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Preparation of bio-polyol via bamboo wastes liquefaction and the effects of bleaching by hydrogen peroxide treatment

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Abstract

In this study, polyols have been prepared via liquefaction of wastes of four types of bamboo, namely, *Dendrocalamus asper* (Betong), *Gigantochloa levis* (Beting), *Bambusa vulgaris* (Minyak), and *G. scortechinii* (Semantan). The effects of reaction temperatures and times on the yield percentage, hydroxyl number and viscosity were investigated. The study revealed that under a temperature of 150 °C and a duration of 60 min, the most optimum results were achieved, including a yield of 94.59%, a hydroxyl number of 342.83 mg KOH/g, and a viscosity of 231.60 cP. The study also suggests that a mixture of bamboo wastes can be used for the liquefication process to obtain a comparable result with bamboo waste of single species, which is more practical for the industries to adopt. The polyols produced were dark brown in colour and they were undergone bleaching process using hydrogen peroxide with potassium carbonate serving as the activator. The colour of the liquefied bamboo polyol was successfully changed to a light yellowish tone by adding 60% hydrogen peroxide and stirring for a period of 12 h. Fourier Transform Infrared (FTIR) results showed that bleached and unbleached bamboo polyols only showed slight distinctions indicates that the chemical composition and structure of the untreated liquefied bamboo did not undergo significant changes as a result of the bleaching process.

Keywords Malaysian bamboo, Wastes, Bleaching, Reaction temperature, Reaction time

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Introduction

Lignocellulosic biomass is a renewable, abundant, and inexpensive source of raw materials for the chemical industry to use in the development of biofuel, chemicals, and biomaterials [1]. Following this trend, several lignocellulosic materials has been successfully modified and been widely used for the production of bio-polymer materials [2–7]. Biomass liquefaction in the presence of polyhydric alcohols and acid catalyst is an efficient thermochemical conversion method for converting solid biomass into liquids for various applications [8]. A wide range of virgin and waste biomass, including wood, bark, cork, corn barn, bagasse, and agriculture crop residue, has been used in liquefaction [9–12].

During liquefaction process, the main chemical component in wood, i.e., hemicellulose, is easily degraded and hydrolysed. Lignin and cellulose, on the other hand, are initially degraded and decomposed to smaller fragments, which then react with themselves, or with the solvent to form higher molecular fragments [8]. In addition to the unreacted solvent present in the liquefaction products, these fragments will yield a significant quantity of active hydroxyl groups (referred to as polyols). These polyols can be utilised in various systems, such as polyurethane (PU), epoxy, and formaldehyde-based systems. The liquefaction products, which have abundant raw materials, high hydroxyl group content, and a stable aromatic polymer structure, are a promising source of bio-polyol [8]. Thus, the selection of appropriate raw materials and liquefaction parameters, such as liquefaction solvent, catalyst, temperature, and time, was crucial to achieve a better conversion degree and improve physical and chemical properties, among other factors.

The encouraging results shown by these studies have prompted the current work in using bamboo mill residues for the production of inexpensive polyols. Nonetheless, all the studies have reported that the liquefied products they produced have dark brown to black colour which may have been contributed by the degradation of polyphenolic lignin, cellulose and sucrose [13]. The dark colour may restrict its applications particularly where aesthetic appearance is important. Cheumani-Yona et al. [13] gave three thoughts to be the causes of this strong coloration. First, as a result of the liquefaction's carbon–carbon double bonds (C=C) and carbonyl groups (C=O). Second, the polymerization process during liquefaction process creates high molecular weight compounds from molecules, such as 5-hydroxymethylfurfural, 2-furaldehyde, 2-methyl-3-hydroxy-4H-pyran-4-one, and 2-hydroxyethyl levulinate, mainly from cellulose, the main wood component. Third, when liquefaction is conducted at higher temperatures (350 to 500 °C), it is possible for char to develop, consequently darkens the colour of the liquefied mixture.

Hydrogen peroxide (H_2O_2) treatment has been used since the 1930s to successfully modify wood colour. H_2O_2 has been used principally for bleaching. It is attractive as a bleaching agent for secondary fibres which most frequently used for high-yield pulp bleaching when high levels of brightness are required. For a mixed wastepaper furnish of old newspaper (ONP) and old magazine (OMG), bleaching has some similarity to mechanical pulp bleaching. H_2O_2 is known to act as a strong oxidizing agent both in basic and acidic mediums. Therefore, when it is been added to the material it helps in breaking the chemical bonds of the colour-producing agents (i.e., chromophores). This part of study reports on the use H_2O_2 to treat liquefied polyol to give it a brighter colour. H_2O_2 is chosen, because it is efficient, quite inexpensive and environmentally friendly. The oxygen-rich molecules of H_2O_2 could perform delignification effectively [14].

Conversely, very few studies have reported on the use and influence of H_2O_2 in the bleaching of polyol [13, 15, 16]. According to Mehats et al. [17], hydrogen peroxide by itself is a weak oxidizing agent without the presence of activators. Besides, peroxide primarily works under alkaline conditions, while bio-based polyols are in acidic conditions [14]. Several activator/hydrogen peroxide systems have been created and employed in organic synthesis, polymerization, oxidation–degradation, such as potassium carbonate, magnesium sulphate, sodium bicarbonate, and sodium hydroxide [16–18] Most of activators act as a buffering agent and helps to maintain the pH of the solution. In this work, potassium carbonate was selected as the oxidation system's activator due to its simplicity, low cost, relative lack of toxicity and commonly employed in Totally Chlorine Free (TCF) bleaching sequences [17, 18]. Besides, the effectiveness of potassium carbonate in improving the brightness and colour of the pulp is relatively better than sodium hydroxide, and at the same time reduce the amount of organic compounds generated during the bleaching process [19].

Bamboo has a significant economic impact in Malaysia, with more than RM 8 million in annual export value of bamboo and bamboo-based products. The local bamboo industry generates a huge amount of residues every day during the strips processing process. In this study, bamboo wastes/residues from four bamboo species: *Dendrocalamus asper* (Betong), *Gigantochloa levis* (Beting), *Bambusa vulgaris* (Minyak), and *Gigantochloa scortechinii* (Semantan) were used to produce polyols. The objectives of this part of study were to determine the effects of temperature and time on the properties of bio-polyols from liquefied bamboo and investigate the effect of

activated hydrogen peroxide on the extent of discoloration of liquefied bamboo polyols.

Experiment

Materials

The residues of four bamboo species that are commonly found in the local bamboo industry were purchased from a local bamboo processing factory in Sungai Senam, Sik, Kedah, Malaysia. The four bamboo species were *Dendrocalamus asper* (Betong), *Gigantochloa levis* (Beting), *Bambusa vulgaris* (Buluh Minyak), and *Gigantochloa scortechinii* (Semantan). Polyethylene glycol (PEG 400), glycerol, sulphuric acid (95%), methanol (99.8%), hydrogen peroxide (H_2O_2) at 30% v/v, sodium hydroxide 0.5 N, potassium carbonate, 1,4-dioxane, pyridine, and phenolphthalein were supplied by R&M Chemicals, India. Phthalic anhydride was supplied by Sigma-Aldrich, Germany. Filter papers grade 4 qualitative was supplied by Whatman, United Kingdom.

Liquefaction of bamboo wastes

The reaction was carried out in a 500 mL round bottom flask with a 9:1 mass ratio of polyethylene glycol (PEG400) and glycerol (Gly) as the reactive co-solvent. Sulphuric acid was used as catalyst. The 9:1 PEG/Gly ratio was chosen based on previous research that found it to be an optimal ratio for wood liquefaction [15, 16, 20]. The amount of bamboo sawdust used was 15% wt. of the co-solvent and the catalyst-to-solvent mass ratio is 3%. PEG400, glycerol and bamboo sawdust were charged into the round bottom flask and refluxed under a mechanical stirrer at the desired reaction temperature. After reaching the desired temperature (120, 130, 140, 150, or 160 °C), sulphuric acid was added dropwise and continuously stirred for the desired reaction time (60, 90, and 120 min). The liquefied mixture was cooled down and diluted to ten times its weight with methanol to stop further reaction. The insoluble bamboo residue in the solution was filtered under vacuum through a porcelain Buchner funnel with Whatman™ filter paper grade No. 4 (25 µm). The liquefaction yield (LY) was determined as follows:

$$\mathrm{LY}\ (\%) = \left(1 - \frac{\mathrm{W_r}}{\mathrm{W_o}}\right) \times 100$$

where W_r is the mass (g) of the liquefaction residue and W_o is the initial dry bamboo sample.

