Evaluation of synthetic bio-phenol resins derived from biomass gasification in bio-adhesive formulation

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Abstract. Bio-phenol formaldehyde adhesives formulated from sustainable biomass sources is an excellent alternative to phenolic petroleum-based adhesives with lower pollution level. This study aimed to formulate bio-adhesive from two types of biomass namely palm kernel shell (PKS) and Leucaena sp. Wood. The intention was to find the best process condition which results in maximum content of bio-phenol in the product. The bio-based phenolic resins (bio-oils) were produced from gasification process and their physical and chemical properties were determined. Both produced bio-oils were involved in formulation of bio-based phenol formaldehyde adhesives (resinification) at different operating conditions (temperature, time and catalyst loading). The chemical functional groups and individual compounds of the phenol resins and bio-adhesive samples were identified by GC-MS. The results indicated that temperature has persistent increasing effect on phenol percent of the bio-oil from Leucaena sp. wood however for the bio-oil from PKS the increase of phenol was until the temperature of 85 ºC. Reaction time and catalyst loading were observed to have similar effects on resinification of both bio-oil samples. The bio-adhesive produced under best operating condition has the highest amount of bio-phenol and therefore is considered an environmental friendly adhesive with lower cost and pollution than the petroleum-based types.

1. Introduction
Special attention has been given nowadays to conserve the supply of petroleum by switching to bio-based products made from biomass. Adhesives used for paper, board or wood products are often made from oil-based raw materials making the final product non-biodegradable or difficult to recycle. However, price fluctuations as well as limited sources of crude oil will increase the competitiveness of renewable raw material based products. Green issues and environmental legislation also direct attention to bio-based materials. Traditionally, renewable starch-based adhesives have been used as glues, e.g. for corrugated board, but the application areas have been limited due to poor water resistance and high water content of the adhesive formulations. Phenolic resins are typically dark-reddish in color and can be synthesized at a variety of temperatures, ranging from room temperature to 135 ºC. Phenol which is the primary component of the resin used in the adhesive is derived from fossil fuel resources (petroleum
and coal) however the cost of resin fluctuates depending on the price of petrol. In addition, this type of resin consists of a material that is considered as a major indoor pollutant and thus very harmful to our health. Thus finding renewable material as another option for the raw material has become the main interest for resin production. This paper studied the potential of palm kernel shell (PKS) and Leucaena sp. wood in production of bio-adhesive (Figure 1).

![Feedstock samples](a) PKS  b) Shredded Leucaena sp. Wood.

Figure 1. Feedstock samples  a) PKS  b) Shredded Leucaena sp. Wood.

In Malaysia there is a high amount of oil palm kernel residue from the oil palm industry of which these shell disposals can cause pollution to the environment [1]. The production of palm kernel oil alone consists of 12% of the production palm oil. The residue of this palm kernel shell has been a concern to the environment because it was still seen as partial waste in 1990s, but in early 2000 more than 350,000 tons of PKS were sold [2]. Lignin, a complex macromolecule, was discovered to be one of the main components in palm kernel shell. Having major functional groups such as phenolic, methoxyl and carbonyl influence the reactivity of lignin thus the phenol which derived from lignin allows for the reduction of cost for adhesive production and also lower toxicity for adhesive industries [3]. However, Leucaena leucocephala, has been studied for biomass production for its numerous advantages such as high reported yield of undergrowth corresponds to a dried mass of 2,000–20,000 kg/ha/year, and that of wood 30–40 m³/ha/year, with up to twice those amounts in favorable climates [4]. It is also efficient in nitrogen fixation, at more than 500 kg/ha/year. Young trees reach a height of more than 20 ft in two to three years so it has a very fast growth rate. On the other hand, Leucaena sp. wood has been a focus of the environmental concern. There is an enormous potential for Leucaena sp. to be used in variety of applications such as in soil restoration, papermaking and the production of other high-value added products [5].

