

As-spun Bio-novolac Fibre Morphological Study based on Resin's Physico-chemical Properties (Kajian Morfologi Gentian Bio-novolak Licin dan Nipis berdasarkan Sifat Fiziko-kimia Resin)

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ABSTRACT

Bio-novolac fibre made from phenol-formaldehyde derived oil palm empty fruit bunch (EFB) was produced using electrospinning method. The bio-novolac phenol-formaldehyde was prepared via liquefaction and resinification at two different molar ratios of formaldehyde to liquefied EFB (LEFB) (F:LEFB = 0.5:1 and 0.8:1). Electrospinning was applied to the bio-novolac phenol-formaldehyde (BPF) in order to form smooth and thin as-spun fibre. The BPF was electrospun at 15 kV and 15 cm distance between needle and collector at a flow rate of 0.5 mL/h. At lower molecular weight of BPF resin, beads formation was observed. The addition of poly(vinyl) butyral ($M_w = 175,000 - 250,000$) has improved the fibre formation with lesser beads hence produced more fibre. Polymer solution with higher molecular weight produced better quality fibre.

Keywords: Electrospinning; molecular weight; oil palm empty fruit bunch; phenolic resin; poly(vinyl) butyral

ABSTRAK

Gentian bio-novolak yang dihasilkan menggunakan fenol-formaldehid terbitan tandan kosong kelapa sawit (TKKS) telah dihasilkan menggunakan kaedah elektroputaran. Bio-novolak fenol-formaldehid telah disediakan melalui pencecairan dan resinifikasi pada dua nisbah molar berbeza iaitu formaldehid kepada TKKS Tercecair (TKKST) (F:TKKST = 0.5:1 dan 0.8:1). Kaedah elektroputaran telah digunakan pada resin bio-novolak fenol-formaldehid (BFF) bagi membentuk gentian licin dan nipis. BFF telah dielektroputaran pada voltan 15 kV dengan jarak 15 cm antara jarum dan pemungut pada kadar aliran 0.5 mL/jam. Pada berat molekul resin BFF lebih rendah, pembentukan manik dapat diperhatikan. Penambahan poli (vinil) butiral ($M_w = 175,000 - 250,000$) telah menambah baik pembentukan gentian dengan kehadiran manik yang berkurang. Larutan polimer dengan berat molekul yang lebih tinggi telah menghasilkan gentian yang lebih berkualiti.

Keywords: Berat molekul; elektroputaran; poli(vinil) butiral; resin fenolik; tandan kosong kelapa sawit

INTRODUCTION

The global production of oil palm was estimated around 64.5 million metric tonnes (Global Palm Oil Production 2016) where Malaysia was the second largest producer after Indonesia with the production of 20 million metric tonnes. The production of the world leading oil palm country; Indonesia was around 35 million metric tonnes. However, in Malaysia, it is reported that the by-product of oil palm which are biomass derived from fronds, trunks, palm press fibre, shells and empty fruit bunch (EFB) were estimated around 90 million metric tonnes. Out of these, Bari et al. (2010) reported that 9% of this biomass has been used as fuel by burning the biomass to generate steam (Kavitha et al. 2013). Hence, this phenomenon has created a serious environmental problem and need further attention. Malaysian National Biomass Strategy 2020 (Agensi Inovasi Malaysia 2016) has highlighted many plans to enhance the utilisation of this biomass into value added products.

Therefore, to ensure the EFB biomass can be fully utilised, various products have been produced using

different techniques such as cellulose hydrogel from dissolving pulp using acid and thermal hydrolysis (Gan et al. 2015), levulinic acid from acid hydrolysis (Chin et al. 2015), anionic and cationic adsorbents via chemical modification (Sajab et al. 2015) and bio-based resin from liquefaction and resinification (Amran et al. 2013).