Bleaching of liquefied polyol

The bleaching of polyol was carried out in two stages with various modifications, as suggested by Cheumani-Yona et al. [15] and Cheumani-Yona et al. [13]. The parameters of the study included varying amounts of hydrogen peroxide (w/w 20%, 40%, 60%, and 80%) and different reaction times (12 h and 24 h). Liquefied bamboo polyol (30 g) was diluted in a 500 mL conical flask with a mixture of 100 mL of 1,4-dioxane and water (4:1 v/v) before 1.5 g of solid potassium carbonate was added. At room temperature, hydrogen peroxide was added dropwise and stirred continuously at 1000 rpm. The colour of polyol was gradually changes over time. Following the completion of the designated reaction time, a 40% aqueous solution of sodium hydroxide (NaOH) solution was employed to adjust the pH of the reaction to approximately 8.5. After the treatment, hydrogen peroxide were removed by filtering under vacuum via a Buchner funnel (porcelain) using Whatman filter paper No. 4. The solvent in the filtrate was evaporated at 80 °C under reduced pressure. The discoloured liquefied polyol sample was redissolved in a fresh solution of 1,4-dioxane and water (4:1 v/v), and the pH was adjusted to 4 to 4.5 using a solution of sulphuric acid (5 M) to allow additional salt precipitation, and filtered. The solvent once again was evaporated under reduced pressure. A sample of a discoloured liquefied polyol was weighed after being dried at 80 °C for 24 h prior to characterisation.

Characterisations of unbleached and bleached polyol

Chemical composition of the bamboo powder

The chemicals analysis was carried out according to TAPPI standards: T 222-74 for lignin content, TAPPI Standard: TS os-73 for extractive content, TAPPI Standard: T 203 os-61 for α-cellulose, TAPPI Standard: T 265 om-88 for moisture content (MC), and the procedure of TAPPI standard T 204 cm-97 was used for the holocellulose analysis. In addition, the pH and ash content of bamboo were also determined. All tests were carried out in triplicates.

Hydroxyl number

Polyols are identified by their hydroxyl number, which is the amount of hydroxyl groups available for the reaction. The hydroxyl number represents the quantity of milligrams of potassium hydroxide (KOH) that corresponds to the active functions (hydroxyl content) of 1 g of the compound or polymer. The hydroxyl number of the polyol was determined according to ASTM D 4274-21 [21].

A mixture of 1.9 g of liquefied bamboo polyol and 25 mL of phthalic anhydride–pyridine reagent is kept in an oil bath at 115 ± 2 °C for 1 h. After heating, the mixture was cooled to room temperature and 0.5 mL of phenolphthalein indicator solution was added. Then, 0.5 N NaOH solution was titrated into the mixture until the colour of the mixture turned pink. The titration was terminated when the pink endpoint remained for at least

15 s. The blank solution was run in the same way as the sample solution. The OH numbers in mg KOH/g of sample were calculated as follows:

$$\text{Hydroxyl number} = [(B - A)\, N \times 56.1]W$$

where A is the amount of NaOH solution for titration of polyol sample (mL); B i the amount of NaOH solution for titration of the blank sample (mL); N is the concentration of NaOH solution, 56.1 (g/mol) is the molar mass of KOH, (mol/L); and W is the polyol sample mass (g).

The acid number of polyols was determined using the ASTM D662-20 [22]; 0.5 g of polyol sample, 60 mL pyridine, 10 mL distilled water, and 0.5 mL phenolphthalein solution were weighed into two 250 mL Erlenmeyer flasks and mixed with magnetic stirrers. The mixed solution was titrated to a pink endpoint with standard 0.1 N potassium hydroxide (KOH) solution. Each sample was examined in triplicate, and the average value was displayed. The acid number was calculated as follows in mg KOH/g:

$$\text{Acid number} = [(A - B)\, N \times 56.1]W$$

where A is the amount of KOH solution for titration of polyol sample (mL); B is the amount of KOH solution for titration of the blank sample (mL); N is the concentration of KOH solution, 56.1 (g/mol) is the molar mass of KOH, (mol/L), and W is the polyol sample mass (g).

Viscosity

The viscosity of liquefied bamboo polyol (in centipoises) was measured at 25 °C using a Brookfield Viscometer LVT. The measurement was taken with spindle number 31 and a speed of 200 rpms.

Fourier transform infrared spectroscopy (FTIR)

The liquefied bamboo polyol was analysed using FTIR spectrometer (Thermo Scientific-Nicolet iS10) in the 4000–400 cm^{-1} range. For the IR measurement, a drop of sample was placed directly on the KBr disc.

Gel permeation chromatography (GPC)

Gel permeation chromatography (HPLC–GPC: WATERS) with Phenomenex column (300×7.8 mm) was used to measure the average molecular weight of liquefied bamboo polyol. The estimation of the number–average molecular weight (Mn) was conducted by utilising polystyrene standards on the calibration curve. The mobile phase used was tetrahydrofuran (THF) at a flow rate of 1.0 mL/min for 1 mg sample.

Colour change

It is known as Delta (Δ) and it represents the variation in absolute colour coordinates. Each measurement of the sample colour and the standard colour should be recorded. The liquefied bamboo polyol samples were put on white paper and placed between two glass plates and was measured with a Color Reader CR-10 (Minolta) Japan. The *CIELAB* parameters (L^*, a^*, and b^*) was used for the study and the value of three colorimetric parameters L^* represents the brightness of the sample, and it varies from 0 (black) to 100 (white); a^*represents the green ($-a^*$) to red ($+a^*$) axis, and b^* is the blue ($-b^*$) to yellow ($+b^*$) axis. The colour change (ΔE^*) of the liquefied bamboo polyol after discolouration reaction is given as follows:

$$\Delta E^* = \sqrt{(L^*_2 - L^*_1)^2 + (a^*_2 - a^*_1)^2 + (b^*_2 - b^*_1)^2}$$

where ΔE^* is the total difference; L^*_1, a^*_1, b^*_1; the *CIELAB* colour values of the polyol (L, a and b) before discolouration reaction; L^*_2, a^*_2, b^*_2; the *CIELAB* colour values of the polyol (L, a and b) after discolouration reaction.

The target was to achieve the lowest possible ΔE^* value which indicates polyol having the lightest colour.

The calculation for the mass loss throughout the process was as follows:

$$\text{Mass loss (\%)} = \frac{W_i - W_f}{W_i} \times 100$$

where W_i is the masses (g) of liquefy polyol before discolouration reaction; W_f is the masses (g) of liquefy polyol after discolouration reaction.

The amount of water in the polyol was calculated using a moisture analyser, specifically the Shidmazu–MOU63u model.

Results and discussion

Chemical content of the bamboo powder

According to Table 1, the chemical content in four species varied from 72.3 to 84.8% for α-cellulose, from 24.74 to 32.65% for hemicellulose, and from 24.41 to 30.36% for lignin content. Kurimoto et al. [23] reported different percentages of cellulose, hemicellulose, and lignin in Japanese softwood and hardwood. Nevertheless, these variations did not have any noticeable impact on the degree of liquefaction. The authors state that all wood species that were used to prepare the PU film were successfully liquefied. These wood species were then transformed into polymer networks through co-polymerisation with polymeric methylene

Table 1 Chemical composition of the four bamboo species used in this study

Species	Lignin	Holocellulose	α-Cellulose	Hemicellulose	Extractive	Ash content	Moisture content
D.asper (B.Betong)	28.67^{B} (0.28)	72.31^{D} (0.31)	45.55^{D} (0.42)	26.77^{B} (0.71)	3.46^{C} (0.12)	3.53^{A} (0.05)	4.34^{A} (0.07)
G.levis (B.Beting	24.51^{C} (0.45)	84.84^{A} (0.36)	52.19^{B} (0.56)	32.65^{A} (0.31)	5.73^{A} (0.13)	2.00^{D} (0)	3.70^{C} (0.02)
G.scortechinii (B.Semantan)	30.36^{A} (0.02)	75.25^{C} (0.33)	47.74^{C} (0.05)	27.55^{B} (0.37)	4.77^{B} (0.12)	2.23^{C} (0.01)	3.70^{C} (0.01)
B.vulgaris (B.Minyak)	24.41^{C} (0.27)	82.04^{B} (0.11)	57.30^{A} (0.49)	24.74^{C} (0.39)	5.69^{A} (0.07)	2.72^{B} (0.08)	4.07^{B} (0.07)

Values in () are standard deviation

Means followed by the same letters $^{A, B, C, D}$ in the same column are not significantly different at $p \leq 0.05$ according to LSD

diphenylene diisocyanate (pMDI). The composition of these woods was 61–75% holocellulose and 20–24% lignin. Due to the high amount of holocellulose present in bamboo, the liquefaction reaction may be facilitated, leading to an increased quantity of liquefied material and consequently a greater production of polyol. However, the higher lignin content of bamboo may have some disadvantages because that component tends to recondense and form crosslinked substances if the reaction time exceeds a certain threshold.

