Preparation and Characterization of Lignin Polyols from the Residues of Oil Palm Empty Fruit Bunch

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In this work, lignin polyols were prepared from the liquefaction of kraft lignin and from the direct liquefaction of *Elaeis guineensis* lignocellulosic waste. The liquefaction reaction was performed with polyhydric alcohols using sulfuric acid as catalyst at 160 °C. The physical and chemical characterizations of lignin and lignin polyols were conducted by elemental analysis, Fourier transform-infrared spectroscopy, ^{1}H and ^{13}C nuclear magnetic resonance (NMR) spectroscopy, molecular weight distribution, and thermogravimetric analysis (TGA). Quantitative ^{13}C NMR showed that all aliphatic hydroxyl group values of polyols noticeably increased with the use of the two methods compared to kraft lignin. The average molecular weight analysis of the liquefied product showed that it exhibited high molecular weight compared to kraft lignin. Both structural and thermal characteristics suggest that lignin polyols would be a good substitute for kraft lignin in the synthesis of polymeric compounds such as environmentally friendly resins or wood adhesives, as it presents higher amounts of activated free ring positions, higher molecular weight, and high thermal stability.

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INTRODUCTION

Biomass is a promising source of energy that can replace fossil oil and is thus important in sustaining production. It is a renewable resource that is abundant on Earth. The annual production of renewable materials is approximately 20×10^{10} tons. Only 3% of renewable materials are used for non-food manufacturing, such as in the pulp and paper industry (Zhang 2008). Biomass resources are locally produced and in some cases can be considerably cheaper than other resources, such as crude oil and natural gas (Zhang 2008). Biomass materials have a short cycle in terms of the cultivation of plants and the process of burning fuel. Additionally, assuming that the land usages are kept the same, the acquisition of products from biomass does not add carbon dioxide to the atmosphere during a cycle of harvest and regrowth. Many studies have been conducted on the production of environmentally friendly products from biomass polymers, with a primary focus on lignin derivatives for the production of polymeric compounds, especially adhesives such as phenolic and epoxy resins (Zhao *et al.* 2013), and polyurethane material (Zou *et al.* 2012). The remnants of biomass must be eliminated in a systematic and scientific manner because the presence of such residue poses a danger to the environment. An important element

extracted from empty fruit bunch (EFB) is lignin (Mohamad Ibrahim *et al.* 2008). Recently, great efforts have been made to use biomass resources through methods such as gasification, pyrolysis, and liquefaction. Because of its simple preparation, high level of production, high energy conversion, and high capacity of organic materials treatment, the liquefaction process has attracted much attention (Ye *et al.* 2014). The liquefaction process of biomass has progressed in relation to raw materials, solvents, and catalysts. The use of raw materials such as crop residue (Zhang *et al.* 2012) and wood (Rivas *et al.* 2013) has been reported. Liquefaction of wood and other lignocellulosic materials with organic solvents, such as phenols and polyhydric alcohols, is a unique approach to thermochemical conversion in biomass applications because this process combines the useful functional groups of the biomass and organic solvent to obtain a large variety of biopolymers. These biopolymers include coatings (Budija *et al.* 2009), carbon fibers (Xiao and Guang 2010), and adhesives (Fu *et al.* 2006). The liquefied wood can be used as part of an adhesive mixture because of the presence of free hydroxyl groups, which can polymerize with different functional groups to produce various polymers or even to self-crosslink (Budija *et al.* 2009). The liquefied wood adhesives can also be combined with a mixture of synthetic adhesives or with other natural materials, such as tannin (Cuk *et al.* 2011). Lignin has also been used for the production of polyol compounds, which can be employed in the preparation of various types of resins. Two alternative approaches have been developed for the production of polyols from lignin: oxypropylation of lignin with propylene oxide (Cateto *et al.* 2009), and the liquefaction of lignin with polyethylene glycol (PEG) and glycerol (Jin *et al.* 2011). The liquefaction of lignin model dimers with phenol (Lin *et al.* 2001) and of enzymatic hydrolysis lignin with PEG and glycerol have also been studied (Jin *et al.* 2011). In the process of liquefaction, lignin is first degraded into small fragments of lower molecular weight with sulfuric acid as a catalyst under high temperature. The reaction of the hydroxyl groups in the fragments with PEG and glycerol then forms ether bonds in the liquefaction products. Residual lignin self-polymerization occurs in the later stage of the process (Jin *et al.* 2011).

Oil palm empty fruit bunch, which has an annual output of 15 million tons from Malaysia (Rahman *et al.* 2007), was used as the raw material. In this study, the preparation of lignin polyols was successfully achieved using two methods, namely, the liquefaction of oil palm empty fruit bunch and of kraft lignin with polyhydric alcohols. These two methods were compared with each other in terms of structure and thermal stability.