Biomass can be liquefied using various solvent such phenol (Zakaria et al. 2013) and polyhydroxy alcohols (Doh et al. 2005) for the production of various types of phenolic derivatives and polyol. These monomers can be utilised for the production of phenolic resin (Ahmadzadeh et al. 2008; Hui Pan 2011; Maldas et al. 1997), bio-fuels (Demirbas & Balat 2006) and polyurethane (Noreen & Zakaria 2013) for many applications. Biomass such as from oil palm empty fruit bunch (EFB) (Zakaria et al. 2014), kenaf core fibres (Juhaida et al. 2010) cotton stalks (Said 2010) and corn starch (Moubarik et al. 2009) have been reported and used as raw materials.

Previous report showed that by increasing the reaction temperature to 150°C and catalyst concentration of 8 wt. % of phenol has contributed to the increment of liquefaction

yield and decreased the EFB residue (Ahmadzadeh et al. 2008). Formaldehyde is the most common crosslinking agent used in the production of phenolic resin. Other aldehyde based such as glutaraldehyde and glyoxal has been attempted as the crosslinking agents to replace formaldehyde. However, the properties of these crosslinking agents are far inferior compared to formaldehyde.

Two types of phenolic resin that are novolac and resole can be produced depending on the ratio of formaldehyde to phenol and the added catalyst. Novolac resin is produced with acidic catalyst at a ratio formaldehyde:phenol less than 1 while resole is obtained using alkaline catalyst at ratio beyond 1 (Raquez et al. 2010). Both resins have their own uses in different fields such as adhesive for plywood (Roslan et al. 2014), matrix for glass fibre composite (Amran et al. 2015), resin for automotive moulding like suspension, seats and bottom plates (Plastics Today 2014) and electronic devices by producing electrospun fibre (Imaizumi et al. 2009).

Electrospinning method uses electrostatic force to produce electrostatic polymer fibre from both natural and synthetic polymer with diameter range of 100 nm to several micrometres (Ahn et al. 2006; Hunley & Long 2008; Lannutti et al. 2007; Reneker & Yarin 2008). Electrospinning has received serious and demand attention due to its controllable diameter and pore structure (He et al. 2005; Zussman et al. 2003). The consistency of fibre production in submicron range also attracted researchers to study more about electrospinning process despite of standard mechanical fibre-spinning technologies techniques (Bhardwaj & Kundu 2010). These electrospun fibre have been broadly used in various application such as filtration, optical, sensors and biological scaffolds (Liang et al. 2007) that mostly made from synthetic polymer.

Fibre mat can be produced from bio-phenolic resin using electrospinning device (Ahn et al. 2012). Before and during the process, various parameters such as such as viscosity, concentration and molecular weight of the polymeric solution need to be optimised (Reneker & Yarin 2008). In this study, bio-novolac resin derived from oil palm EFB were electrospun to form novolac fibre. The goal of this study was to determine the effect of resinification temperature of the bio-novolac resin and the addition of PVB on the formation of electrospun novolac fibre.

METHODS

Oil palm empty fruit bunch fibre (EFB) was supplied from Szetech Engineering Sdn Bhd, Malaysia. Phenol and sulphuric acid (97%) were purchased from R&M Chemicals used as liquefied agent and catalyst respectively. Poly(vinyl) butyral (PVB) (Mw = 175,000 - 250,000) was purchased from Shanghai Aladdin Bio-Chem Technology. All chemicals were used without further purification.

EFB and phenol were mixed at a ratio of (1:3) in the presence of sulphuric acid (5% of weight phenol) as reported by Ahmadzadeh et al. (2008). The liquefaction

was carried out in a four-necked glass flask (1000 mL) equipped with a reflux condenser and stirred continuously at 150°C. Then, the reacting temperature were lowered to three different temperature (80, 85 to 90°C) and 37% formalin was added to the liquefied EFB (LEFB) at a molar ratio of 0.5:1 and 0.8:1 for 70 min for the formation of Bio-novolac phenol-formaldehyde resin (BPF). On the other hand, 2-10 wt. % of PVB were dissolved in methanol and added into the BPF resin to increase the molecular weight and adjusted to target a certain viscosity.