Table 2 Percentage of yield, hydroxyl number, and viscosity on liquefied wood towards different reaction temperature

Temperature		Species				
		Betong	Beting	Minyak	Semantan	Mixed
Yield (%)	120 °C	61.44 (18.94)	40.79 (9.06)	63.85 (6.95)	80.62 (1.52)	35.04 (1.99)
	130 °C	68.11 (20.70)	44.67 (9.65)	70.47 (8.15)	85.74 (2.50)	38.64 (2.48)
	140 °C	71.86 (19.76)	49.33 (10.79)	74.64 (4.87)	90.19 (2.25)	43.68 (2.55)
	150 °C	96.18 (3.26)	94.87 (3.73)	97.97 (0.22)	96.37 (0.88)	95.15 (0.59)
	160 °C	84.75 (4.10)	82.72 (4.52)	85.98 (2.16)	81.38 (5.69)	84.17 (0.66)
Hydroxyl number (mg KOH/g)	120 °C	338.94 (20.24)	321.40 (10.12)	313.22 (4.05)	350.62 (17.53)	326.91 (27.34)
	130 °C	328.35 (11.20)	301.22 (13.18)	298.55 (11.77)	312.71 (16.28)	301.22 (6.37)
	140 °C	310.81 (5.97)	285.93 (21.88)	194.08 (12.25)	267.08 (26.09)	275.09 (11.67)
	150 °C	297.31 (3.56)	286.33 (30.61)	298.25 (9.03)	275.83 (21.43)	276.34 (22.11)
	160 °C	254.85 (9.00)	237.99 (29.93)	260.87 (18.27)	239.55 (12.26)	235.82 (20.67)
Viscosity (cP)	120 °C	193.02 (13.96)	186.78 (8.23)	176.75 (7.87)	182.34 (9.79)	208.55 (27.91)
	130 °C	195.55 (22.07)	201.56 (11.31)	185.03 (5.51)	182.30 (15.26)	215.66 (22.70)
	140 °C	206.88 (14.38)	210.74 (13.27)	194.08 (12.25)	182.50 (17.19)	219.27 (22.30)
	150 °C	268.47 (8.75)	305.00 (9.15)	248.04 (37.09)	257.13 (22.59)	258.46 (8.12)
	160 °C	238.31 (5.69)	271.54 (8.71)	222.30 (11.59)	233.05 (39.75)	207.39 (9.23)

Values in () are standard deviation

Effect of species and temperature on yield, hydroxyl number, and viscosity

Table 2 shows the yield, hydroxyl number, and viscosity percentages of liquefied wood at various reaction temperatures. It shows that increasing the reaction temperature resulted in an increase in the percentage of liquefied bamboo yield. When compared to other reaction temperatures (120, 130, and 140 °C), nearly all of the bamboo powder was liquefied at 150 and 160 °C, as shown by the high yield. Nonetheless, the percent yield decreased when the temperature was raised to 160 °C. This could be due to the high temperature, which promotes lignocellulosic decomposition and water re-condensation back into liquefaction solution intermediates [12]. Therefore, to maintain maximum yield, the reaction temperature should not exceed 150 °C. Similarly, the viscosity increased with an increase in reaction temperature except for hydroxyl number. The conversion of bamboo can be enormously increased with increasing temperature but not on hydroxyl number. That is, when bamboo is heated, the glycosidic linkage is attacked, resulting in dehydration, decarbonylation, and cleavage of the molecules into smaller soluble fragments, and the bamboo liquefies when the temperature reaches 150 °C [24].

The significant effect on percent yield is shown in Fig. 1, as a result of the interaction between species and temperature. The yield exhibits an upward trend, with the highest recorded at 150 °C, followed by 160 °C. At both temperatures, the bamboo species did not have a significant effect on the percent yield, as indicated by the "A" ranking. The yield of liquefied bamboo was greatly influenced by the bamboo species at lower temperatures (≤ 140 ℃). Beting exhibits lower yield percentages at 120, 130, and 140 °C, which appears to indirectly impact the yield percentages of mixed species. Surprisingly, it is able to reach a high yield percentage at 150 °C, ranking similar to other species. It was indicated that, at temperatures of 150 and 160 °C, all species were able to achieve a higher percentage of yield. On the other hand, Semantan consistently proved to be superior compared to other species, even when exposed to temperatures as high as 120 ℃.

Figure 2 depicts the impact of reaction temperature on the hydroxyl number of bamboo. Among the species, only Betong was affected by the reaction temperature and it yielded the highest hydroxyl number. The hydroxyl numbers of other species ranged by only 2 to 4%, which is not considered significant.

Figure 3 depicts the effect of reaction temperature on polyol hydroxyl number. The hydroxyl number decreased as the reaction temperature increased from 120 to 160 °C, reaching 330.22, 308.41, 285.53, 286.83, and 244.83 mg KOH/g, respectively. The lower temperature appeared to yield more hydroxyls; however, it did not have a high percentage of yield.

The viscosity is significantly affected by the interaction of species and temperature, as shown in Fig. 4. The viscosity exhibits an increasing trend, with the highest recorded at 150 °C, followed by 160 °C for all species

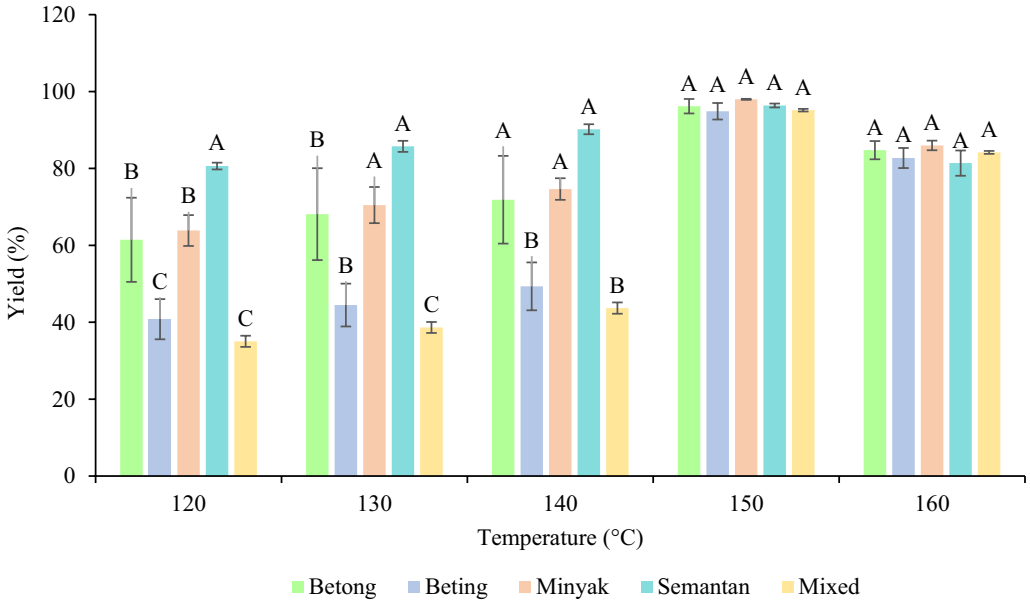


Fig. 1 Effects of bamboo species and temperature on the percent yield of polyol**.** Bar charts followed by the same letters [A,B,C] were not significantly different at $p \leq 0.05$ according to LSD

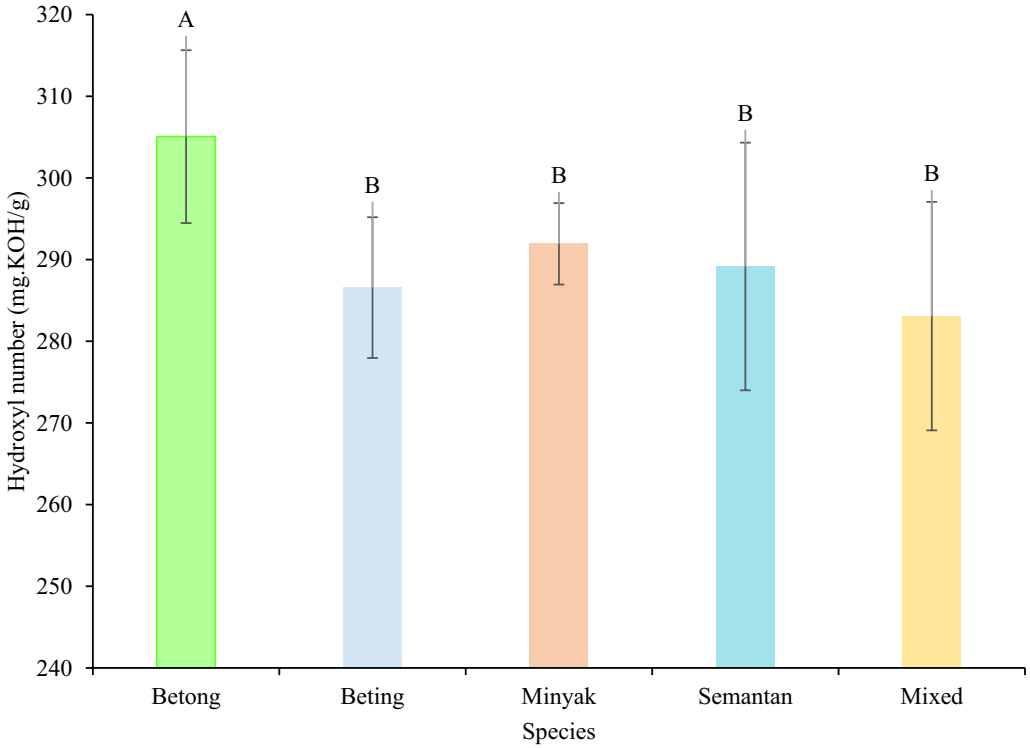


Fig. 2 Hydroxyl number of different species at different reaction temperature. Bar charts followed by the same letters [A,B] were not significantly different at $p \leq 0.05$ according to LSD