EXPERIMENTAL

Materials

The oil palm empty fruit bunch (*Elaeis guineensis*) long fiber used for kraft pulping was supplied by SabutekSdn. Bhd., a Malaysian company specializing in recycling lignocellulose wastes. Polyethylene glycol (PEG 400) from R & M Chemicals (Essex, UK), and glycerol (HmbG Chemicals, Germany), were used as liquefaction reagents. Pyridine and acetic anhydride were purchased from Merck-Schuchardt (Germany) and used as acetylation reagents. 1,4-dioxane was supplied by HmbG Chemicals, and sulfuric acid was supplied by QRec (Malaysia). All chemicals were of analytical grade and were used without further purification.

Kraft Pulping

The kraft pulping process was carried out in a 4-L digester. Solutions were prepared by dissolving 190 g of NaOH in 1.5 L of distilled water (19 % based on raw material) and 250 g of Na_2SO_3 in 1.5 L of distilled water (25% based on raw material) with a solvent-to-fiber ratio of 8:1. The cooking temperature was ramped from room temperature to 170 °C for 1 h and maintained at 170 °C for 2 h. The contents in the digester were stirred by rotating the digester using a motor. The pressure of kraft pulping was approximately 12 to 15 bar. After cooking, the pulp was washed and separated by screening through a sieve, after which the black liquor was collected.

Kraft Lignin Extraction

The pH of the Kraft black liquor was found to be 12.5. Kraft lignin (KL) was precipitated from the concentrated black liquor by acidifying it using 20% (v/v) sulfuric acid until a pH of 2 was achieved (Mohamad Ibrahim *et al.* 2011). Precipitated lignin was filtered and washed with pH 2 dilute sulfuric acid and then dried in a vacuum oven at 45 °C for 48 h. The purification of lignin was conducted by extracting it in a Soxhlet apparatus for 6 h with *n*-pentane to remove lipophilic non-lignin matter such as wax and lipids. The precipitate was filtered and washed twice with pH 2 dilute sulfuric acid to remove the excess *n*-pentane and non-lignin phenolic compounds that may remain after the pulping process. The purified KL was then further dried in the vacuum oven at 45 °C for another 48 h. KL was treated with hot water to dissolve the residual sugars (Lin 1992). The lignin was then dried in a vacuum oven at 45 °C for another 48 h.

Liquefaction of Kraft Lignin

The liquefaction process was conducted as proposed by Jin *et al.* (2011) with slight modifications. PEG 400 and glycerol were used as liquefaction solvents with a weight ratio of 9:1 and with sulfuric acid as a catalyst (3% wt of solvent weight ratio). KL/liquefying reactants were also applied at a ratio of 1:5. PEG 400 is a low-molecular weight grade of polyethylene glycol (M_n = 380 to 420 g mol^{-1}). Liquefying reactants were placed into a 250-mL three-necked glass flask, immersed in a silicon oil bath, and then heated. The flask was equipped with a reflux condenser and thermometer under constant magnetic stirring in a nitrogen atmosphere. KL was gradually added to the liquefying reactant when the temperature reached 160 °C. The liquefaction reaction was performed for 1 h to obtain a homogeneous liquefied product. The flask was then rapidly cooled in an ice bath to quench the reaction, and the liquefied products were oven-dried at 105 °C to a constant weight before further analysis.

Liquefaction of Oil Palm Empty Fruit Bunch

The air-dried oil palm empty fruit bunch (OPEFB) was ground (20-to 60-wire mesh) using a ball mill. It was then dried overnight at 105 °C in a laboratory oven prior to the liquefaction process. The mass ratio between dried OPEFB and the used solvent was 1:10, in which 3% of the sulfuric acid (based on the solvent content) was added. Then, 30 g of dried OPEFB was placed in a round-bottom flask equipped with a mechanical stirrer, a condenser, and a thermocouple. Next, 9 g of 95% (w/v) aqueous sulfuric acid was added as a catalyst, together with 300 g of PEG (400) and glycerol (GLY). The flask was then immersed in a silicon oil bath that was preheated to 160 °C for 4 h. The flask was then cooled to room temperature in an ice-water bath to quench the reaction. Then, 600 mL of

80% (w/v) 1,4-dioxane aqueous solution was added to the reaction mixture. The resulting mixture was filtered through a filter paper to remove the insoluble parts of the wood. The mixture of 1,4-dioxane and water was evaporated under reduced pressure after filtration. The mixture was obtained using a water pump to acquire the liquefied wood, which contains PEG 400 and GLY. The resulting residual was transferred to a three-necked flask equipped with a mechanical stirrer, condenser, and thermocouple. The flask was heated at 140 °C for 2.5 h. The solution was mixed with 2 L of distilled water with continuous stirring for 30 min after the flask was cooled. The viscous lignin polyols were then collected by centrifugation at 3500 rpm for 15 min, and then PEG/GLY-lignin was obtained by freeze drying.

