Production of Composite Fibers from Natural Rubber and Lignin

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Abstract. The interest of natural rubber (NR) product applications have been increased as natural rubber can be obtained from nature and it can be produce into sustainable products. It's widely used in applications such as tires, wires, latex products, medical devices, and sport components. In this work, the use of electrospinning technique to produce fibers has been carried out because this technique has been proved to be a simple and effective technique for fiber production. This work aimed to study morphology, diameter, and functional group of natural rubber fibers, DPNR fibers, and different loadings of lignin in DPNR/lignin composite fibers produced by electrospinning technique. The condition for electrospun fibers were as following; needle-target distances of 15 cm and flow rate of 2.5 ml/h. The results of deproteinization by urea, SDS solution, and acetone showed protein in NR latex decreased. Moreover, increasing load of lignin resulted in an increase of the diameter of DPNR/lignin composite due to increasing of viscosity of the solution related to increasing of viscoelastic force. Characterizations of these fibers were reported using scanning electron microscopy to examine fibers surface and measure diameter of the fibers. Fourier transform infrared spectroscopy was used to examine the functional group of the fibers.

Keywords: Natural rubber, lignin, electrospinning, fibers, deproteinization.

1. Introduction

Natural rubber (NR) latex is a product from Hevea brasiliensis. In 1876, the seeds of Hevea brasiliensis brought from Brazil were planted in tropical Asia [1]. In present, south east asia (SEA) countries are major plantation and production of NR. The most cultivated countries in SEA are Thailand, Indonesia, Malaysia, and Vietnam [2]. Aye Aye Khin et al., (2019) studies price of NR in world market. The study showed that increasing the production in these countries such as China, India, Indonesia, Malaysia, Sri Lanka, and Thailand have the negative effect of NR on NR price [1]. From above, the production of NR in Thailand led to an increase of quantity of NR and the price of NR in the world market was decreased.

NR is natural polymer and sustainable soft material [3]. It composes of very long molecules of cis-1,4 polyisoprene. Fresh NR latex consists of 94% rubber hydrocarbon and 6% non-rubber components [4]. NR has many good properties such as tear resistance, fatigue resistance, high resilience, excellent dynamic properties [5] and high elasticity due to entropy [3]. The application for the manufacture of NR products including tires, wires, footwear, medical devices, and sport components [6].

Deproteinization of NR can be done by removal protein from NR latex which contains of proteins rubber and non-rubber in the component [7]. Removal protein is important because some protein in natural rubber latex can induce type I allergy with immunoglobulin E when product from natural rubber latex attach with skin of human [8]. The application of deproteinization NR are such as medical materials.

Lignin is second of the most abundant natural polymers, after cellulose. The major industries of lignin production are paper and pulp industries. These industries generated 50 million tons lignin per year [9]. Lignin is an important component of plants. Lignin in softwood contains about 18-25 wt% and hardwood contains about 27-33 wt%. Lignin is a natural polyphenol which is formed from monolignols in plants. The monolignols forming the repeat units of lignin includes para-coumaryl alcohol (H-type), coniferyl alcohol (G-type), and sinapyl alcohol (S-type) [10]. Lignin has many properties such as antioxidant, UV-absorption, anti-fungal, and antibiotic activity [11].

Electrospinning is a technique to produce fibers from organic and inorganic polymer [12]. This method produces fibers with diameters range from micrometers to as low as tens of nanometers [13] by controlling the properties of polymer solutions and the processing parameters [14]. In the process of electrospinning technique, the first droplet of polymer solution can be held at the needle tip by surface tension. When the applied voltage is increased, the interactions of the electrical charges in the polymer solution with the external electric field leads to the droplet deforming into a Taylor cone.

When electrostatic forces overcome the surface tension force, an electrically charged jet erupts. the jet leaves the tip of the high voltage needle to the collector that is grounded [15]. The application of fibers from electrospinning technique such as fiber mats in biomedical industry, flexible scaffolds for cell growth and tissue engineering, and implantable membranes with controlled drug delivery capability [14].

