have been reported as being from various synthetic polymers, and natural polymers were electrospun from polymer solutions, such as poly(vinyl chloride) [28], poly(ethylene-co-vinyl alcohol) [29], polycarbonates [30], nylon-6 [31], polyurethane [32], polyacrylonitrile [33] polylactide [34], and poly( $\epsilon$ -caprolactone) [35].

Poly(vinyl chloride) is a hard, stiff material with low thermal stability, can be extensively modified with plasticizer and thermal stabilizer to enhance flexibility and thermal stability, respectively. Almost all plasticizers are commonly phthalate ester. The plasticizers interpose between every individual strand of polymer causing breakdown of polymer-polymer interaction. The

polymer structure is modified into more porous, flexible, and with less cohesive structure resulting in lower glass transition temperature ( $T_g$ ). The plasticized PVC behaves like a rubber at room temperature. However, plasticizer can slowly leach out with time and deform PVC at a lower tensile force. Moreover, the phthalate esters are endocrine disrupters or mutagens.

In this chapter, the effects of electrospinning condition including electric voltage, tip-to-collector distance, and PVC solution concentration were studied on the morphology of formation of PVC nanofibers. The physical properties of PVC nanofibers were studied by varying the blending ratio of PVC with polyurethane and PVC with epoxidized natural rubber.

### 2.1. Electrospinning of poly(vinyl chloride) blending with polyurethane

Poly(vinyl chloride) with a  $K$ -value of 580 was kindly supplied by Thai Plastic and Chemicals Co., Ltd. The solvents *N,N*-dimethylformamide and tetrahydrofuran were used as received. DMF was mixed with THF at the ratio 50:50 and was used to prepare the PVC solution with varying PVC concentration for 10 and 15 wt% at 50°C. Polyurethane (Dow Chemical Co., Ltd.) was dissolved in the same solvent mixtures as PVC solution at a concentration of 10 wt%. The PU solution was directly mixed with PVC solution at room temperature.

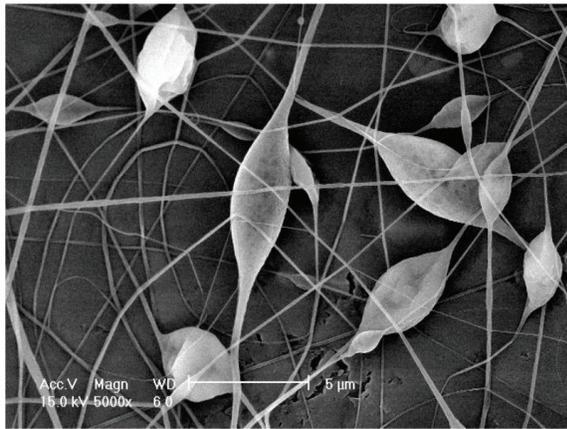
The picture of PVC nanofibers obtained from SEM shows the morphological variation that depends on PVC concentration and electrospinning conditions as illustrated in **Figure 2** [36]. PVC nanofibers were prepared from PVC solution at 10 and 15 wt% with an applied voltage of 12 kV and various distances between the syringe tip and the collector (12, 15, and 18 cm). The surface instabilities were induced by electrospun jet motion and charge interaction. Low concentration of PVC solution (10 wt%) led to the formation of beads on the fiber. On increasing the concentration of PVC solution (15 wt%), the beads formation decreased with a gradual increase in the average diameter which can be attributed to higher viscosity.

The morphology of PVC nanofiber is modified by blending with polyurethane solution at 10 wt% as shown in **Figure 3** [39]. PU blending with PVC smoothens the fiber without beads which was found in PVC nanofiber. PU component can be attributed to higher viscosity and surface tension of polymer blend solution. Viscosity and surface tension play an important role in determining the electrospun nanofiber formability and diameter.