Analytical Methods

Elemental analysis

In preparing lignins for further analysis, lignin powder was obtained by drying the lignin samples at 105 °C in a vacuum oven until a constant weight was obtained. The contents of carbon, hydrogen, nitrogen, and sulfur were analyzed using a Thermo Finnigan model Eager 300 analyzer (Italy). The percentage of oxygen was calculated by subtracting the C, H, N, and S contents from 100%. The percentage of proteins was calculated as N (%) $\times$ 6.25. Based on the elemental composition $C_aH_bO_cS_d$, the average double bond equivalent (DBE) was calculated according to Eq. 1 (Mohamad Ibrahim *et al.* 2011),

$$DBE = \frac{(2a+2)-b}{2}$$

(1)

where *a* is the molar ratio of carbon and *b* is the molar ratio of hydrogen.

Viscosity

The viscosity of the polyol products was determined by a rotary Brookfield viscometer at 30 °C with spindle 3.

Ash content analysis

Approximately 0.5 g of lignin was weighed into a crucible and calcined at 900 °C for 4 h. The ash content was calculated by determining the weight percentage of the sample in the crucibles as the burning process completed (Mohamad Ibrahim *et al.* 2011).

FTIR spectrophotometer

Fourier transform infrared spectra (FTIR) of kraft lignin and polyol products were obtained from a Perkin Elmer model System spotlight 200 instrument (USA). Each spectrum was recorded in the frequency range of 600 to 4000 cm^{-1}.

Acetylation for GPC and NMR analysis

Lignin and lignin polyols samples underwent an acetylation process to improve their solubility in the organic solvent for nuclear magnetic resonance (NMR) analysis and gel permeation chromatography (GPC). All hydroxyl functional groups were substituted into acetyl groups during the acetylation process. The acetylation was conducted using the method proposed by Young and Lovell (1991). Briefly, 30 mL of a 1:1 (v/v) mixture of pyridine and acetic anhydride was added to 2 g of lignin in a 100-mL conical flask and stirred for 48 h at room temperature. The mixture was then treated with 300 mL of 1%

(w/v) HCl at 0 °C. The resulting precipitate was filtered, washed with deionized water, and dried at room temperature.

NMR spectroscopy

Liquid-state ^{1}H and ^{13}C NMR spectra were obtained with a Bruker Avance 500 NMR spectrometer (USA). The acetylated lignin and lignin polyol samples (150 mg) were dissolved in dimethyl sulfoxide (DMSO-d$_6$) (0.40 mL). Spectral analyses were performed using Bruker software.

GPC analysis

Weight-average molecular weight (M_w), number-average molecular weight (M_n), and polydispersity index (M_w/M_n) were determined by GPC. Tetrahydrofuran (HPLC grade) was used as a mobile phase with a flow rate of 1.0 mL min^{-1} using a Waters 2414 Binary HPLC Pump (USA). Three Waters Stryragel columns of doubled HR 5E THF and HR 4E THF as well as a Waters 2414 Refractive Index detector were used. The columns were calibrated using a narrow-molecular weight monodisperse polystyrene standard ranging from 580 to 8,500,000 g mol^{-1} in the eluent. Lignin and lignin polyol samples were dissolved in the eluent at a concentration of 10 mg mL^{-1}. The solution was filtered using a 0.45-µm filter. Then, 20 µL of the filtered solution was injected into the HPLC system.

Thermal analysis

The thermal behaviors of KL and lignin polyols were studied by thermogravimetric analysis (TGA) using a Perkin Elmer TGA 7 thermogravimetric analyzer (USA). Scans were recorded from 30 to 900 °C with a heating rate of 20 °C min^{-1} in a nitrogen atmosphere.

RESULTS AND DISCUSSION

Table 1 shows the elemental composition, the ash and moisture content, the DBE, and percentage of protein derived from the results of the elemental analysis of untreated KL extracted from OPEFB.

Table 1. Ash and Moisture Contents, Elemental Composition, Protein Content, and Double Bond Equivalent (DEB) of Kraft Lignin

Ash content (%)	8.3
Moisture content (%)	3.79
Elemental analysis (%)	
Carbon (%)	57.83
Hydrogen (%)	3.42
Oxygen (%)	33.66
Nitrogen (%)	0.47
Sulfur (%)	4.62
Protein (%)	2.94
DBE	6.80

The protein and ash contents in the KL (sample a) were low because of the treatment with hot water. The average C9 formulas for KL calculated from the elemental

composition were $C_9H_{6.4}O_{3.93}S_{0.27}N_{0.06}$. The lignin polyols were obtained through liquefaction of KL (liquefied KL with PEG/ GLY) and of OPEFB (liquefied OPEFB with PEG/ GLY) were characterized by spectroscopic, chromatographic, and thermal methods. Additionally, the viscosity of polyol products was 410 cP of liquefied lignin and 380 cP of Liquefied OPEFB.