The goal of our study includes functional group, morphology, and diameter of NR fiber, DPNR fiber, and DPNR/lignin (2, 5, and 10 wt%) composite fibers produced by electrospinning technique. The condition for electrospun fibers were as following; needle-target distances of 15 cm and flow rate of 2.5 ml/h. Characterizations included protein content in natural rubber latex, functional group by Fourier Transform Infrared Spectroscopy (FTIR) and morphology by Scanning Electron Microscope (SEM).

2. Materials and Methods

2.1. Materials

NR latex supplied from National Metal and Materials Technology Center. Acetone CH_3COCH_3 99.5% purity was purchased ACI Labscan. Lignin was purchased from SIGMA-ALDRICH Co., USA. Sodium dodecyl sulfate $CH_3(CH_2)_{11}OSO_3Na$ (SDS) was purchased from KEMAUS, Australia. Urea NH_2CONH_2) was purchased from KEMAUS, Australia. Chloroform $CHCl_3$ 99.8% purity from Cambridge Isotope Laboratories.

2.2. Preparation of Deproteinized Natural Rubber (DPNR)

NR latex (supplied from National Metal and Materials Technology Center) consisted of dry rubber 60% w/w (ammonia 0.7% Lauric acid, Tetramethyl Thiuram Disulfide and Zinc Oxide). It was incubated with 0.1 wt% urea (KEMAUS), 1.0 wt% Sodium dodecyl sulfate solution (SDS, KEMAUS) and acetone (C_3H_6O, 99.5%, = 0.79 g/cm^3, purity from ACI Labscan) at room temperature. It was followed by centrifugation at 8000 rpm. After that, the cream fraction was separated. Then, the cream fraction was washed twice with 0.5 and 0.1 wt% SDS solution and acetone, respectively.

2.3. Preparation of NR fibers, DPNR fibers, and DPNR/Lignin Composite Fibers by Electrospinning

NR latex was poured into a petri dish. After that, acetone (C_3H_6O, 99.5%, = 0.79 g/cm^3, purity from ACI Labscan) was poured into cover natural rubber latex in petri dish and dried in oven at 65 °C to remove solvent. Preparation of dry rubber form DPNR was the same method as that of dry rubber from NR. Then, dry rubber (from NR or DPNR) was dissolved in 2% w/w chloroform ($CHCl_3$, 99.8%, Cambridge Isotope Vaboratories) and magnetic stirred for three days. The solution was sonicated for ten mins using an ultrasonic to remove trap air bubbles.

The spinning solution was transferred into a plastic syringe having 15.9 cm needle diameter, equipped with a 20 G needle, and voltage at 20 kV. Electrospun in air at room temperature in the following conditions: feed rate of 2.5 ml/h, needle-target distances of 15 cm for fixed targets, voltage of 20 kV, and load of lignin at 2, 5, and 10 wt%. The fibers were collected in an aluminum sheet. DPNR dry rubber mixing with lignin was dissolved in chloroform and stirred at room temperature for three days. The conditions of the electrospun fibre was the same as that of NR fibers and DPNR fibers

2.4. Characterization Methods

Fourier Transform Infrared Spectroscopy (FTIR; Bruker TENSOR2, Attenuated Total Reflectance (ATR) mode) at wavenumber at 500-4,000 cm^{-1} by scanning five times in a resolution of 4 cm^{-1} to investigate functional group of the NR fibers, DPNR fibers, and DPNR/lignin fibers in different loading was carried out.

Scanning Electron microscope (SEM; Quanta 450, FEI) was used to study the morphology of the fibers. The average fiber size was determined from SEM micrographs using Image J software.

Protein content was reported in water-extractable protein content base on modified Lowry method, using phosphate buffer saline about 40 grams as extractant. Water extractable protein content was median value from three test pieces per sample.