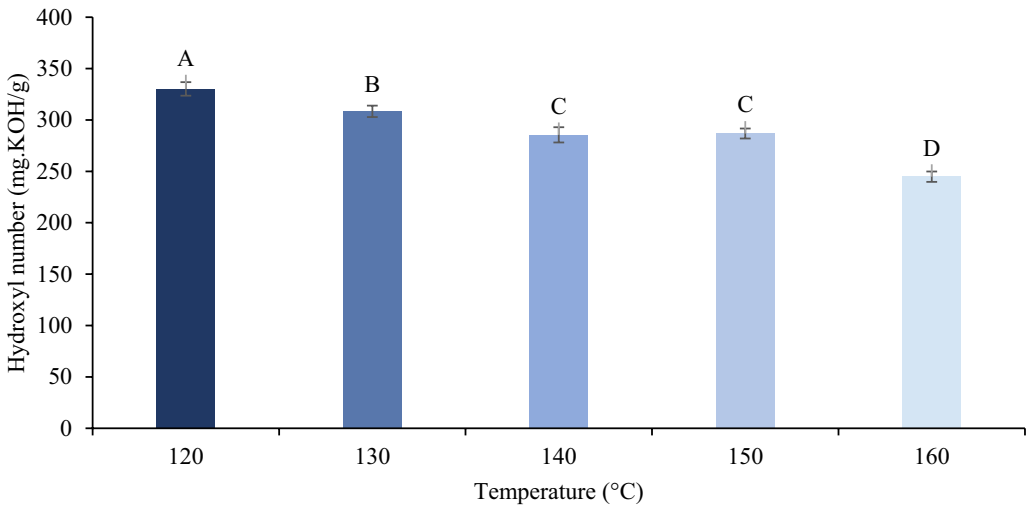


Fig. 3 Hydroxyl number of polyol at different reaction temperature. Bar charts followed by the same letters [A,B,C,D] were not significantly at $p \leq 0.05$ according to LSD

except mixed species. Meanwhile, a temperature range of 120–140 °C exhibited the lowest viscosity values among all species. The indication was that a temperature of 150 °C was appropriate for all bamboo species to achieve the highest number of viscosities for polyol.

Effect of species and reaction time on the yield, hydroxyl number, and viscosity

Along with the extension of reaction time, the yield percentage and viscosity have increased at a reaction time of 90 min and then gradually decreased at

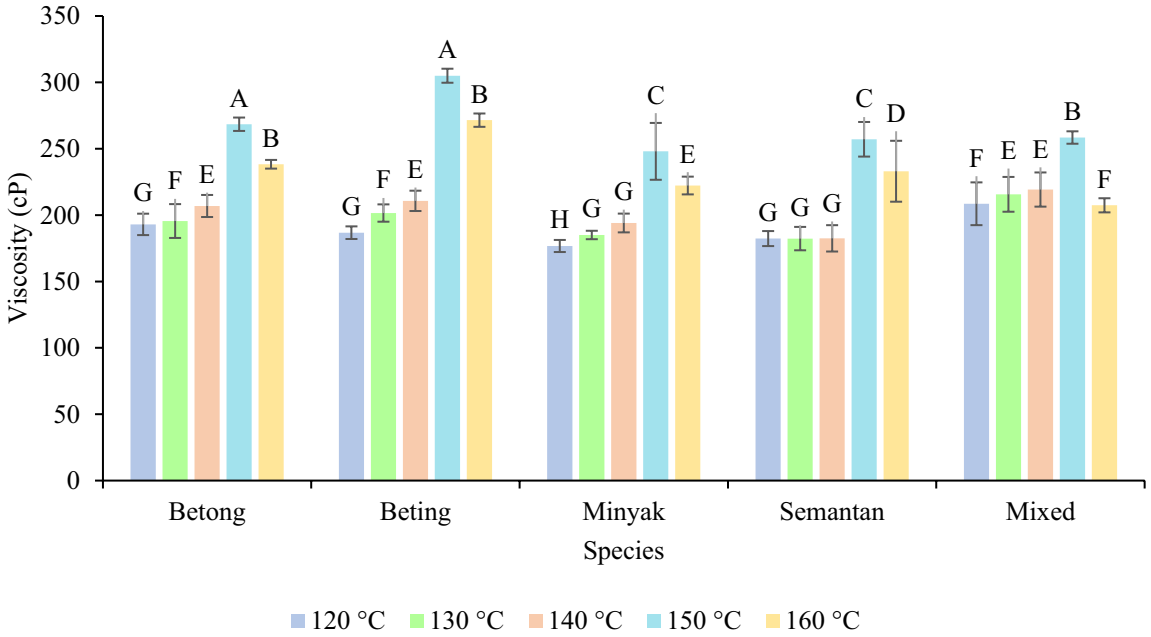


Fig. 4 Viscosity of polyol from interaction between species and temperature. Bar charts followed by the same letters ^{A,B,C,D,E,F,G,H} were not significantly at $p \leq 0.05$ according to LSD

Table 3 Percentage of yield, hydroxyl number, and viscosity on liquefied bamboo towards different reaction time

Property	Time (min)	Species				
		Betong	Beting	Minyak	Semantan	Mixed
Yield (%)	60	94.91 (0.68)	93.51 (0.59)	95.04 (0.37)	94.19 (0.77)	94.59 (1.34)
	90	96.18 (3.26)	94.87 (3.73)	97.97 (0.22)	96.73 (0.88)	95.15 (0.59)
	120	93.91 (1.65)	9.90 (1.47)	91.83 (1.21)	85.60 (9.61)	93.92 (0.73)
Hydroxyl number (mg KOH/g)	60	309.15 (0)	315.56 (0)	322.23 (11.55)	315.22 (18.05)	342.83 (10.24)
	90	297.31 (3.56)	286.33 (30.61)	298.25 (9.03)	275.83 (21.43)	276.34 (22.11)
	120	257.66 (17.05)	272.11 (9.94)	226.43 (11.78)	276.27 (11.15)	222.27 (8.39)
Viscosity (cP)	60	278.99 (16.44)	240.05 (12.43)	219.43 (16.35)	296.27 (14.33)	231.60 (7.47)
	90	268.47 (8.75)	281.50 (6.86)	248.04 (37.09)	257.13 (22.59)	258.46 (8.12)
	120	257.66 (17.05)	232.72 (10.23)	226.43 (11.78)	276.27 (11.15)	222.27 (8.39)

Values in () are standard deviation

Liquefaction was carried out at 150 ℃

120 min, except for the hydroxyl number presented in Table 3. This result was similar to the analysis done by Juhaida et al. [12] using kenaf core. The bamboo conversion requires a specific reaction time. This increase may be attributed to the recondensation of the liquefied component, which is thought to be caused primarily by lignin decomposition during the additional time [25]. Kurimoto et al. [20] state that wood liquefaction occurs in three stages. During the first 30 min of reaction time, approximately 60–70% of the wood was liquefied,

resulting in much higher residue that could be composed of degraded cellulose and hemicellulose. The second stage was distinguished by a gradual decrease in reaction residue, which is primarily dependent on the difficult-to-access cellulose. The third stage of the reaction shows an increase in the number of residues, indicating the formation of other by-products. The same author discovered that most of the reactions recorded and increased reaction residue after 90 min of liquefaction and their time-course study on the formation of residue during liquefaction of softwoods and hardwoods using the PEG system. In particular, at 120 min, suggesting some recondensation or reprecipitation of liquefied wood components had occurred. Such increases are undesirable, because they do not produce the most significant number of polyols.