FTIR Spectroscopy

Fourier-transform infrared spectroscopy (FTIR) was used to determine the changes that occurred in the functional groups of the lignin compounds and their derivatives. The FTIR spectra of the KL and the two polyol samples are shown in Fig. 1. The assignments of the KL spectrum (sample a) are in accordance with a previous report related to the present work (Hussin *et al.* 2013). A large band at ~3460 cm^{-1} was assigned to phenolic and aliphatic OH stretching vibrations, and peaks near 2937 and 2844 cm^{-1} were attributed to CH stretching in the methyl and methylene groups in the side chains and aromatic methoxyl groups. The absorption band at 1030 cm^{-1} can be assigned to aromatic CH in-plane deformation for guaiacyl-type lignin.

Compared to KL (sample a), the FTIR spectra of liquefied KL (sample b) and OPEFB (sample c) reveal an important increase in peak intensity: at ~1091 cm^{-1} (sample b) and ~1099 cm^{-1} (sample c), assigned to aliphatic C-OH groups and ether (C-O-C) groups (compared with a small peak in kraft lignin at ~1030 cm^{-1}); at 3100 to 3700 cm^{-1} (alcoholic and phenolic OH); and at ~2872 cm^{-1} (aliphatic CH bonds).

The marked increase in these peak intensities can be attributed to the structure of PEG and glycerol and could indicate that the liquefaction reaction is caused by a successful chain extension reaction that converts kraft lignin and lignocellulosic feedstock into polyols. These FTIR results for polyol products are in agreement with reports of other researchers (Jin *et al.* 2011) for liquefaction of agricultural residues of biomass.
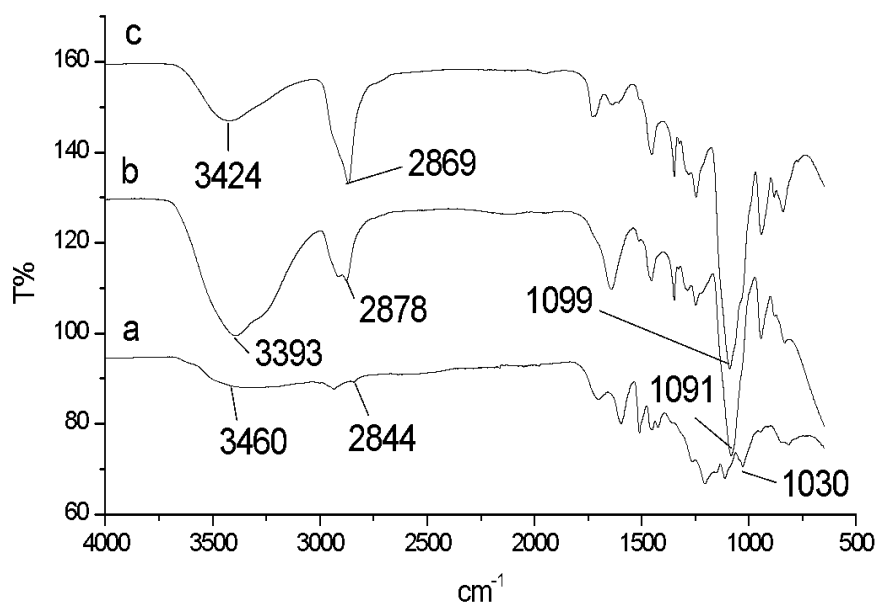
Fig. 1. FTIR spectra of (a) KL, (b) liquefied KL with PEG/ GLY, and (c) liquefied OPEFB with PEG/GLY