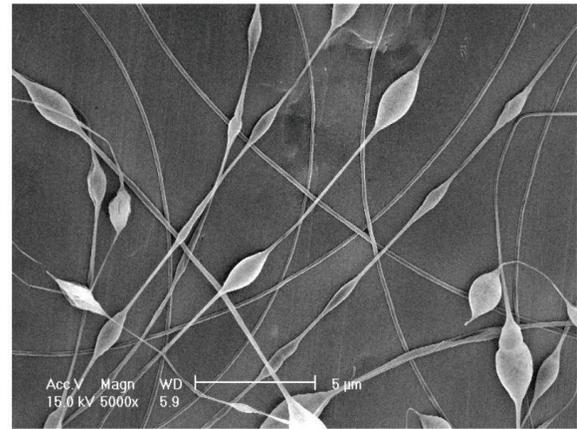
PVC nanofiber prepared from 10 wt% is rigid than that from 15 wt% which corresponds with thermal properties determined using differential thermal analysis (DTA). The nanofiber prepared from 10 wt% PVC has a high exothermal peak at 30 and 275°C, while the nanofiber from 15 wt% PVC shows endothermal peak. The thermal properties of nanofibers were improved after mixing PVC with PU solution. The mixture of PVC with high PU content gave more flexible nanofiber with a lower glass transition temperature ( $T_g$ ) and heat resistance as illustrated in **Figure 4** [39]. For higher concentration of PVC (15 wt%) with high viscous, it is not easily blended with PU solution which led to heterogeneous solution.

The thermal stability of the nanofibers was determined using thermogravimetric analysis. The thermogram of the PVC and PVC/PU blends as shown in **Figure 5** [39] has two obvious decompositions. The thermogram of the PVC/PU blend nanofiber shows the decomposition temperature ( $T_1$ ) at 250°C, weight-loss rate is the largest at approximately 300°C. There are

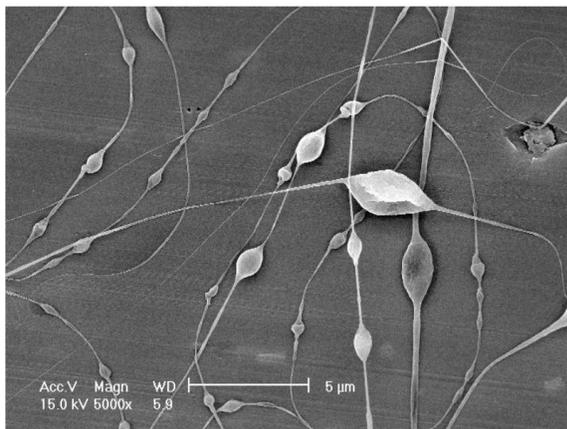
two decompositions of PVC/PU nanofiber. The first decomposition occurs at 275°C ( $T_2$ ) with a weight loss of about 10%, which may be due to the decomposition of PVC in the nanofiber. The second weight loss of about 62% occurs at around 433°C ( $T_3$ ), indicating that the PVC/PU blend nanofiber has a good thermal stability.



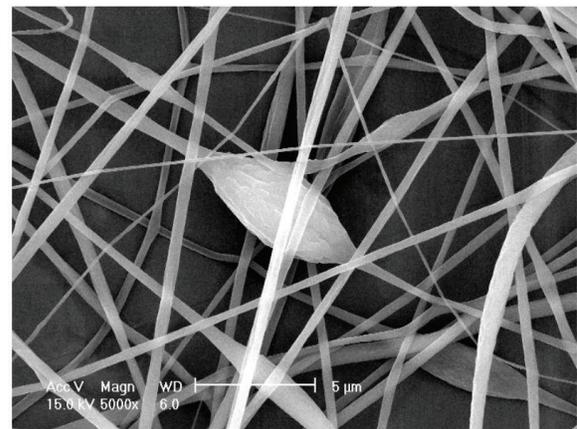
(a) Tip to collector 12 cm.



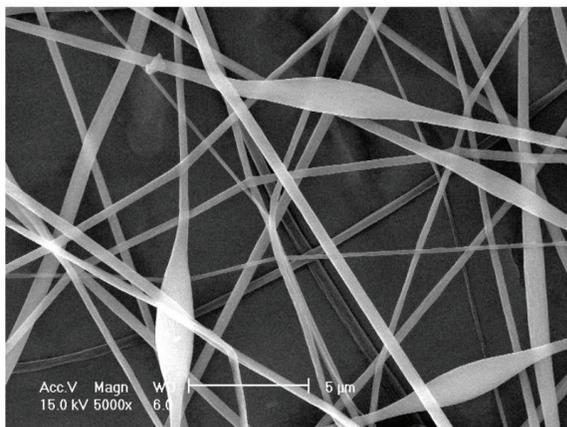
(b) Tip to collector 15 cm.