The percentage of polyol yield is significantly affected by time, as shown in Fig. 5. The highest yield percentage

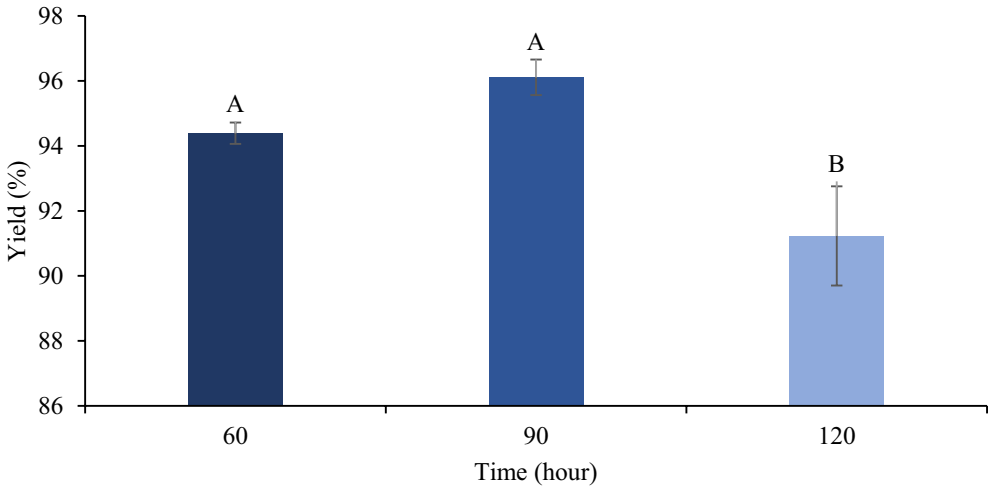


Fig. 5 Yield of polyol at different reaction time. Bar charts followed by the same letters [A,B] were not significantly at $p \leq 0.05$ according to LSD

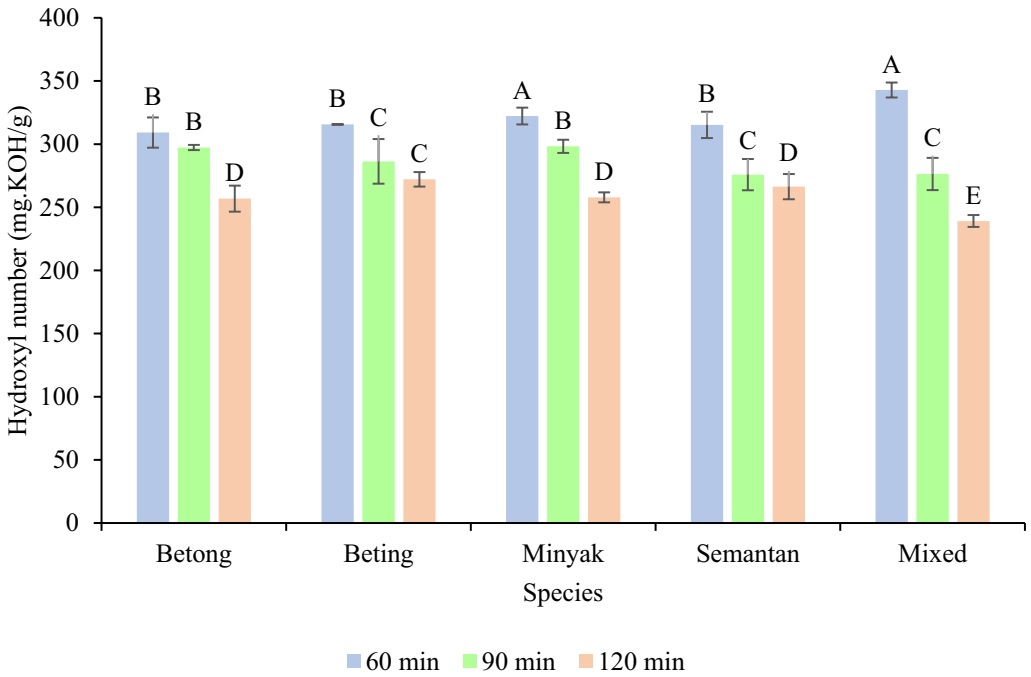


Fig. 6 Hydroxyl number of polyol from interaction between species and reaction time. Bar charts followed by the same letters [A,B,C,D,E] were not significantly at $p \leq 0.05$ according to LSD

was achieved in 90 min at 96.11%, followed by 60 and 120 min at 94.39% and 91.23%, respectively.

Figure 6 displays the interaction effect of hydroxyl number on the species and reaction time. The hydroxyl number decreased as the reaction time increased, from 60 to 120 min, for all species. A lower reaction time of 60 min resulted in a higher hydroxyl number, particularly for the Minyak and Mixed bamboo species.

As illustrated in Fig. 7, viscosity has an interaction effect on the species and reaction time. The yield percentage gradually increased at 90 min for species Beting, Minyak, and Mixed bamboo. The yield percentage of Beting, Minyak, and Mixed bamboo was increased at 90 min of reaction time and swiftly at 120 min of reaction time. Meanwhile, Betong and Semantan were slightly influenced by reaction time with approximately 10% differences (as indicated by LSD ranking A and B).

The bamboo polyol was analysed for percent yield, hydroxyl number, and viscosity. These results demonstrated the influence of the liquefaction temperature and time on the liquefaction behaviours of the all-bamboo species for yield, hydroxyl number, and viscosity. Depending on the specific liquefaction parameters and biomass type, biomass liquefaction-derived polyols showed hydroxyl numbers ranging from approximately 100 to 600 mg KOH/g (hydroxyl number is a key factor to determine the reactivity of polyol with isocyanates in the polyurethane synthesis process, besides giving vital information of final polyurethane product: hardness, flexibility, and other physical characteristics), viscosities from 300 to 4500 cP (higher viscosities were more rigid than those from lower viscosity ones, probably because of their structural differences), and last but not least, yield from 60% to 95% (critical parameters that reflect the efficiency of the conversion process) [26]. In general, polyols derived from biomass liquefaction are suitable for the production of rigid or semi-rigid PU foams. However, it has also been reported that they are used in the preparation of PU adhesives and films. The best operating conditions were observed at a temperature of 150 °C and a duration of 60 min for the mixture of bamboo. These conditions resulted in a yield of 94.59%, a hydroxyl number of 342.83 mg KOH/g, and a viscosity of 231.60 cP. This combination appears to provide values that are closest to the mentioned properties.

Fourier transform infrared (FTIR) spectroscopy measurements for liquefied polyol

The functionality groups that existed in the samples were identified using FTIR. The spectra were taken from 500 to 4000 cm^{-1}. The FTIR spectra were divided into two information-rich regions: the fingerprint region, which ranged from 1800 to 500 cm^{-1}, and the stretching vibration mode region, which ranged from 3800 to 2800 cm^{-1} [27]. Figure 8 displays the FTIR spectra of bamboo polyol for reaction temperature and time, specifically at 150 °C

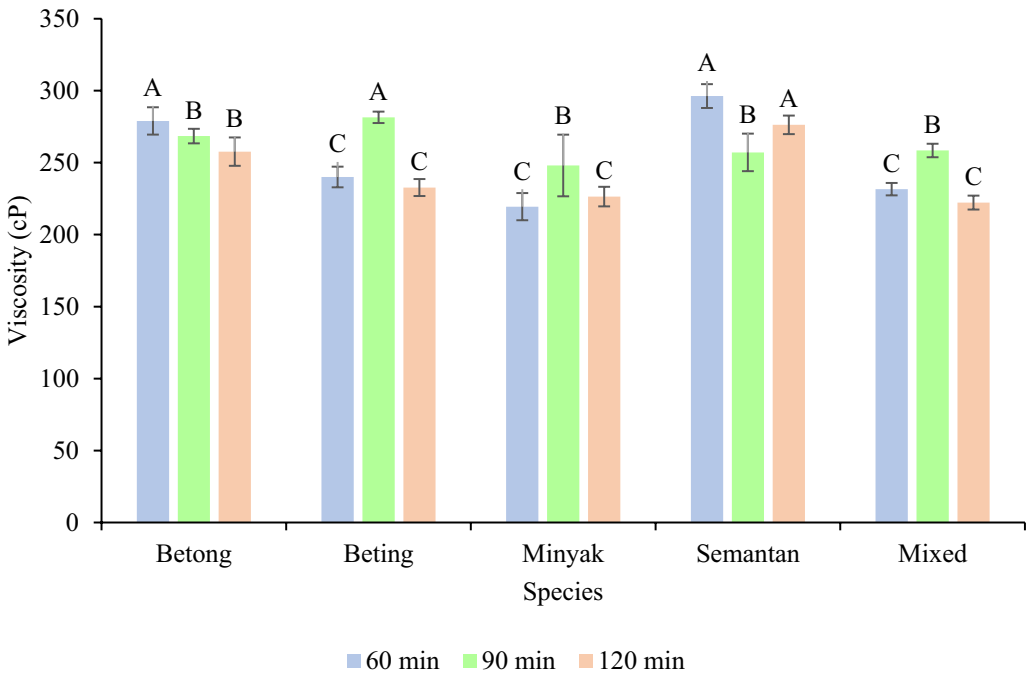


Fig. 7 Viscosity of polyol from interaction between species and reaction time. Bar charts followed by the same letters ^{A,B,C} were not significantly different at $p \leq 0.05$ according to LSD