¹H NMR Spectra

The chemical structures of acetylated kraft lignin and acetylated polyol products were studied *via* ¹H NMR spectroscopy. Figure 2 shows the ¹H NMR spectra of kraft lignin and lignin polyols. The signal at approximately 2.5 ppm is indicative of protons in DMSO. Figure 2 shows that in the KL (sample a), the signals between 6.5 and 7.5 ppm can be ascribed to aromatic protons in syringyl (S) and guaiacyl (G) units (6.8 ppm aromatic proton in S unit, 7.4 ppm aromatic proton in G unit), respectively, whereas the signals between 0.8 and 1.5 ppm can be assigned to the aliphatic moiety. In addition, aromatic and aliphatic acetyl groups are shifted differently (2.4 to 2.1 and 2.1 to1.9 ppm, respectively). The PEG/GLY-KL (sample b) and liquefaction of OPEFB (sample c) display a marked increase in the peak at region 3.5 ppm compared with kraft lignin. This noticeable increase is caused by the presence of polyethylene glycols, glycerol, and ether linkages (C-O-C). The peaks near 3.8 ppm can be assigned to methyl groups, which also show an increase in intensity compared with the kraft lignin. This observation is in accordance with the increase of the aliphatic C-H band in FTIR. Strong peaks in the 6.5 ppm to 7.5 ppm regions can be assigned to protons from the aromatic rings of KL identified in the lignin polyol samples. These strong peaks in the KL spectrum, however, are quite diluted forboth samples b and c. The peaks observed at 4.5 ppm correspond to protons in hydroxyl groups of the lignin polyols.
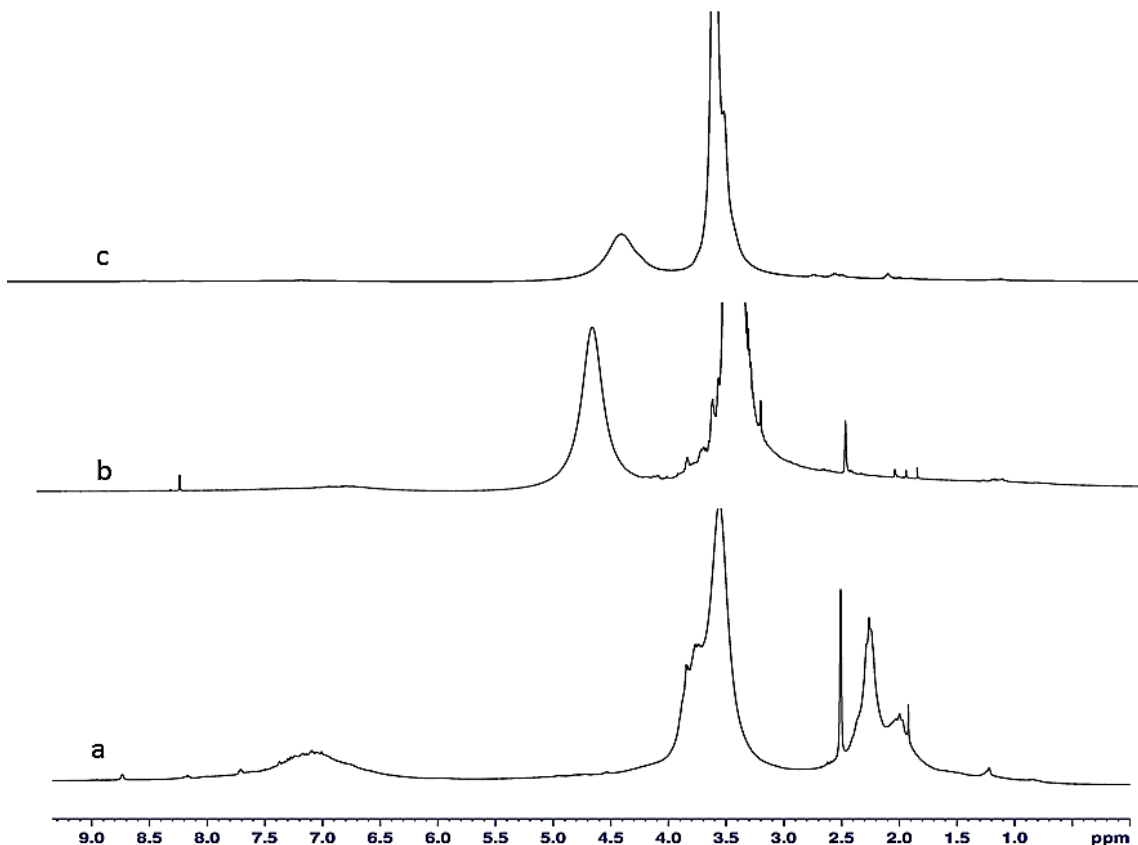
Fig. 2. ¹H NMR spectra of (a) acetylated KL, (b) acetylated liquefied KL with PEG/ GLY, and (c) acetylated liquefied OPEFB with PEG/GLY
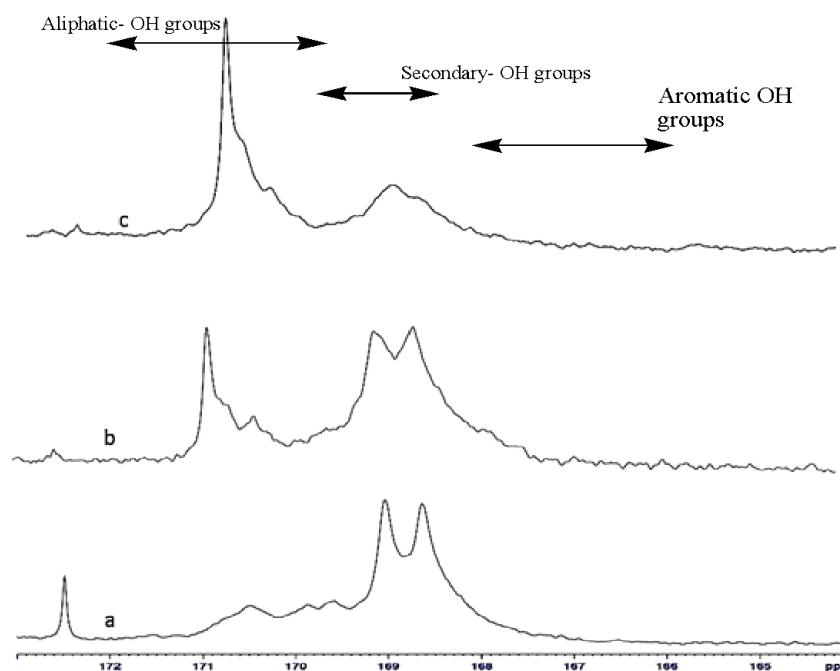
¹³C NMR Spectra