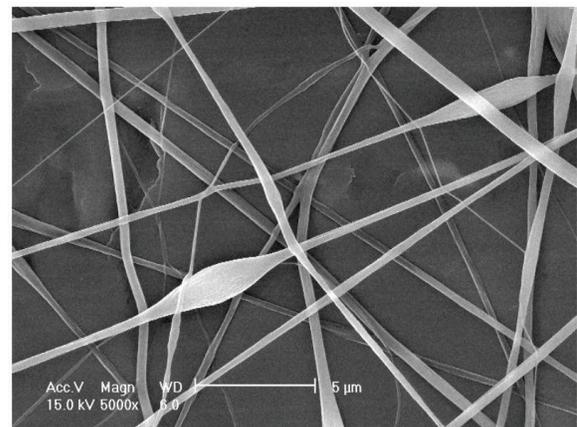
(c) Tip to collector 18 cm.



(d) Tip to collector 12 cm.

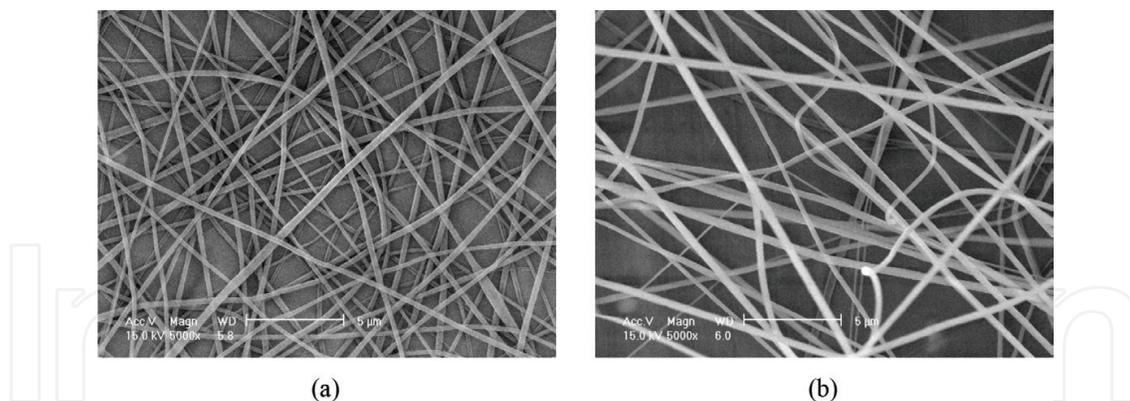


(e) Tip to collector 15 cm.



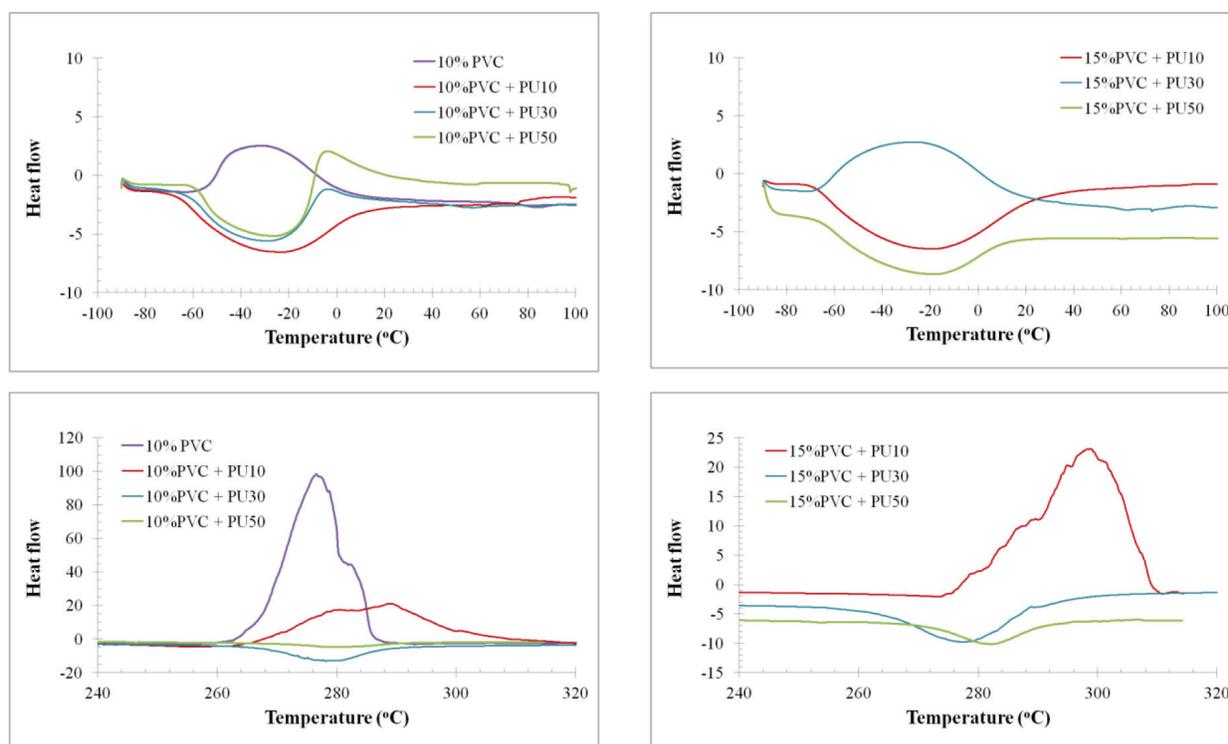
(f) Tip to collector 18 cm.