Thermogravimetric analysis allowed to characterize the thermal stability of the samples. The measurements were carried out with the use of a NETZSCH TG 209F3 analyzer. The samples were analyzed under nitrogen atmosphere. The temperature used ranged from 35 to 600 °C at a heating rate of 20 °C/min.

3. Results and Discussion

3.1. Deproteinization of Natural Rubber Latex

Protein content in NR latex and DPNR latex shown in Table 1. Protein content was reported by water-extractable protein content value.

Table 1. Water-extractable protein content of NR latex and DPNR latex.

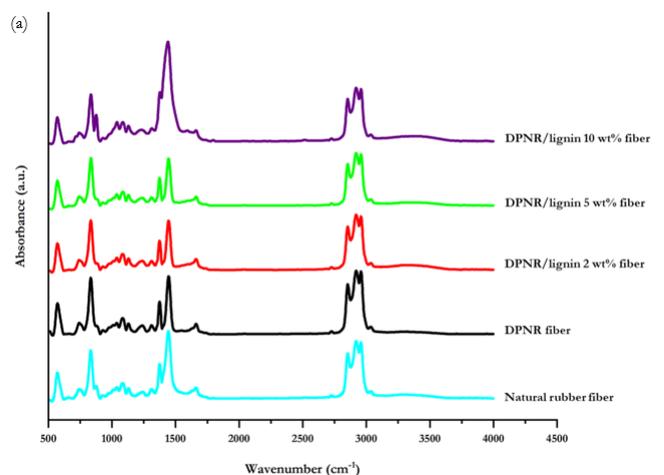| Sample	Water-extractable protein content (micrograms per grams)
NR latex	80.87
DPNR latex	10.31

Water-extractable protein content of DPNR latex was lower than that of NR latex because DPNR latex was purified by urea, SDS solution, and acetone while NR latex had not been purify. Because urea and polar organic solvents can change the structure of the proteins and interact with the lipids, the removal of proteins from

natural rubber by urea was the removal of proteins in NR that attached on the surface of particles with physical interaction [16]. Moreover, SDS solution reduced attractive forces of hydrophobic parts between proteins and rubber on the surface of the particles. Moreover, the centrifugation also help removing protein dispersed in water in NR latex solution [17].

3.2. Chemical Structures by Fourier Transform Infrared Spectroscopy (FTIR)

The chemical structures of NR fibers, DPNR fibers, and DPNR with lignin fibers in different loading were studied by FTIR spectra in the wavenumber range of 500–4000 cm^{-1} as shown in Fig. 1 (a). The wide peak presented in the wavenumber range of 3570–3170 cm^{-1} related to the hydroxyl groups (OH) of lignin [9]. The peaks at 3280 cm^{-1} related to the peptide groups such as the proteins [18]. The peaks at 2960, 1450, and 1377 cm^{-1} belonged to the methylene groups (–CH$_2$) of NR [9]. The peaks at 2922 cm^{-1} and 2850 cm^{-1} related to stretching vibration of C-H in methyl and methylene of NR and lignin [19]. The peak at 1030 cm^{-1} was deformation vibrations of –C–O methoxyl groups of lignin [14]. The peak at 830 cm^{-1} related to the stretching of the C-H bond in NR and lignin [7].