¹³C NMR spectroscopy was used to qualitatively and quantitatively measure the different categories of hydroxyl groups in kraft lignin and polyol derivatives. The discrimination between the primary aliphatic, secondary aliphatic, and phenolic hydroxyl groups was performed after acetylation and is summarized in Table 2. The content of aliphatic and phenolic hydroxyl groups were obtained by integration of the following regions: primary aliphatic hydroxyl groups (172.0 to 169.6 ppm), secondary aliphatic hydroxyl groups (169.6 to 168.6 ppm), and phenolic hydroxyl groups (168.6 to166.0 ppm), as shown in Fig. 3. The content of secondary aliphatic hydroxyl groups noticeably decreased (Jasiukaityte *et al.* 2010). On the other hand, the content of primary aliphatic hydroxyl groups increased from 0.13/Ar (sample a) to 0.28/Ar (sample b) and 0.27/Ar (sample c), indicating that a lignin polyols mixture was formed.

Table 2.¹³C NMR Spectroscopy Analysis of Acetylated (a) KL, (b) Liquefied KL with PEG/ GLY, and (c) Liquefied EFB with PEG/ GLY, Amount (per Ar)

| Sample	(a)	(b)	(c)
Aliphatic- OH			
Primary	0.13	0.28	0.27
Secondary	0.15	0.05	0.03
Phenolic- OH	0.60	0.63	0.66
Methoxy groups	1.11	0.91	0.88

The NMR spectra of acetylated lignin and lignin polyol samples are given in Fig. 4. The lack of signals between 90 and 102 ppm indicates that the concentration of sugars that remained in KL was low and could be considered negligible (Capanema *et al.* 2004). In accordance with the ¹H NMR results, the peaks between 102 and 162 ppm in Fig. 4 assigned to aromatic lignin carbons were diluted in the other samples (b and c).

Fig. 3. ¹³C NMR spectra of expanded hydroxyl region of (a) acetylated KL, (b) acetylated liquefied KL with PEG/ GLY, and (c) acetylated liquefied OPEFB with PEG/ GLY

The methoxy resonance at approximately 56 ppm was sharp in the kraft lignin and decreased along with a noticeable decrease in aromatic signal in the polyol products (samples b and c) (Koda *et al.* 2005). In samples b and c, sharp peaks at 61, 63.5, 69, and 73 ppm were assigned to glycerol and PEG. Interestingly, a new strong chemical shift was also detected around 70 ppm in the liquefied samples. This signal could be assigned to benzyl ether-type groups (Zhao 2013) and may be taken as an indication of a covalent linkage between the α position of the lignin lateral chain and the polyols. The same kind of observation was previously made after formation of a benzyl-alcohol type group by the reaction of glyoxal with ortho site of the aromatic ring of lignin (El Hage *et al*. 2011).
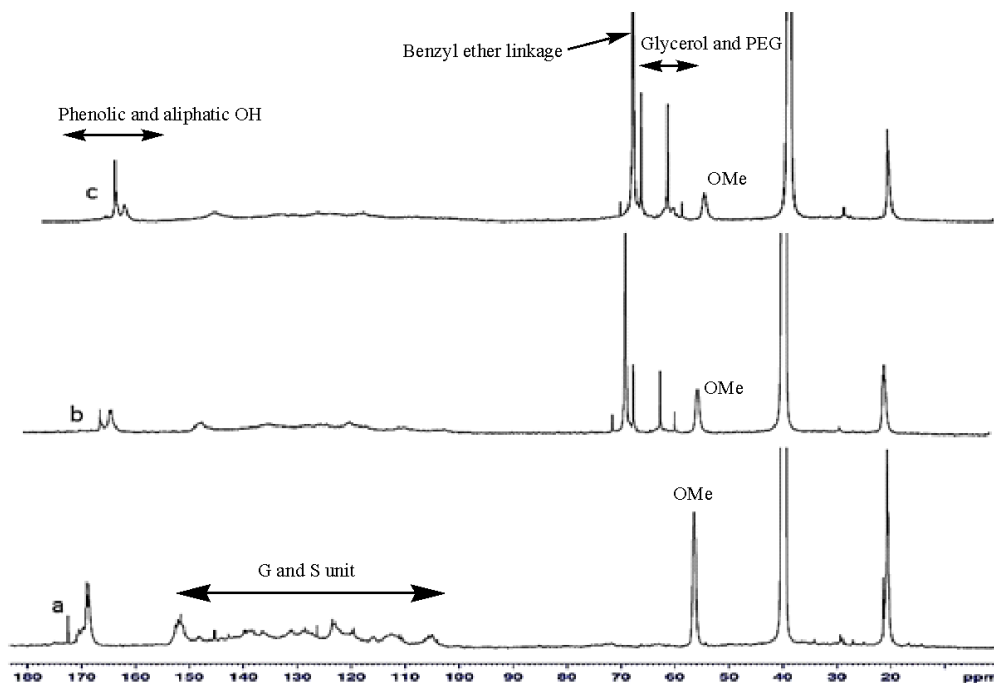
Fig. 4. ^{13}C NMR spectra of (a) acetylated KL, (b) acetylated liquefied KL with PEG/GLY, and (c) acetylated liquefied OPEFB with PEG/GLY