**Figure 2.** SEM images of electrospun nanofiber from PVC solution concentration of 10 wt% (a–c) and 15 wt% (d–f).



**Figure 3.** SEM images of electrospun nanofiber from PVC/PU blending of (a) PVC 10% with PU 10 wt% and (b) PVC 15% with PU 10 wt%.

The permittivity of PVC and PVC/PU blend nanofibers, which were measured over the frequency range from 75 kHz up to 30 MHz at room temperature, is illustrated in **Figure 6**. The dielectric constant of the nanofiber, which was prepared from 10 wt% of PVC mixed with high PU content, reduced the dielectric constant due to denseness of PU phase. On increasing the concentration of PVC solution to 15 wt% blending with PU solution, it was found that the dielectric properties were not changed with PU contents due to the prepared electrospun nanofiber membranes that were dense.



**Figure 4.** DTA thermogram of nanofibers from PVC and PVC blending with PU by varying PVC concentration and blending ratio.

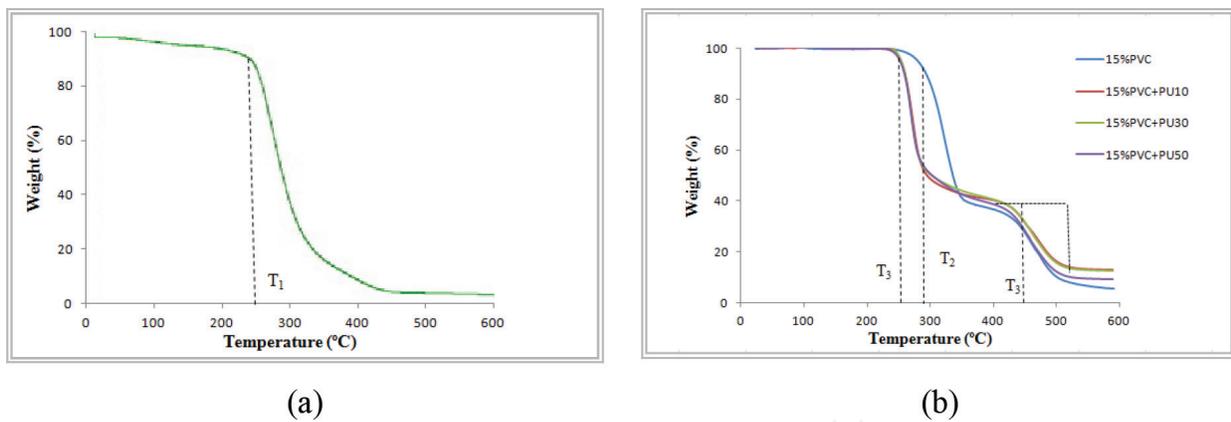


Figure 5. TGA of nanofibers from (a) PVC and (b) PVC blending with PU at difference ratio.

It was reported that [37] electrospun poly(vinyl chloride)/polyurethane-blended solutions (10/0, 9/1, 8/2, and 7/3 wt%) were prepared by adding PVC and PU in the mixture solvent of THF/DMF (1/9, w/w). It was found that membranes exhibited relatively high tensile strength (9.9 MPa), good air permeability (154.1 mm/s), and excellent abrasion resistance (134 cycles).