In this study, physiochemical properties of phenolic resins produced from gasification of both PKS and Leucaena sp. wood were analyzed by GC-MS and discussed. Moreover, the effect of different operating conditions (temperature, time and catalyst loading) on the phenol composition of final bio-adhesive product were investigated by using GC-MS analysis.

2. Materials and methods

The supply of Leucaena sp. wood was from Sarafiah Natural Resources Sdn. Bhd. (Port Klang, Selangor Darul Ehsan, Malaysia) and was dried (to 10% moisture content) and shredded (3-5cm of particle size) as received. Kernel shells were also dried to the same level of moisture content with Leucaena sp. wood particles. Other chemical used for bio-adhesive preparation includes ethanol with a concentration of 95%, formaldehyde with a concentration of 37% - 40% and sodium hydroxide solution (alkaline catalyst).

2.1 Preparation of phenol resin (bio-oil)

Feedstock were initially undergo the gasification process in an updraft reactor at 800 °C and with feeding rate of 13.88 gr/s for 60 minutes of operation. The reactor was connected to a column condenser which
was set at 15°C. Heavier compounds like phenol and methanol which has been produced earlier in pyrolysis stage will exit the reactor in vapor form along with other volatiles [6], [7]. The produced phenol resin as bio-oil were collected as the condensates of the syngas. By adding formaldehyde to the phenol resin, it can transform into bio-adhesive. The bio-oil in vapor form was condensed and separated from the syngas as it passed through the condenser hence collected for analysis (Figure 2).

![Figure 2. Phenol resin (bio-oil) as product of gasification (PKS).](image)

2.2 Characterization of synthesized phenol resin (bio-oil)
Characterization of phenol resins produced from gasification of PKS and Leucaena sp. wood were performed for both physical and chemical analysis. Physical characteristics was obtained through a set of standard laboratory procedures and the properties of the bio-oil (viscosity, pH and solid content) were determined. The chemical characterization was performed to identify the composition of compounds (phenol in particular) via GCMS analysis.

2.3 Preparation of phenol formaldehyde (bio-adhesive) by resignification
The produced bio-oil was treated in a rotary evaporator to separate the oil and water and to its viscosity. The improved bio-oil was then mixed with ethanol, sodium hydroxide as alkaline catalyst and formaldehyde. The alkaline catalyst was used to provide the composition of pH and formaldehyde. The mixture was poured into the flask and heated up at 90°C in silicon oil bath under constant stirring for 5 to 15 minutes. Then, the system was cooled down at room temperature (25°C). The ethanol was removed at 60°C by using an air circulated oven. The product was characterized by using Gas Chromatography Mass Spectrometry (GCMS). The procedure of experiment has been illustrated in Figure 3.

![Figure 3. Procedure of formulating bio-adhesive.](image)

According to [8] the amount of formaldehyde to phenol ratio (F/P ratio) considers one of the most important factors in formulization of bio-adhesive which is normally around 2.0-3.0. Higher F/P ratios enhance hydroxymethylation and increase the degree of polymerization. The ratio of NaOH/liquefied PKS is also an important factor due to the role of catalyst loading in the lignin structure.
of bio-adhesive, materials were added to the bio-oil with ratio of formaldehyde/phenol, 2.5 and NaOH/phenol of 0.3 which are adopted from a previous study by [9]. The effects of resinification time, temperature and catalysts loading have been investigated on the resinification process.

2.4 Product gas analysis
Gas chromatography (GC) was employed to determine the composition of a unit volume of the sample gas. The gas analyzer (Agilent 6890N, LabX, Ontario, Canada) was equipped with a thermal conductive detector (TCD) and a capillary column (Carboxen-1010 plot), and was used to perform the analysis. Each gas composition was determined by the help of retention time and its concentration which was calculated based on the area under the related peak.

A column of 30 m length, 530 µm diameter, and 30 µm film thickness were selected for the chromatography analysis. Argon was used as the carrier gas with the flow rate of 3 mL/min and the oven temperature was set to 35°C. The heater temperature was set to 200°C and the splitless inlet temperature was 60°C. The calibration of TCD was based on the standard gas (RSS instruments Sdn. Bhd., Malaysia) mixture containing CO, CO₂, H₂, and CH₄ in nitrogen at periodic intervals.