BPF was diluted with methanol before the electrospinning. The electrospinning procedure was carried out as shown in Figure 1. A needle syringe (2D) was fixed horizontally on the syringe pump connected with collector plate. Voltage of 15 kV was applied at a distance of 15 cm from the needle tip and the collector with the flow rate of 0.5 mL/h. The process was carried out at room temperature.

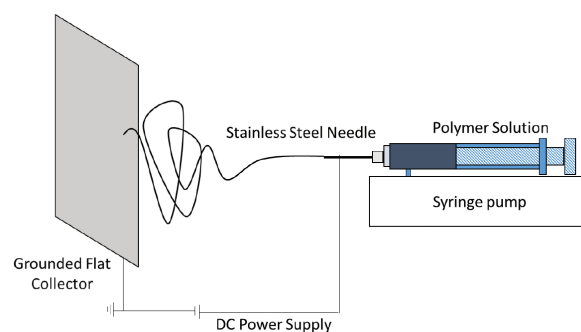


FIGURE 1. Apparatus set-up for electrospinning process

The viscosity of the BPF solution was recorded using Brookfield viscometer. While the molecular weight were measured using gel permeation chromatography model Breeze equipped with Styragel column, HR 5e and calibrated based on monodisperse polystyrene standard, respectively. Samples were dissolved in tetrahydrofuran (THF) and 150 μL sample were injected to the system. As-spun fibre surfaces were viewed under scanning electron microscope, ZEISS EVO MA 10 (UK). All samples were sputter-coated with Au and the analysis was operated at 10 kV. The functional group of BPF and PF were determined by Fourier Transform Infra-Red (FT-IR), Bruker with OPUS software of 7.5 version. Transmittance spectrum of the selected samples were recorded using ATR technique at wavenumber of 4000-800 cm^{-1} , 32 scans and 4 cm^{-1} resolution.

RESULTS AND DISCUSSION

Figure 2 shows the functional groups that exist in the bio-novolac resin (BPF) prepared at different molar ratios but similar temperature. Both spectrums showed same trend and peaks at various parameters thus two of them were selected in this study. The peak at 3360 cm^{-1} was assigned

to the OH-group that is from the contribution of cellulose, hemicellulose and lignin from EFB fibre.

The peaks at 2925 and 2880 cm^{-1} corresponded to methyl and methoxyl group, respectively. As reported previously the functional group were absorbed by aromatic CH and methylene CH (Roslan et al. 2014). The C=C stretching at 1610 and 1480 cm^{-1} indicated methylene bridge at ortho-para position (Yoshida & Okabe 2005). C-C-OH asymmetric stretch of phenolic was detected at 1080 cm^{-1} . Region between 1500 and 1400 cm^{-1} were identified as the deformation vibration of CH bonds on CH_2 (Jianying et al. 2005). At peak 998 and 907 cm^{-1} , both were the contribution of C-O bond in hydroxymethyl group attached to aromatic ring that decreased as methylene bridge formed. In this case, 1,2,4-substituted benzene ring were formed that being absorbed at peak 998 cm^{-1} and 1,2,4,6-tetrasubstituted benzene ring were absorbed at peak 898 cm^{-1} (Roslan et al. 2014). Peak 998 cm^{-1} was absorbed by sample BPF 1:0.5 at 85°C but absence in BPF 1:0.8 at 85°C. This is due to high formaldehyde content which has not reacted with the LEFB. Based on both spectrum, phenol-formaldehyde resin were produced successfully and can be further be used as electrospinning polymer solution.