GPC Analysis

The changes in the molecular weight of KL and polyol products were analyzed using gel permeation chromatography (GPC). GPC was also used to determine the effect of the liquefaction process on kraft lignin and lignocellulosic feedstock. The weight-average (M_w) and number-average (M_n) molecular weights and polydispersity (M_w/M_n) of the kraft lignin and lignin polyol samples are shown in Table 3. The increase in M_w and M_n after the liquefaction reaction occurred under acidic conditions. Compared with sample a (M_w, 1564 gmol^{-1} and M_n, 597 g mol^{-1}), a marked increase in M_w and M_n was observed after the liquefaction reaction (M_w, 16730 g mol^{-1} and M_n, 1200 g mol^{-1} for sample b and M_w, 12200 g mol^{-1} and M_n, 1072 g mol^{-1} for sample c).

The increase in the molecular weights of the lignin polyols could indicate that condensation reactions occurred between the lignin and multifunctional alcohols. The high molecular weights of samples b and c were primarily observed in the condensed structures formed by the incorporation of the aliphatic PEG and glycerol moieties into the lignin structure. The high value of polydispersity indicates that the samples had a wide molecular

weight distribution. Most of the research works available in the literature support the utilization of high-molecular weight fractions of lignin for the synthesis of modified phenolic resins (Olivares *et al.* 1988; Van der Klashorst 1989).

Table 3. Weight-Average (M_w) and Number-Average (M_n) Molecular Weights and Polydispersities (M_w/M_n) of (a) Acetylated KL, (b) Acetylated Liquefied KL with PEG/ GLY, and (c) Acetylated Liquefied OPEFB with PEG/ GLY

| Sample	(a)	(b)	(c)
M_w (g mol^{-1})	1564	16730	12200
M_n (g mol^{-1})	597	1200	1072
M_w/M_n	2.62	13.94	11.40

Thermogravimetric Analysis (TGA)

Considerable research has been conducted on the thermal decomposition of lignin (Lin and Dence 1990). The TG and DTG of KL and lignin polyols are presented in Fig. 5A and 5B, respectively. Thermal degradation of KL includes a wide temperature range between 105 and 900 °C. Approximately 38.1% of non-volatile residue for KL still remains in the solid form at 900 °C and is not completely burned. Approximately 62% of the lignin was lost during the TGA analysis.

Studies drawn from the literature review reported that 40% to 60% of lignin is lost through thermal decomposition (Kubo and Kadla 2010). KL (sample a) shows the three thermal cases identified. The first degradation of KL samples from room temperature to approximately 128 °C is characterized by the weight loss of water, carbon monoxide, carbon dioxide, and other volatile products from the breaking of the side chains (Hussin *et al.* 2013). The second thermal degradation, at approximately 280 °C, is characterized by the weight loss of hemicelluloses that are attached to the lignin structure. The hemicellulose was found to degrade at a temperature range of 200 to 300 °C (Garcia *et al.* 2009). A similar phenomenon was observed in earlier reports, thus indicating the existence of hemicellulose as a byproduct of lignin precipitation (Tejado *et al.* 2007).

The maximum thermal decomposition temperature of kraft lignin (DTG_{max}) was approximately 380 °C. This temperature region includes the degradation of the complicated structure of KL, including fragmentation of inter-unit linkages between phenolic hydroxyl, carbonyl groups, and benzylic hydroxyl, which releases monomeric phenols into vapor phase (Alriols *et al.* 2009).