As shown in **Figure 7**, when an external load is applied, the hydrogen bonding between C=O groups of PU and the  $\alpha$ -hydrogen of PVC, and the dipole-dipole-C=O $\lambda$ Cl-C- interactions tend to bear the stress on intermolecular levels, resulting in the elongation of less than 6% in the first linear elastic behavior.

## 2.2. Electrospinning of poly(vinyl chloride) blending with epoxidized natural rubber [38]

Poly(vinyl chloride) with a  $K$ -value of 580 was kindly supplied by Thai Plastic and Chemicals Co., Ltd.  $N,N$ -dimethylformamide was mixed with tetrahydrofuran at the ratio of 50:50 and was used to prepare the solution of PVC and epoxidized natural rubber at 50°C. The various concentrations of ENR solution were added to PVC solution at room temperature. The electrospinning process was operated at a flow rate of 0.5 mL/h with an applied voltage between 12 and 15 kV using e-spinning machine (eS-robot<sup>®</sup>) capable of generating voltages up to 30 kV at room temperature. The polymer solvent was injected from a syringe tip and

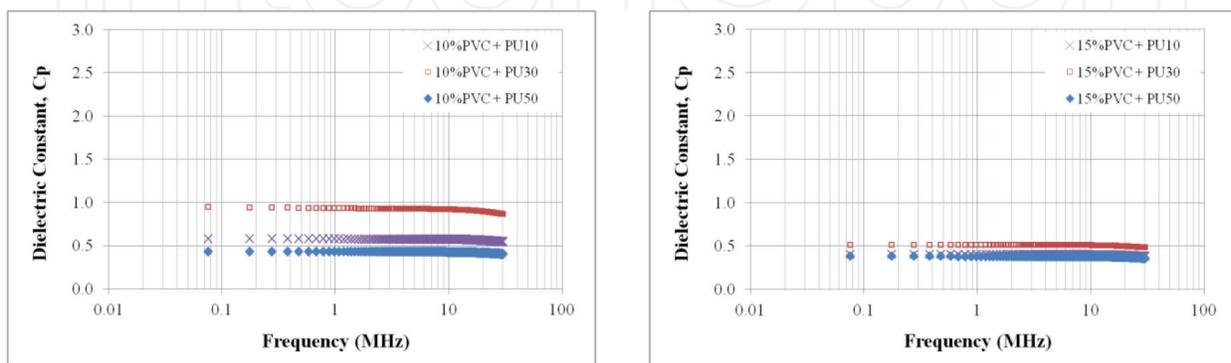
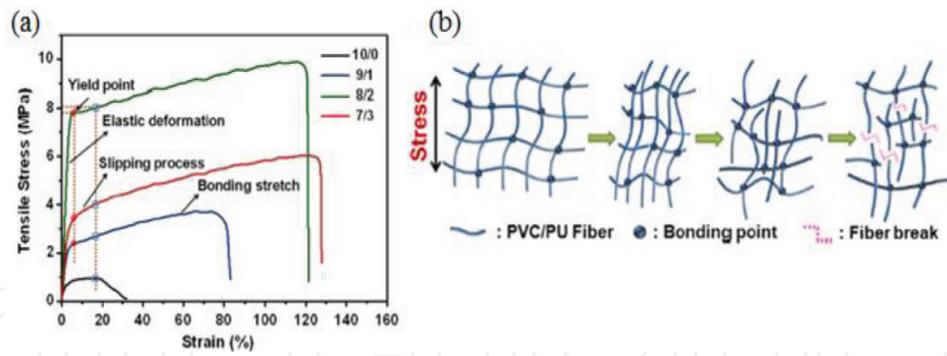


Figure 6. Dielectric properties of nanofibers prepared from PVC blending with PU at difference ratio [38].