Table 1 shows the physical properties of bio-novolac phenol-formaldehyde resin. The molecular weight (Mw) of resin obtained from both 0.5:1 and 0.8:1 molar ratios were increased from temperature 80°C to 85°C and

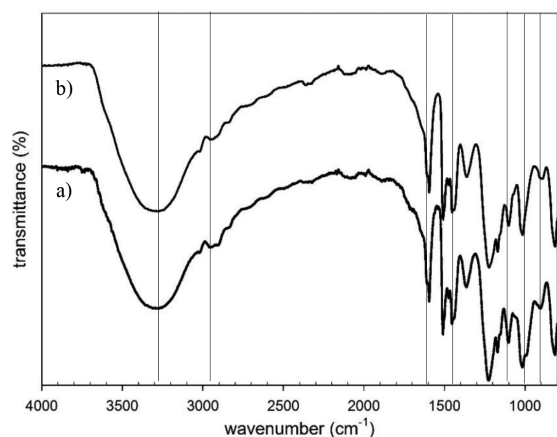


FIGURE 2. FT-IR spectra of a) BPF 1:0.5 at 85°C
b) BPF 1:0.8 at 85°C

decrease at 90°C. This is due to the changes of molecular weight which depend on the amount of combined phenol but independent on the molar ratios (Alma et al. 1995; Zakaria et al. 2013). However, molar ratio of 0.5:1 gave higher Mw compared to 0.8:1 due to the active CH_2 group reduction from formaldehyde (Brydson 1975). It also showed that the higher the Mw, the higher the solution viscosity regardless of the molar ratio. Viscosity increase exponentially with Mw (Robert & Terry 1994). This is due to polymer molecule entanglement and drag along the neighbour molecules under viscous shear forces.

Figure 3(a) and 3(b) shows the electrospun fibres were discontinuously formed for both PF and BPF resin. This could be due to the low molecular weight of polymer solution (Suzuki et al. 2007). At lower molecular weight, the fibre structure was not completely uniform and tends to form bead-on-string structure thus the fibre diameter increased with the increasing of solution's molecular weight (Koski et al. 2004). Beads formation was due to the polymer breakup during injection. The entanglement of polymer macromolecules were stretched and transformed into orientation then solidified the fibre. However, the jet strain had caused the solution to produce beads.

The electrospun BPF resin formed were in beads-shaped and beads-on-string fibres as shown in Figure 3(b)-3(g). This was due to the polymer jet cannot be stretched and elongate. As viscosity, molecular weight and concentration were directly proportional to each other, polymer with low viscosity tends to undergone electro sprayed instead of electrospun thus resulting in beads formation due to the breakage of capillary of the jet by surface tension (Demetrius et al. 2007; Thompson et al. 2007). At high viscosity, polymer solution tends to exhibit longer stress relaxation times which could prevent the fracturing or spraying of the ejected jets (Bhardwaj & Kundu 2010).

From Figure 3, less beads and more fibre were formed as the resinification temperature increased. For sample 2, the viscosity was quite low and the molecular weight were the highest compared to others. Therefore, sample b with molar ratio 0.5:1 at temperature 85°C was used for the addition of high-molecular-weight polymer.

Moreover, solution with higher viscosity gave larger and uniform fibre diameter as shown in Figure 4. The addition of high-molecular-weight polymer like PVB and

TABLE 1. Molecular weight and viscosity of BPF solution before electrospinning

Samples	Molar ratio (F:LEFB)	Resinification temperature (°C)	Molecular weight (Mw)	Viscosity (Cp)
a		80	4241	7.4
b	BPF	85	6314	8.7
c		90	5993	8.6
d		80	3920	6.9
e	BPF	85	5387	8.5
f		90	4415	8.0