3. Result and Discussion
3.1 Physical characteristics of bio-oil
The appearance, odor, viscosity, pH value, solid content and free formaldehyde content of the bio-based phenolic-oils from both biomass were measured and enlisted in Table 1 in comparison with pure phenolic resin which is taken from literature [10].

Table 1. Physical characteristics of the bio-based phenolic resins from PKS and Leucaena sp. wood in comparison with pure phenolic resin.

| Type of resole          | Pure phenolic resin [10] | Bio-based phenolic resin | Leucaena sp. wood | Palm kernel shell |
|-------------------------|--------------------------|--------------------------|-------------------|------------------|
| Appearance              | Dark brown liquid        | Dark brown liquid        | Light brown liquid|                  |
| Odor                    | Pungent smell            | Pungent smell            | Pungent smell     |                  |
| Viscosity, cP (20°C)    | 100 to 200               | 92                       | 86                |                  |
| pH                      | 3.78                     | 3.76                     | 2.90              |                  |
| Solid content           | 48                       | 39.88                    | NA                |                  |
| Free formaldehyde content| 0.2%                    | 0.1%                     | NA                |                  |

According to the results, the appearance of bio-based phenolic resin from Leucaena sp. wood is dark brown liquid and has a pungent smell which is similar compared to pure phenolic resin. The bio-oil from PKS has lighter color but same odor with other two samples. The viscosity of bio-based phenolic resins are 92 and 86 cP for Leucaena sp. wood and PKS respectively which is slightly lower than the viscosity of a typical pure phenol resin (100-200 cP at 20°C [10]). The viscosity of an adhesive must ensure its penetration to a useful depth into micro- and macro-roughnesses and pores, increasing the surface area of mutual contact. At the same time, the adhesive composition should not be too low-viscosity, since in this case the majority of it penetrates into roughnesses and anatomical pores, and the remaining amount is insufficient to form a continuous adhesive film [11]. Bio-oils typically have a pH in the range of 2-4 due to the presence of organic acids among their contents. By comparison, the pH levels were observed at 3.76 and 2.90 for Leucaena sp. wood and PKS based phenolic resin respectively, while 3.78 for pure phenolic resin. Solid content is the proportion of non-volatile material contained in the phenolic oil [12]. Most typical solid content in pure phenolic is around 50% [13]. The result obtained shows only 39.88% of solid content for leucaena sp. wood bio-oil. This may be due to the unreacted phenol evaporated during the heating period, leading to a lower non-volatile content for the bio phenol resin.
3.2 Chemical characteristics of bio-oil

Formaldehyde is a toxic component that is present in phenolic resin. Its quantification is important to the characterization of the phenolic resin. The method developed for identification and quantification of free formaldehyde content in phenolic resin is based on the determination of free carbonyl compound by derivatization with 2,4-dinitrophenylhydrazine (DNPH), using gas chromatography (GCMS) [12]. They also mentioned that the standard of free formaldehyde content in phenolic resin must be less than 0.2%. According to the result from GCMS, the free formaldehyde content in the bio-based phenolic resin from Leucaena sp. wood is only 0.1% which indicates the low toxicity caused by formaldehyde in the resin.

Gas chromatography analysis was carried out for the phenolic resins from both biomass in a variant GCMS instruments analyzer under 375 °C, heating rate of 30 °C min-1 and cracking time of 30 minutes. The detected compounds and their relative area% for the major component (with relative concentration higher than 0.02%) are summarized in Table 2.