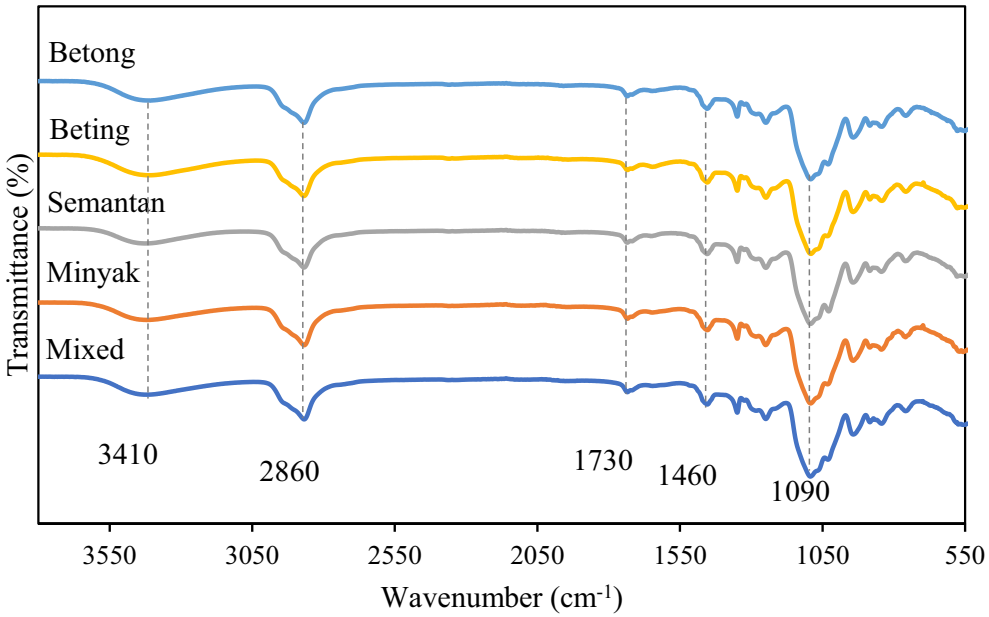


Fig. 8 FTIR of polyol at temperature 150 °C and 60 min

and 60 min, respectively. The broad hydroxyl peak at 3410 cm^{-1} indicates significant hydrogen bonding. The broad and intense nature of this peak suggest the presence of hydrogen bonding. The intensity appears relatively consistent across all samples, suggesting a similar hydroxyl content among bamboo. The strong peak at 2860 cm^{-1} corresponded to an indistinguishable C–H stretching vibrations of ($–CH_2$) and methyl ($–CH_3$) groups. This peak indicated the presence of long hydrocarbon chains, which is likely derived from fatty acids or triglycerides in the samples. As bamboo is primarily made up of cellulose, hemicellulose and lignin, Yip et al. [26] concluded from its FTIR spectra that the peak at 1734 cm^{-1} attributed to xylans in hemicellulose (C=O). The lignin is represented by absorption at 1349 cm^{-1} (benzene ring), and the glycosidic bond vibration cellulose is represented by adsorption at 1092 cm^{-1} with additional contributions from PEG and glycerol, and the lower frequency range below 1500 cm^{-1} was dominated by C–H bonding and CH_2 wagging [10]. All bamboo shows nearly identical spectra, suggesting highly similar chemical compositions. Mixed bamboo samples exhibit a balanced combinations of spectral features from all individual samples, with consistent peak intensities reflecting uniform distribution.

Gel permeation chromatography (GPC) of liquid products

GPC is used to determine the molecular weight distribution of polyol products. The calibration curve derived from standard samples was used in this work to estimate the molecular weight through the retention time. This calibration curve shows a relationship between retention duration and molecular weight. According to GPC results, all polyols have similar molecular weight distribution curves that can be divided into two prominent peaks: one has a shorter retention time, corresponding to a higher molecular weight, and the other is sharp and narrow, corresponding to a lower molecular weight. This could be due to the unreacted solvent and smaller molecules produced by the in-depth decomposition of bamboo lignocellulosic [28].

In work done by D'Souza and Yan [10], the polydispersity correction factor is needed to account for the nature of the biopolymer, whose value is determined iteratively based on values for Mw, Mn, and Mz from GPC analysis. According to Table 4, the PDI index of liquefied polyol exhibits the closest value and the highest molecular weight among Betong, Semantan, Minyak, and Mixed, with the exception of Beting. The result is related to the study conducted by Palle et al. [29], which demonstrated that higher Mw data is accompanied by polydispersity indices. The moderate molecular weight obtained from this study may be able to replace polyol in the manufacturing of bio-based PU, as has been achieved by others [28, 30].

Colour measurement

There is a significant difference in colour (ΔE^*) ranging from 20.96 to 43.08, which indicates that the dark brown colour of liquefied bamboo has been successfully

Table 4 GPC analysis of liquefied polyol

Species	Mn	Mw	MP	Mz	Mz^{+1}	Mz/Mw	PDI
Betong	3466	3489	3295	3513	3538	1.006894	1.006636
Beting	3153	3154	3280	3155	3156	1.000333	1.000317
Semantan	3485	3510	3343	3537	3566	1.007674	1.007174
Minyak	3450	3474	3188	3500	3527	1.007411	1.006957
Mix	3466	3489	3295	3513	3539	1.006979	1.006636

Mn Number-average molecular weight, *Mw* Weight-average molecular weight, *MP* Peak molecular weight, *Mz* Z-Average molecular weight, Mz^{+1} Z + 1 average molecular weight, *Mz/Mw* Different measures of molecular weight in distribution on a polymer, *PDI* Polydispersity index

Table 5 Change in colour in liquefied bamboo polyol after hydrogen peroxide treatment compared to untreated (unbleached) sample

Sample	Bleaching parameters		Bleaching colour			
	Time	H_2O_2%	*L**	*a**	*b**	Δ*E**
ULB (CONTROL)	–	–	38.7 (0.10)	+ 1.23 (0.15)	+ 1.13 (0.15)	0
BLB1	12	20	50.33^{G} (0.32)	$+7.03^{B}$ (0.21)	+ 14.9 (0.17)	28.61
BLB2	12	40	60.07^{E} (0.15)	$+8.37^{A}$ (0.06)	+ 26.6 (0.20)	34.01
BLB3	12	60	68.33^{C} (0.40)	$+1.57^{D}$ (0.29)	+ 27.8 (0.26)	39.87
BLB4	12	80	70.40^{B} (0.20)	-0.7^{E} (0.10)	+ 22.83 (0.15)	38.46
BLB5	24	20	51.23^{F} (0.15)	$+7.53^{B}$ (0.06)	+ 16.7 (0.36)	20.96
BLB6	24	40	66.07^{D} (0.49)	$+9.2^{A}$ (0.10)	+ 28.2 (0.30)	39.31
BLB7	24	60	71.7^{A} (0.96)	$+2.4^{C}$ (0.20)	+ 28.8 (0.30)	43.08
BLB8	24	80	72.5^{A} (0.66)	-1.2^{F} (0.10)	+ 25.23 (0.84)	41.58

Δ*E** colour difference between the unbleached liquefied bamboo (ULB) and the bleached liquefied bamboo (BLB)

Values in () are standard deviation

Means followed with the same letter ^{A,B,C,D,E,F,G} in the same column are not significantly different at $p \leq 0.05$ according to LSD

transformed into a light-yellow colour, as shown in Table 5 and Fig. 9. The colour difference measured by Cheumani Yona et al. [15] using black poplar, which is 48.9, closely resembles it. The colour shift is readily discernible during the 12 h reaction time of the treatment. The concentration of hydrogen peroxide that exhibited the greatest colour difference was 60%, followed by 80%, 40%, and 20%, with corresponding values of 39.87, 38.64, 34.01, and 28.81, respectively. The trend that was observed after 24 h of reaction was comparable to the trend that was observed after 12 h. Once again, the highest percentage was 60%, which was followed by 80%, 40%, and 20%, with respective values of 43.08, 41.58, 39.31, and 20.93.

This argument was similar to previous study from Cheumani Yona et al., [13] using liquefied of cork with ascending order of 4 > 20 > 40 > 60 > 80% of hydrogen peroxide. The other major changes were observed at axis *a** and *b**, liquefied bamboo tended to become orange (mixture of red and yellow) after oxidation reaction, especially at 40% and 60% of hydrogen peroxide for both reaction time. The decomposition of hydrogen peroxide in the presence of carbonates leads to the formation of peroxy-carbonates (CO_4^{2-}) as intermediate oxidant species. In addition, these oxidants have been reported to have a capability to initiate the oxidation of various organic substrates. It may be possible to adjust treatment conditions to enhance the formation of (CO_4^{2-}) as a means of accelerating rates of contaminant removal [13, 31, 32].

Figure 10 shows the impact of varying the concentration of hydrogen peroxide and the duration of the reaction on the *b** measurement. Notably, the highest *b** value was achieved at a hydrogen peroxide concentration of 60% after a reaction period of 24 h. The result suggests a noticeable shift in colour from green towards red.