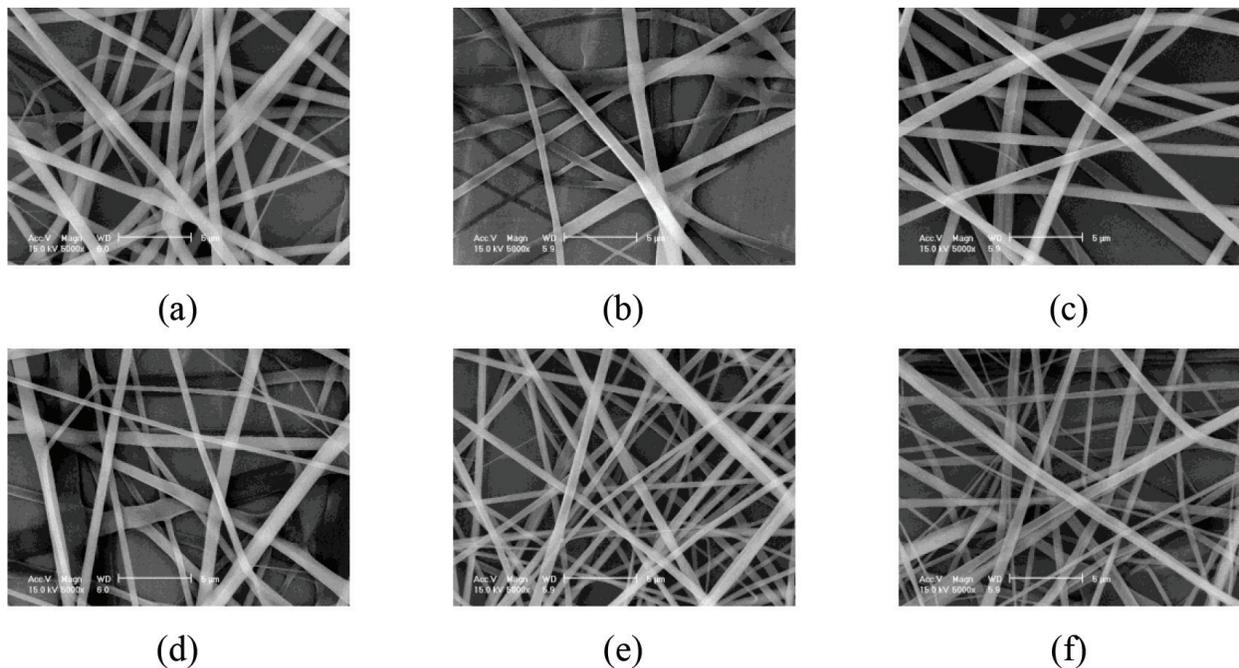


**Figure 7.** (a) Stress-strain curves of PVC/PU fibrous membranes fabricated from varied weight ratios and (b) a proposed three-stage break mechanism of tensile fracture process upon external stress [37].

was deposited on an aluminum collector plate to form the electrospun mats with a thickness of around 0.1 mm.

The morphological structure of PVC/ENR nanofibrous membranes was changed by changing the ENR composition as shown in **Figure 8** [38]. PVC blending with ENR clearly shows that the beads on the fiber disappear as well as the blending of PVC with PU, where ENR composition can be attributed to higher viscosity and surface tension with increasing ENR composition. Viscosity and surface tension play an important role in determining the fiber formability and diameter.

The thermal properties of the PVC nanofibrous membrane and PVC blending with ENR nanofibrous membranes were determined using differential thermal analysis; the results are



**Figure 8.** SEM images of electrospun nanofibrous membrane from PVC blending with ENR (a) 0.7, (b) 1.0, (c) 2.2, (d) 3.0, (e) 4.0, and (f) 5.0 by wt.