### Table 2. GCMS Analysis of bio based phenolic resin.

| Bio-based phenolic resin sources | Leucaena sp. wood | Palm kernel shell |
|---------------------------------|------------------|------------------|
| **Compound**                    | **Area (%)**     | **Compound**     | **Area (%)**     |
| Propionate, 2 methyl-1-nitro    | 1.282            | 2-cyclohexane-1-one, 3-methyl | 0.72 |
| 2-Propenamide, N-methyl        | 2.279            | 2-propenamide, N-methyl | 1.39 |
| 2-Cyclopenten-1-one, 3-methyl  | 1.757            | 2,4-Dimethyl-2-oxazoline-4-methanol | 0.53 |
| Phenol                         | 2.945            | Thiophene, 2-pentyl | 0.206 |
| 2,4-Dimethyl-2-oxazoline-4-methanol | 5.128     | Phenol            | 32.98 |
| 2-Cyclopenten-1-one, hydroxyl-3-methyl | 3.943 | 2-Cyclopenten-1-one, 2-hydroxy-3-methyl | 1.912 |
| Phenol, 4-methyl               | 2.271            | Phenol, 4-methyl | 1.708 |
| Phenol, 2-methoxy              | 4.687            | Phenol, 2-methoxy | 2.87 |
| 2-Methoxy-5-methylphenol       | 1.542            | 2-Chloroethanol | 0.468 |
| 2,3-Pyridinediamine            | 0.979            | 2-Cyclopenten-1-one, 3-ethyl-2-hydroxy | 0.731 |
| 2-Chloroethanol                | 2.035            | 2-Methoxy-5-methylphenol | 1.642 |
| 1,2-Benzenediol, 3-methoxy     | 0.934            | 1,2-Benzenediold, 3-methoxy | 1.164 |
| Phenol, 4-ethyl-2-methoxy      | 2.821            | Phenol, 4-ethyl-2-methoxy | 1.834 |
| Phenol, 2,6-dimethoxy          | 7.16             | Phenol, 2,6-dimethoxy | 7.77 |
| 1,2,4-Trimethoxybenzene        | 2.815            | Benzaldehyde, 3-hydroxy-4-methoxy | 2.286 |
| Benzaldehyde, 3-hydroxy-4-methoxy | 1.042          | 2-Propanone, 1-(4-hydroxy-3-methoxyphenyl) | 1.209 |

The relative area% for each compound was defined by the percentage of chromatographic area of the specific compound out of the total area of 50 largest identified peaks. The area% value presented here shows only the relative concentration of the specific compound among the volatile fraction of the degraded lignin that could pass through the GS column [14], [15].

From the result of GCMS analysis, it shows that the bio-based phenolic oil produced from Leucaena sp. wood contains ester, phenolic derivatives, alcohol and carboxylic acids compound. The detected esters were mainly consist of ethyl esters with carboxylic acid. The ester might have been generated by esterification between ethanol and some lignin-derived intermediates. The presence of alcohol compound could have been generated by both of the cleavage of ether linkages and lignin side chains [16]. The major group is monomeric phenolic compounds and derivative including phenolic groups with
short-chain alkane substitutes such as –methyl, -ethyl, -methylethyl, and propyl groups. It can also observed that the percentage of phenol and phenolic derivative were the largest compared to the other chemical component. As regards to results of GCMS analysis, the bio-based phenolic oil produced from gasification of Leucaena sp. wood can be considered a good potential to be used in formulation of bio-adhesive. According to Table 2 it can also observed that bio-based phenolic oil from palm kernel shell, consists of mainly phenolic derivatives and alcohol. The presence of alcohol can be predicted from the interaction between lignin side chain and the compounds present are derivatives of phenolic group with alkane substituents such as –methyl, -ethyl and -pentyl. It seems that phenolic compound shows the highest percentage of phenol area which is 32.98% compared to other chemical compounds present, thus this allows the phenolic oil to be a substitute as the component for adhesive production [17].

3.3 Parametric study of bio-adhesive

Bio-adhesive or phenol formaldehyde was produced once the chemical substances (i.e. the ethanol, formaldehyde and sodium hydroxide) as catalysts, were mixed with phenol resin. After the resinification process was done for both bio-oils, the chemical characteristics analysis was conducted using the GCMS analyzer to study the effect of reaction parameters including temperature (in values of 80, 85 and 90°C), time (in values of 30, 60 and 90 minutes), and catalyst loading (in values of 0.4, 0.5 and 0.6%) on the concentration of phenol in produced bio-adhesives for the total number of 9 samples.