Effect of hydrogen peroxide treatment on hydroxyl number, viscosity and mass loss

An important requirement for products intended to be used as polyol components in the synthesis of PU is their hydroxyl number. Table 6 reports the hydroxyl number of unbleached liquefied bamboo (ULB) and bleached liquefied bamboo (BLB). As the amount of hydrogen peroxide use increase, there was a noticeable decrease in the hydroxyl value of the bleaching

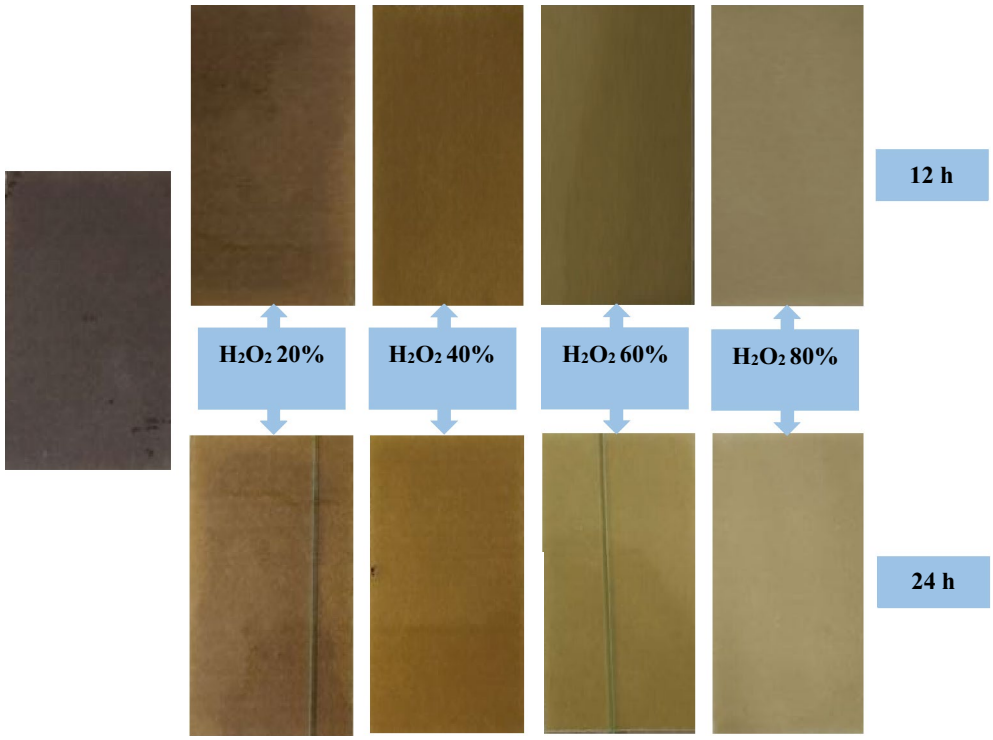


Fig. 9 Colour difference as affected by different hydrogen peroxide percentage at 12 h and 24 h reaction time

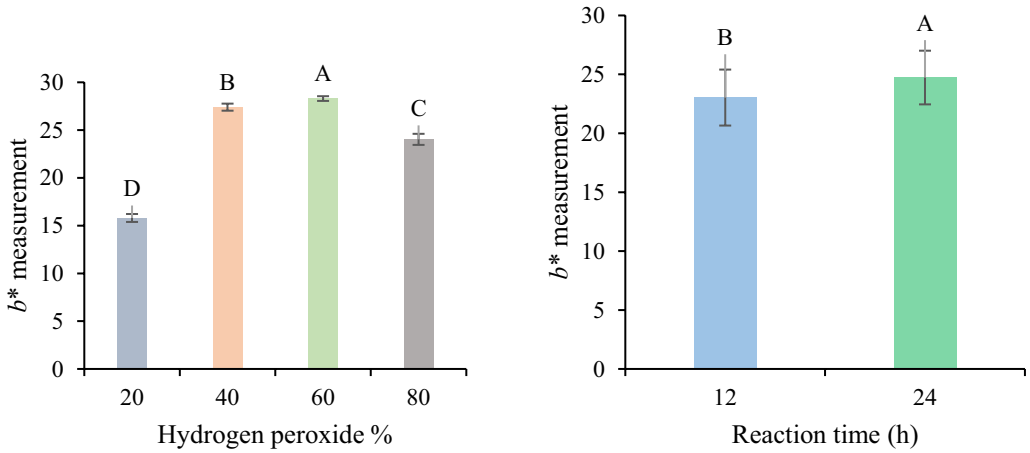


Fig. 10 *b** measurement as affected by the hydrogen peroxide percentage and reaction time. Bar charts followed by the same letters [A,B,C,D] were not significantly different at $p \leq 0.05$ according to LSD

polyol at both 12- and 24-h reaction times. After 12 h, the hydroxyl value (mg KOH/g) begins to fall to 271.20 (20%), 216.73 (40%), 214.09 (60%), and 208.82 (80%) from 342.83 (ULB), with different values of 71.63, 126.10, 128.74, and 134.01, respectively. The declining tendency was more pronounced during the 24 h reaction time compared to the 12 h reaction time. The likely cause of this is the prolonged stirring, which resulted in the decline of the hydroxyl number due to the occurrence of more oxidation reactions. The number of hydroxyls (mg KOH/g) decreased to 217.59 (20%), 212.86 (40%), 211.94 (60%) and 206.29 (80%) from 342.83 (ULB), with corresponding differences of 125.24, 129.97, 130.89, and 136.54. This result differed with Cheumani-Yona et al. [15]; Cheumani-Yona et al. [13] who reported that the number of hydroxyls of black poplar wood polyol was

Table 6 Change of hydroxyl number, viscosity and mass loss of liquefied wood after hydrogen peroxide treatment

Type of liquefied bamboo polyol	Bleaching parameters		Hydroxyl number (mg KOH/g)	Viscosity (cP)	Mass loss (%)
	Time	H_2O_2%			
ULB (CONTROL)	–	–	342.83^{A} (0.63)	231.60^{A} (1.72)	–
BLB1	12	20	271.20^{A} (3.04)	208.05^{B} (1.25)	0.67^{G} (0.04)
BLB2	12	40	216.73^{B} (9.23)	203.58^{C} (1.09)	2.00^{E} (0.11)
BLB3	12	60	214.09^{B} (3.54)	200.72^{D} (1.97)	2.00^{E} (0.09)
BLB4	12	80	208.82^{B} (2.38)	194.70^{E} (1.11)	4.00^{C} (0.12)
BLB5	24	20	217.59^{B} (4.44)	207.82^{B} (0.76)	1.89^{F} (0.29)
BLB6	24	40	212.86^{B} (8.26)	202.49^{C} (0.45)	3.33^{D} (0.15)
BLB7	24	60	211.94^{B} (3.80)	197.25^{D} (1.21)	4.62^{B} (0.14)
BLB8	24	80	206.29^{C} (3.99)	196.05^{E} (0.22)	6.27^{A} (0.29)

Values in () are standard deviation

Unbleached liquefied bamboo (ULB); bleached liquefied bamboo (BLB)

Means followed with the same letter ^{A,B,C,D,E,F,G} in the same column is not significant different at $p \leq 0.05$

somewhat greater after bleaching. They suggest that the slight increase may be attributed to (1) the formation of carboxylic acid groups, (2) the incomplete oxidation of a significant portion of the hydroxyl number in the liquefied wood, and/or (3) the formation of hydroxyl number from the epoxidation of carbon–carbon double bonds and hydrolysis of epoxy groups. Meanwhile, the values recorded for acid number and viscosity did not show significant changes. As previously mentioned, the acid number ranged from 12.5 to 24.3 mg KOH/g, and the viscosity measured 231.60 cP. Following the bleaching procedure, the acid number experienced an increase to a range of 14.7–27.4 mg KOH/g, while the viscosity witnessed a decrease to 204.50 cP after a 12-h reaction time and 186.55 cP after a 24-h reaction time.

The mass loss of BLB samples during the bleaching procedure showed a range of 0.67% to 4.00% for a 12 h reaction time and 1.89% to 6.27% for a 24 h reaction period. A lower percentage of mass loss corresponds to a less pronounced degradation of the product, while a higher mass loss indicates a more substantial degradation. According to a separate study conducted by Cheumani-Yona et al. [13], the mass loss following the bleaching procedure was limited to around 1.2%. That finding is comparable to this study of bleaching parameters at 12 h: 20% (BLB1), 40% (BLB2) and 60% (BLB3); and 24 h: 20% (BLB5), respectively. Consequently, it is assumed that the discoloration

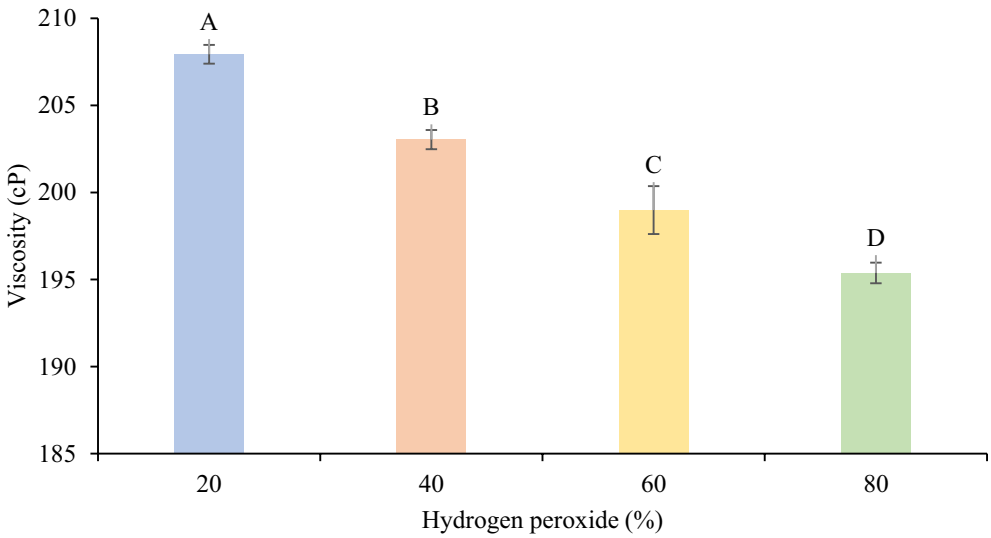


Fig. 11 Viscosity of polyol at different hydrogen peroxide percentages. Bar charts followed by the same letters ^{A,B,C,D} were not significantly at $p \leq 0.05$ according to LSD

of the BLB was accomplished with minimal product degradation.