FTIR spectrum peaks at 2960, 1445, and 1377 cm^{-1} were still appeared in NR fibers, DPNR fibers, and DPNR/lignin composite fibers. Figure 1 (b) showed that the absorbance of the peak at 1030 cm^{-1} increased when increasing lignin in the composite fibers. FTIR spectrum of NR fibers had a peak at 3280 cm^{-1}. After deproteinization of NR, the peak at 3320 cm^{-1} appeared to replace the peak at 3280 cm^{-1} as the peak at 3320 cm^{-1} was the spectrum of urea-treated NR [18] as shown in Fig. 1 (c). FTIR spectrum for DPNR/lignin composite showed the peak at 3570–3170 cm^{-1} because lignin has hydroxyl group (OH) as shown in Fig. 1 (d).
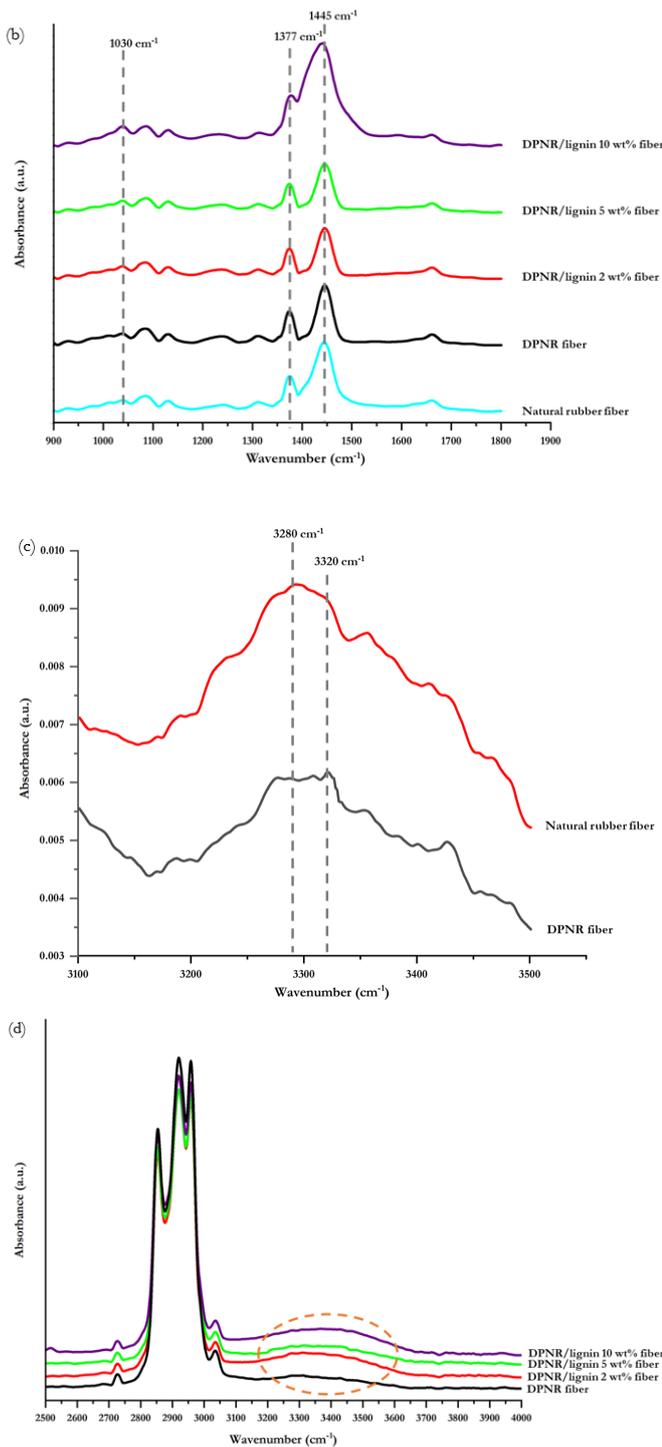
fibers with different amounts of lignin are shown in Fig. 2 and 3.

Table 2. Average diameters of NR fibers, DPNR fibers, and DPNR/lignin fibers.

Sample	Average diameter (um)	SD
NR fibers	4.790	0.301
DPNR fibers	4.752	0.617
DPNR/2 wt% fibers	4.741	0.799
DPNR/5 wt % fibers	4.887	0.693
DPNR/10 wt% fibers	5.026	0.776

Fig. 1. FTIR spectra of (a) the wavenumbers ranging from 4000 to 500 cm^{-1}, (b) the wavenumbers ranging from 1800 to 900 cm^{-1}, (c) the wavenumbers ranging from 3500 to 3100 cm^{-1}, (e) the wavenumbers ranging from 4000 to 2500 cm^{-1}.