3.3.1 The effect of temperature on phenol content in bio-adhesive

Temperature was varied in values of 80, 85 and 90 ºC whereby the controlled amounts of time and catalyst loading were set at 60 minutes and 0.5 g of NaOH. Figure 4 shows the effect of resinification temperature on the relative area of phenol in phenol formaldehyde samples formulated from both bio-oils from Leucaena sp. wood and PKS. According to the results related to the bio-adhesive from Leucaena sp. wood, the percentage area of phenol increased slightly as the resinification temperature increased from 80°C to 85°C. The increasing trend however became steeper at above 85 ºC until it gets to the maximum of 19.1% at 90°C. This can be explained by faster degradation rate of lignin in higher temperature which produces more phenolic compounds as the result [18]. Different trends were observed for the bio-adhesive from PKS-based oil. The highest amount of phenol at 36.13% was obtained at 85°C. Further increase in temperature above 85 ºC caused the decrease in phenol content to 21.04% at 90 ºC which probably due to the increase of reactivity of phenolic compound with formaldehyde in production of resole [12]. It is known that formaldehyde could react at ortho- or para- position of the phenol to produce ortho-methylophenols, para-methylophenols, dimethylophenols and other higher methylophenols during resinification. Moreover, according to [19] in some cases where the temperature is too high, the lignin content will be decreased and results in a decrease in phenol content inside the adhesive. It could also be explained for the decrease of phenol from 85 to 90 ºC where the overheating of the compound occurred.
3.3.2 The effect of time on phenol content in bio-adhesive

The second set of experiments was conducted to investigate the effect of different reaction time on the phenol content of bio-adhesive in resinification of bio-oils from PKS and Leucaena sp. wood. The catalyst loading were maintained at 0.5 gr of NaOH and the temperature was set at 85°C for this part of the DOE. For the bio-adhesive from PKS, according to the results which are shown in Figure 5, the content of phenol was increased with time until it reached the maximum of 36% at 60 minutes of the process. This can be explained by the progressive cracking of lignin and formation of phenol compounds with reaction time. Once the reaction time surpassed 60 minutes, the resinification rate started to increase and led to an intensive decline in the phenol area percentage as the phenolic compounds were involved in further reactions with formaldehyde. Similar trend was observed for resinification of bio-oil from Leucaena sp. wood but with more gradual fashion particularly at the onset of reaction until 60 minutes in which the phenol concentration slightly increased from initial value of 14.2% to 14.6%. Then, in the following of the reaction the phenol percent dropped to minimum of 1% after 90 minutes. The more gradual fluctuations comparing to the resinification of bio-oil from PKS can be due to lower rate of lignin degradation to phenol compounds at constant heat flux of 85°C for the bio-oil from Leucaena sp. wood. The slower degradation rate can be explained by the higher content of carbohydrate and aromatic polymers (cellulose, hemicellulose and lignin) in bio-oil from leucaena sp. wood which led to relatively higher viscosity. The phenolic resin was fully converted to bio-adhesive after 90 minutes of resinification reaction.

Figure 4. The effect of temperature on the phenol content in the bio-adhesives from Leucaena sp. wood and PKS.

![Figure 4](image-url)

**Table 1**: Phenol content (area %) at different temperatures for bio-adhesives from Leucaena sp. wood and PKS.

| Temperature (°C) | Leucaena sp. wood | PKS |
|-----------------|-------------------|-----|
| 80              | 13.208            | 24.42 |
| 85              | 14.847            | 36.13 |
| 90              | 19.1              | 21.04 |

**Table 2**: Phenol content (area %) at different reaction times for bio-adhesives from Leucaena sp. wood and PKS.

| Reaction Time (min) | Leucaena sp. wood | PKS |
|---------------------|-------------------|-----|
| 0                   | 14.2%             | 24.42 |
| 30                  | 21.04             | 36.13 |
| 60                  | 36.13             | 21.04 |
| 90                  | 1%                | 19.1  |
Figure 5. The effect of time on the phenol content in the bio-adhesives from Leucaena sp. wood and PKS.