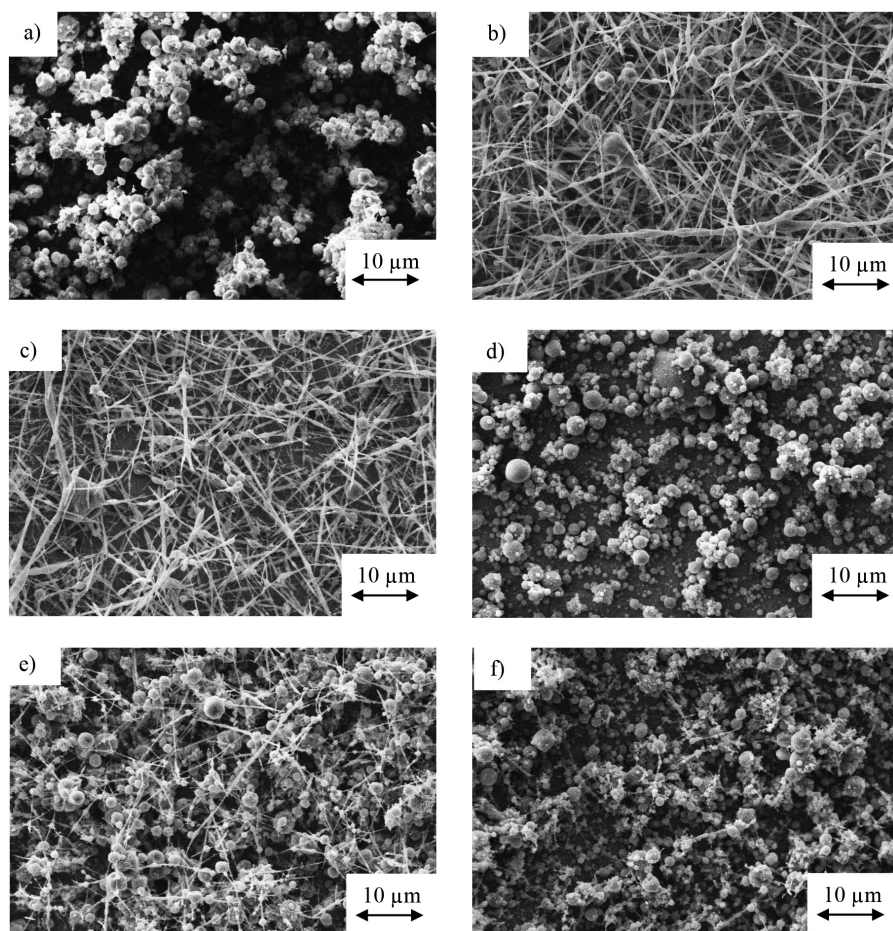


FIGURE 3. Morphology of a) BPF 0.5:1 at 80°C, b) BPF 0.5:1 at 85°C, c) BPF 0.5:1 at 90°C, d) BPF 0.8:1 at 80°C, e) BPF 0.8:1 at 85°C and f) BPF 0.8:1 at 90°C

different concentration had adjusted and increased the viscosity of the polymer solution for electrospinning. As the resin was pumped out from the needle at high voltage, the jet was stretched as it travelled towards the collector. With the addition of PVB and high solution viscosity, the molecule chains entanglement prevented the jet from breaking up and maintained to produce continuous fibre (Ramakrishna et al. 2005). Figure 4(b) to 4(d) shows that as the molecular weight of solution increased, the chance for fibre formation will also increase.

There was no significant difference between the addition of 2% and 6% PVB regarding fibre diameter but the addition of 2% PVB resulting in broken fibre. This is because of the molecular weight of that polymer solution was still low as well as the viscosity itself. The addition of 10% PVB formed bigger fibre diameter which could be due to short distance between needle and collector because the solvent in the solution had inadequate time to evaporate and solidified before reaching the collector (Demetrius et al. 2007).

The addition of PVB has increased the molecular weight of the resin solution. It is a high-molecular-weight polymer in the range of (170,000 - 250,000) that helped improve the fibre formation as PVB showed good compatibility with

phenolic resin (Suzuki et al. 2007). Imaizumi et al. (2009) reported that PVB could improve the electrospinnability by adjusting the solution viscosity (Table 2). Therefore electrospay deposition could be avoided and produced smooth fibre.