Lignin polyol (samples b and c) exhibited slightly greater thermal stability than KL. Samples b and c also showed significantly similar features of the weight loss and maximum decomposition temperature. DTG curves of polyols exhibited peaks ranging from 147 to 311 °C and 311 to 410 °C (sample b), and from 168 to 316 °C and 316 to 410 °C (sample c). These results are in accordance with those of Briones *et al.* (2012) on the DTG analysis of pure PEG-glycerol, which presented a glycerol decomposition from 153 to 267 °C and PEG decomposition from 267 to 392 °C. The results of weight losses in the lignin polyol samples were relatively high, at 89.8% for sample b and 86.9% for sample c. The higher weight loss of the polyol products may be attributed to the presence of the moieties of PEG and glycerol. This thermal behavior supports the hypothesis of the use of lignin polyols in the applications of phenolic resins within a wide temperature range.
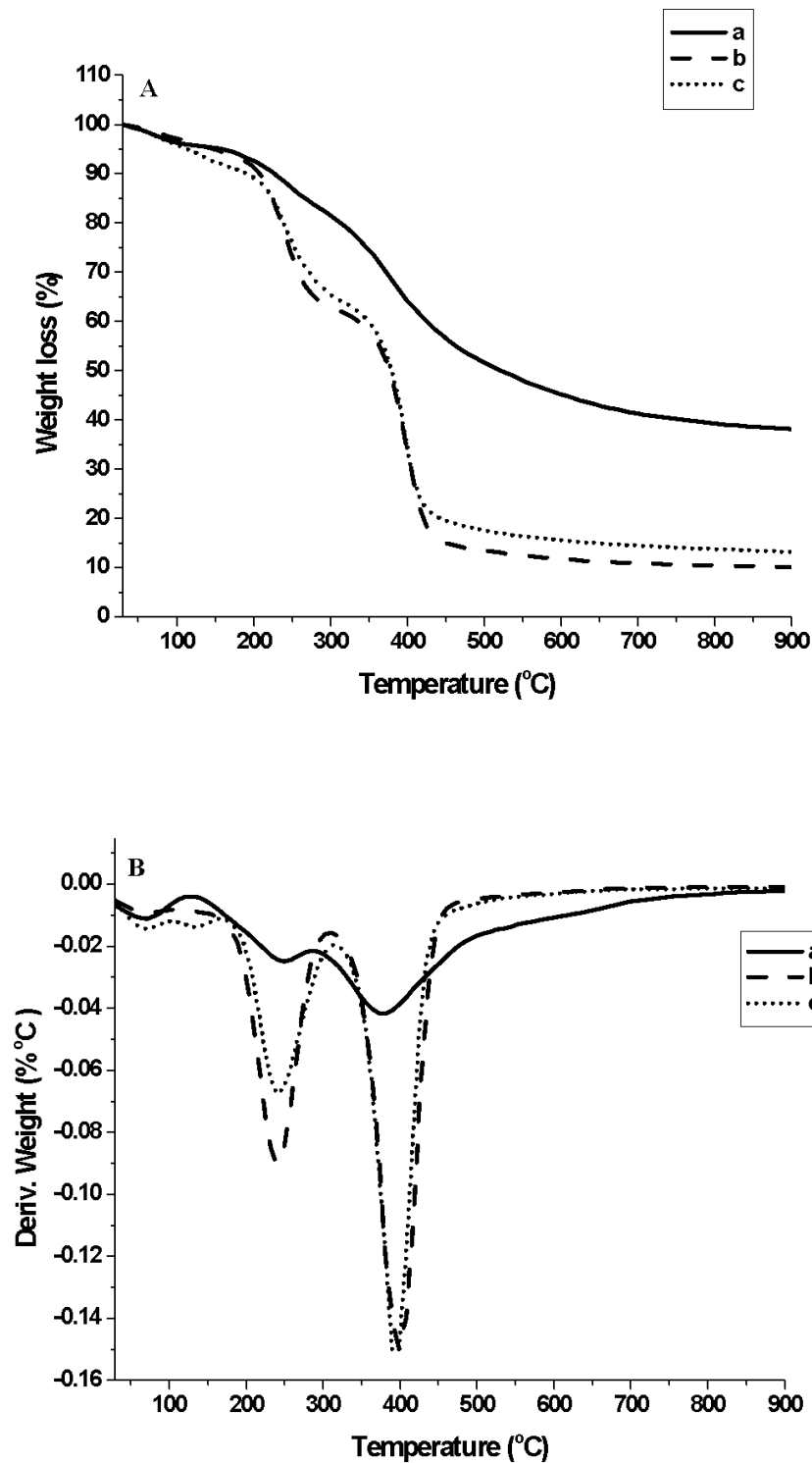
Fig. 5. (A)TG curves and (B) DTG curves for (a) KL, (b) liquefied KL with PEG- GLY, and (c) liquefied OPEFB with PEG/GLY

CONCLUSIONS

1. Viscous lignin polyols were prepared by a liquefaction process using kraft lignin extracted from oil palm empty fruit bunch and fibers of oil palm empty fruit bunch. The products were successfully characterized using FTIR, ^{1}H, and ^{13}C NMR spectroscopy, as well as GPC.

2. Molecular weight distribution patterns demonstrated the change in molecular weight of the polyols, which is a major confirmation of the successful preparation of targeted lignin polyol products.

3. The resulting products are promising and economically feasible. Furthermore, lignin polyols can be used as an alternative to petroleum resources for the production of natural phenolic resins and some other important polymers, as they are more effective than kraft lignin.

4. The molecular weight of the reported polyols is suitable for preparing phenolic resins along with feasible thermal stability. However, liquefaction processes for the preparation of natural phenolic resin warrant further studies.

ACKNOWLEDGEMENTS

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