Figure 11 illustrates the impact of varying hydrogen peroxide concentrations on the viscosity of bleached liquefied bamboo (BLB). As the hydrogen peroxide percentage increased from 20 to 80%, the viscosity values were measured at 207.94 cP, 203.04 cP, 198.99 cP, and 195.38 cP, respectively. This data indicates that higher levels of hydrogen peroxide result in a more pronounced reduction in the viscosity of BLB, suggesting a correlation between hydrogen peroxide concentration and the liquefied bamboo bleached viscosity. This information is valuable for understanding how chemical treatments affect the properties of bamboo-derived products, potentially aiding in industrial processes or product development.

Figure 12 depicts the IR spectrum of unbleached liquid bamboo (ULB) and bleached liquefied bamboo (BLB) after a reaction time of 24 h. The analysis revealed only minor differences in the spectra of ULB and BLB, suggesting that the chemical structure of ULB was not significantly impacted. The changes are mostly observed in terms of the relative intensity of the band at 1720 cm^{-1}, which corresponds to carbonyl (C=O) groups. According to Cheumani-Yona et al. [15], the change happened could be due to the oxidation of some of the functional groups (alcohols, phenols, or carbon–carbon double bonds) in the liquefied wood to ketones, aldehydes, or carboxylic acids). Moreover, as reported in the literature by Araujo et al. [33], hydrogen peroxide can cause various chemical reactions, depending on its concentration and the nature of the activators. Such reactions include the epoxidation of carbon–carbon double bonds (and the subsequent hydrolysis of the epoxy groups, forming hydroxyl groups); the oxidation of carbon–carbon double bonds into two carboxylic acid groups; the oxidation of hydroxyl or phenolic groups to ketones, aldehydes, and carboxylic acid groups; or the severe degradation of organic compounds, forming carbon dioxide and water. Clearly, bleaching caused certain modifications to the liquefied wood combination, and these modifications could potentially affect the qualities of polyurethane films in the future. The other vibrational bands were not affected considerably after treatment process. The spectra of original liquefied bamboo and other treatment liquefied polyol shows that s strong broad OH stretching at 3410 cm^{-1} and C–H stretching in methyl and methylene group at 2860 cm^{-1}. As mentioned by Daneshvar et al. [34], the peak at 3410 cm^{-1} (hydroxyl group) is a significant site that appeared in all samples and indicated a treatment process was applied. It can be utilised in numerous reactions, including chemical modification and the production of numerous polymers, such as polyurethane.

Conclusion

The chemical composition of all bamboo species varied significantly ($p < 0.0001$). However, these differences did not have a noticeable impact on the extent of liquefaction. Increasing the reaction temperature enhance the yield percentage of liquefied bamboo. Meanwhile, viscosity generally increase with rising reaction

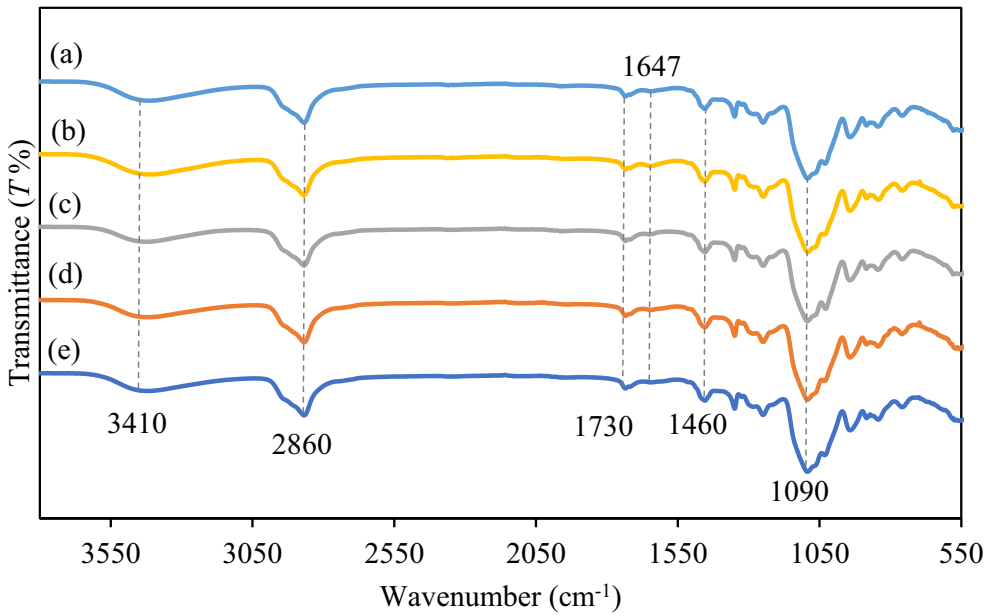


Fig. 12 FTIR spectra of (**a**) unbleached liquefied bamboo (ULB), (**b**) liquefied bamboo bleached (BLB) at 20%, (**c**) liquefied bamboo bleached (BLB) at 40%, (**d**) liquefied bamboo bleached (BLB) at 60%, (**e**) liquefied bamboo bleached (BLB) at 80% at reaction time 24 h

temperature, except for hydroxyl number. These findings are essential for optimizing the liquefaction process of bamboo, considering factors, such as yield, viscosity, and yield. To maintain these factors, it is recommended that the temperature reaction should not exceed 150 °C. Extension of reaction time from 60 to 90 min shows an increase in yield percentage and viscosity; however, these values gradually decrease at 120 min. Notably, the hydroxyl number remains stables. These findings found that the liquefied bamboo need to optimize at 60 min reaction times for desired outcomes. By utilising an ideal combination of reaction conditions, which includes a temperature of 150 °C and a duration of 60 min, impressive results were achieved with the bamboo polyol. These results include a yield of 94.59%, a hydroxyl number of 342.83 mg KOH/g, and a viscosity of 231.60 cP. The study also suggests that a mixture of bamboo wastes can be used for the liquefication process to obtain a comparable result with bamboo waste of single species. A yellowish coloured polyol can be produced from dark-brown liquefied bamboo through a bleaching process involving the use of hydrogen peroxide, with potassium carbonate serving as the activator. The effectiveness of the treatment in altering the colour of liquefied bamboo polyol can be observed when different amounts of hydrogen peroxide and reaction time are used. The colour of the liquefied bamboo polyol was successfully changed from dark-brown to a more yellowish tone by specifically adding 60% hydrogen peroxide and stirring for a period of 12 h. The process of effectively carrying out discoloration in liquefied bamboo bleached while maintaining mass loss. However, it is important to note that the utilisation of a higher percentage of a certain treatment and allowing it to react for a longer period resulted in a more significant deterioration of the product, in contrast to using a lower percentage and shorter reaction time. This suggests that there is a need for careful optimisation to balance the removal of discoloration and the preservation of the product. The analysis of the spectra of untreated liquefied bamboo and liquefied bleached bamboo following bleaching demonstrated that there were only slight distinctions between them. This indicates that the chemical composition and structure of the untreated liquefied bamboo did not undergo significant changes as a result of the bleaching process.

Abbreviations

PU	Polyurethane
ONP	Old newspaper
OMG	Old magazine
TCF	Totally chlorine free
LY	Liquefaction yield
MC	Moisture content
FTIR	Fourier transform infrared
GPC	Gel permeation chromatography
THF	Tetrahydrofuran
ULB	Unbleached liquefied bamboo
BLB	Bleached liquefied bamboo

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Redzuan Mohammad Suffian James: methodology, investigation, and writing—original draft. Norwahyuni Mohd Yusof: formal analysis and data curation. Syeed SaifulAzry Osman Al-Edrus: investigation and data curation. Zurina Zainal Abidin: methodology and data curation. Paridah Md Tahir: conceptualization, formal analysis, and writing—review and editing. Mohd Zuhri Mohammed Yusoff: writing—review and editing. H'ng Paik San: writing—review and editing and funding acquisition. Seng Hua Lee: conceptualization, supervision, project administration, and writing—review and editing.

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Availability of data and materials
The data sets used and/or analysed during the current study are available from the corresponding author on reasonable request.

Declarations

Ethics approval and consent to participate
Not applicable.

Consent for publication
Not applicable.

Competing interests
The author (s) declare (s) that they have no competing interests.

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