From Figure 4(c), the net-like fibre with large number of crossing points dispersed were formed. According to Suzuki et al. (2007), this happened because of too high polymer concentration. Higher solution concentration leads to bigger diameter size followed by beads formation. The coalescence fibre (Figure 4(d) caused by the content of macromolecular PVB that might clustered together in the solution before electrospun (Tian et al. 2016). Furthermore, the fibre diameter of 6% PVB addition was the lowest in the range of 2.826 μm compared to the others (Table 3).

CONCLUSION

The study showed that high resinification temperatures caused less beads formation due to the crosslink reaction of LEFB and aldehyde, leading to higher molecular weight. Molar ratio of F:LEFB = 0.5:1 was the best for modification with the addition of 6% Poly(vinyl) Butyral (PVB) for good fibre formation.

TABLE 2. Molecular weight and viscosity of BPF with PVB solution before electrospinning

	Molar ratio (F:LEFB)	Resinification temperature (°C)	PVB amount (%)	Viscosity (Cp)
BPF+PVB	0.5:1	85	2	18.1
			6	24.6
			10	30.9

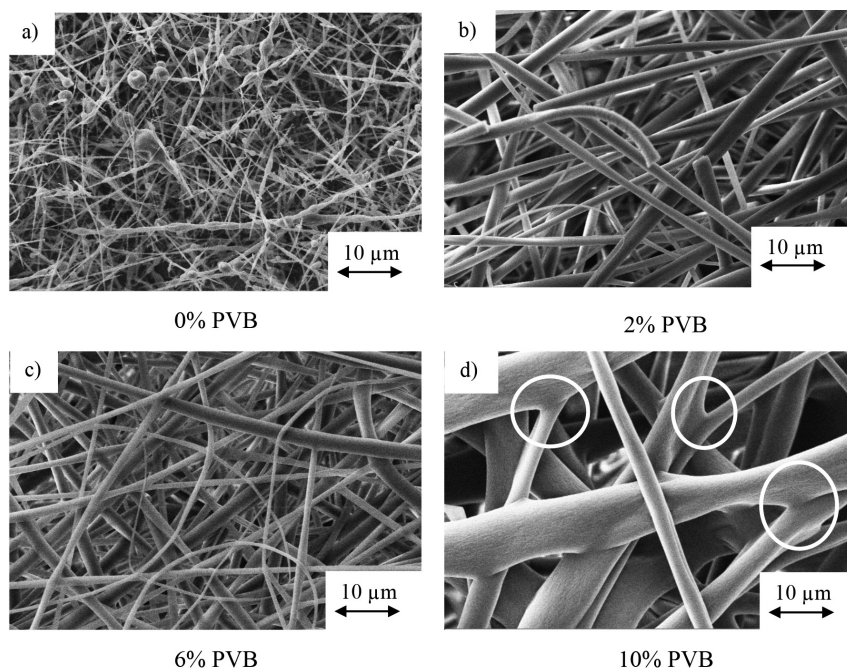


FIGURE 4. Morphology of BPF at molar ratio 1:0.5 at 85°C with the addition of a) 0% PVB b) 2% PVB, c) 6% PVB and d) 10% PVB

TABLE 3. Diameter size of fibre and beads obtained from electrospinning process

Samples	Molar ratio (LEFB:F)	Mean string fibre (µm)	Standard deviation string fibre (µm)	Mean bead diameter (µm)	Standard deviation bead diameter (µm)
BPF	1:0.5	0.441	908.0	3.359	2.595
BPF	1:0.8	0.315	0.047	1.733	0.643
BPF + 2% PVB	1:0.5	3.808	1.223	-	-
BPF + 6% PVB	1:0.5	2.826	0.712	-	-
BPF + 10% PVB	1:0.5	6.244	1.644	-	-

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