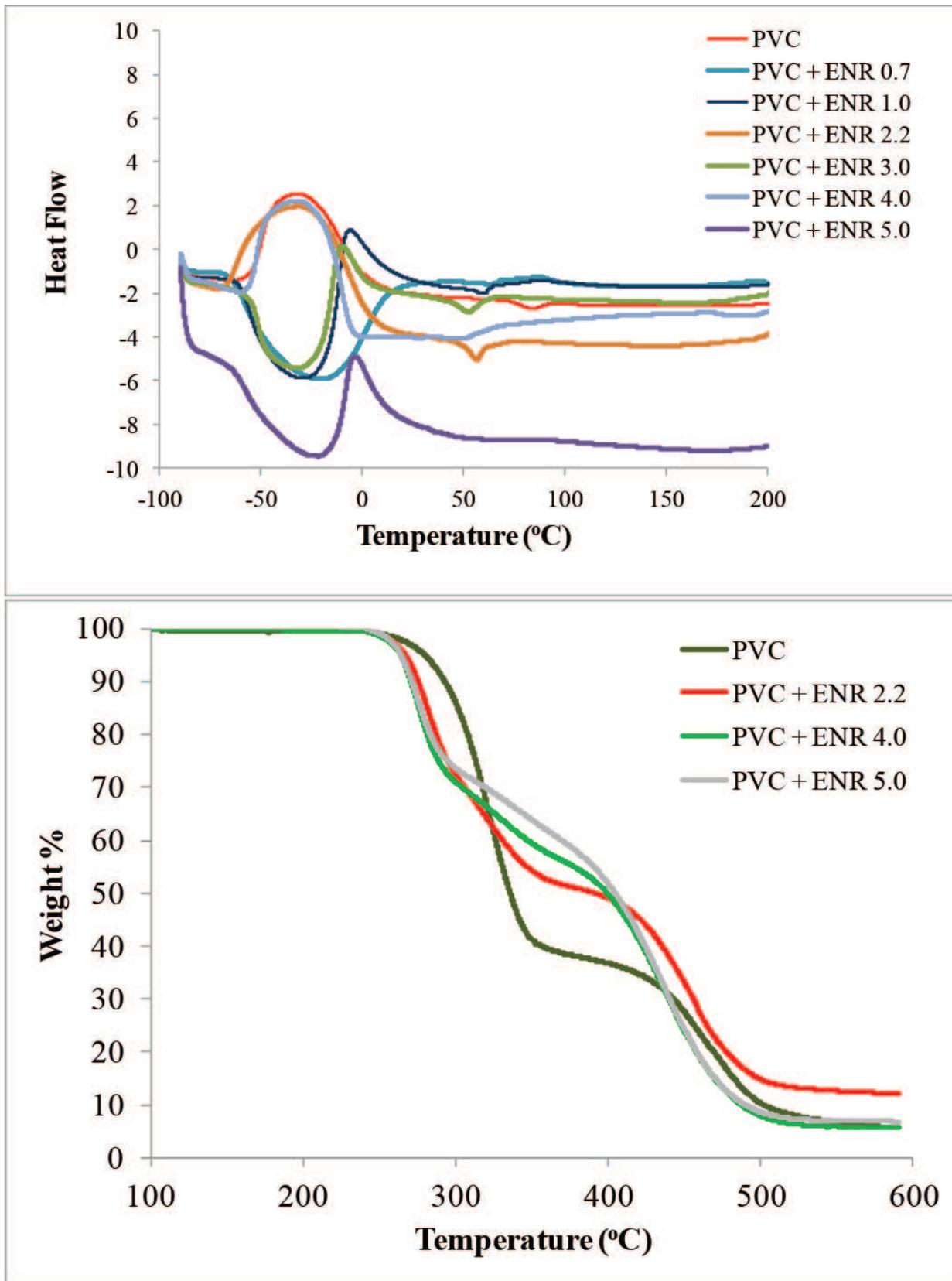


Figure 9. DTA and TGA thermogram of PVC and PVC/ENR blend nanofibrous membrane.

illustrated in **Figure 9** [38]. The PVC nanofibrous membrane, which was prepared from 10 wt% of PVC, is rigid than that from 15 wt% of PVC. The low concentration of PVC solution (10 wt%) shows a high exothermal peak at 30 and 275°C, while a higher concentration of PVC solution (15 wt%) shows an endothermal peak. The thermal properties of nanofibrous membranes were improved after mixing PVC with ENR. The mixture of PVC with high ENR content gave more flexible nanofibrous membranes with a lower glass transition temperature ( $T_g$ ). For higher concentration of PVC solution (15 wt%) with high viscous not easily to mixed with ENR solution as found in PVC/PU nanofiber. The nanofiber spun from higher concentration of PVC solution blended with rubber solution, which was a heterogeneous solution, needs to be optimized based on the electrospinning condition.

The thermal stability of the nanofibrous membrane was determined using thermogravimetric analysis. The thermogram of the PVC and PVC/ENR blends as shown in **Figure 9** has two obvious decompositions, which is basically according to the reported TGA curve. The TGA curve of the PVC/ENR nanofibrous membrane clearly indicated that the blending of ENR with PVC enhances good thermal stability of the nanofibrous membrane.

### 3. Conclusions

The nanofibers of PVC and PVC blending with rubber can be prepared from electrospinning method. The electrospinning condition with an applied voltage of 12kV and different distance between the syringe tip and the aluminum collector plate were optimized. The blending of PVC with rubber using either PU or ENR smoothens nanofiber and enhances desired properties of nanofibrous membrane.

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