Fig. 2. SEM micrographs of (a) NR fibers, (b) DPNR fibers, (c) DPNR/2 wt% fibers, (d) DPNR/5 wt% fibers, (e) DPNR/10 wt% fibers at 1,000 magnification.

3.3. Morphology by Scanning Electron Microscope (SEM)

The average diameter of NR fibers, DPNR fibers and DPNR/lignin fibers with different amounts of lignin measured by image j software are shown in Table 2. The morphology of NR fibers, DPNR fibers, and DPNR/lignin
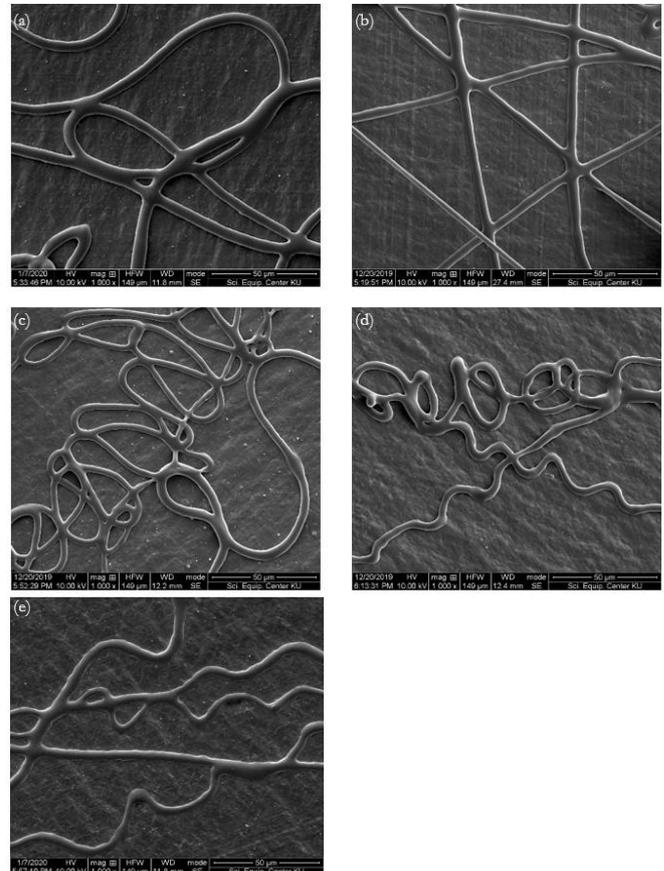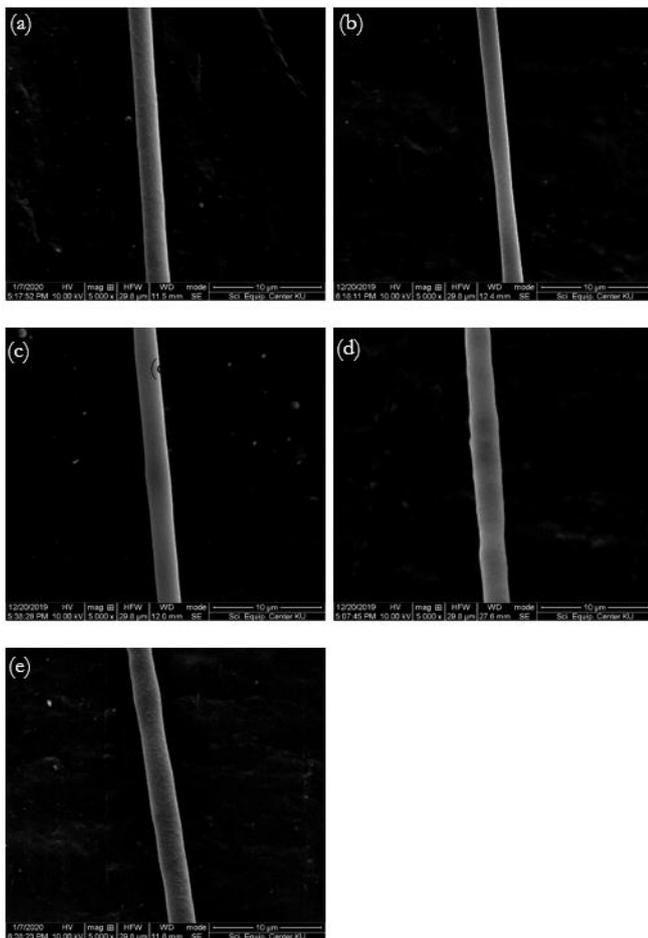
Fig. 3. SEM micrographs of (a) NR fibers, (b) DPNR fibers, (c) DPNR/2 wt% fibers, (d) DPNR/5 wt% fibers, (e) DPNR/10 wt% fibers at 5,000 magnification.

The morphology of fibers was uniform and bead-free for NR fibers, DPNR fibers and DPNR/lignin fibers (2, 5, and 10 wt%) as shown in Fig 2 (a)-(e) and Fig 3 (a)-(e). When adding lignin to the DPNR solution, the viscosity of solution was increased. The composite fibers formed were uniformed without beads due to high viscosity of latex solution. Very low viscosity was not enough to maintain the elongation of the liquid jet. In that case, the thin jet of solution exits the nozzle immediately and shrunk into droplets [20]. The result has the same trend as the fibers from Christian Harito et al., 2016 [21]. The results of adding fillers in the electrospun fibers increased the size diameter of the fibers. The diameter of fibers was increased as the lignin content was increased as shown in Table 1. DPNR/2 wt% lignin fibers have the smallest diameter, followed by DPNR/5 wt% lignin while DPNR/10 wt% lignin fibers have the largest diameter. It was also found that NR and DPNR fibers have similar diameters. This was due to the relationships between viscosity and concentration [22]. Due to the increase in viscosity of high concentration solutions created high viscoelastic forces that withstand the axial stretching during whipping, larger fibers diameter were obtained [23]. The result has the same trend as those in Christopher Drew et al., (2006) [24] and Siqi Huan et al., (2015) [20]

3.4. Thermal Properties

Thermal degradation of NR fibers, DPNR fibers and DPNR/lignin fibers with different amounts of lignin are shown in Table 3.

Table 3. Thermal degradation of NR fibers, DPNR fibers and DPNR/lignin fibers with different amounts of lignin. where $T_{5\%}$ is a temperature of 5% weight loss.

Sample	Thermal degradation $T_{5\%}$ (°C)
NR fibers	320.3
DPNR fibers	345.7
DPNR/2 wt% fibers	341.3
DPNR/5 wt % fibers	338.3
DPNR/10 wt% fibers	336.1
Commercial lignin	294.4

The degradation of NR fibers occured at the decomposition temperature (T_d) at 5% weight loss of 320.3 °C. However, DPNR fibers showed a higher decomposition temperature of 345.7°C because when protein was removed, the spaces between the rubber molecules became more closely. Thus, DPNR behaved a better heat resistance than NR [25]. When the amount of lignin increased, the decomposition temperature decreased because decomposition temperature of lignin was at 294.4 °C [26]. Lignin influence on decomposition temperature of composite fibers.

4. Conclusions

Deproteinization of NR from NR latex by urea, SDS solution, and acetone showed that water-extractable protein content of DPNR latex was lower than that of NR latex. The peak at 3280 cm^{-1} was disappeared while the peak at 3320 cm^{-1} was appeared due to the peak at 3320 cm^{-1} was the spectrum of urea-treated NR. It was found that fibers from electrospinning technique was uniform and bead-free for NR fibers, DPNR fibers, and DPNR/lignin fibers (2, 5, and 10 wt%). The fibers was uniformed without beads because the viscosity of the solution wasn't too low. It was also found that the diameter of fibers decreased when viscosity was decreased. Moreover, the decomposition temperature of DPNR/lignin fibers decreased when load of lignin increased.

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