3.3.3 The Effect of catalyst loading (NaOH) on the Percentage of Phenol (%)
The effect of catalyst loading on resinification of bio-oil has been investigated experimentally. For the resinification of both bio-oil from PKS and Leucaena sp. wood, the temperature and reaction time were maintained at constant values of 85°C and 60 minutes respectively for the best condition as obtained in previous sets of experiments. Loading of NaOH was set at three values of 0.4, 0.5 and 0.6 gr and the area percentage of phenol for each value was measured and the results are illustrated in Figure 6. For both graphs, phenol percent increases with higher catalyst loading until reaches to the maximum level with 0.5 gr of NaOH. Furthermore, increasing the catalyst loading to above 0.5 gr was observed to have no more effects on the composition of phenol which remains almost steady between 0.5 to 0.6 gr of NaOH. The existence of catalyst impacts the resinification process in two different aspects according to [18]. One aspect is related to faster degradation of lignin and hence higher production of phenolic compounds in bio-liquid solution and another is related to faster resinification reaction (as catalyst lowers the activation energy) and thus consumption of phenol with formaldehyde contents as the result which seems to have an adverse effect on phenol percent and cause it to decrease. Former effect was dominated at lower loadings of catalyst from 0.4 to 0.5 gr and caused the phenol contents to increase to maximum of 36.2% for resinification of bio-oil from PKS and 14.72% for that of bio-oil from Leucaena sp. wood. Loading of excess NaOH (more than 0.5 gr) was observe to have no significant effect on lignin degradation nor on resinification reaction and hence the composition of phenol maintained steady with just a very gradual decrease.
The process repeated in optimum condition at temperature of 85ºC, catalyst loading of 0.5 gr and within 60 minutes of resinification period for both bio-oil samples and the phenol contents were analysed via GCMS and the results are indicated in Table 3.

### Table 3. Phenol content of bio-adhesives at optimum operating condition.

| Operational Factors in best operating condition | Phenol percent % |
|-----------------------------------------------|-----------------|
| T (ºC)                                        | Bio-oil from PKS| Bio-oil from leucaena sp. wood |
| Time (minutes)                                | 36.1            | 14.7                          |
| Catalyst loading (gr of NaOH)                 | 0.5             |                               |
|                                               | Leucaena sp. wood| PKS                           |

4. Conclusions

Synthesis of bio-phenol resin was performed via updraft gasification of two types of biomass including Leucaena sp. wood and PKS for the purpose of formulating bio-adhesive with lowest possible environmental pollutants (i.e. formaldehyde). Chemical characterization was performed on the produced bio-oils and high amount of phenol contents were observed in both samples. The physical properties of bio oils were analyzed and compared to a petroleum-based pure phenolic resin which was observed to have almost similar characteristics. Bio-oil samples were treated in the production of bio-adhesive and the effect of operating parameters including reaction temperature, process time and catalyst loading on phenol content of bio-adhesive were investigated by the help of GCMS analysis. Temperature was found to have persistent increasing effect on phenol content of the bio-oil from Leucaena sp. wood however the trend was not consistent for the bio-oil from PKS. Process time was observed to have similar effect on resinification of both bio-oil samples. By introducing sodium hydroxide as catalyst to the solution, first it increased the phenol percent by effecting the rate of lignin degradation until it reaches the maximum value. Any further loading of catalyst was observed to not have any significant effect on the phenol percent. Finally, the best operational condition were found at temperature of 85 ºC, catalyst loading of 0.5 gr and within 60 minutes of resinification process for both bio-oil samples. The bio-adhesive produced at this condition has the highest amount of bio-phenol and therefore is considered an environmental friendly adhesive with noticeably lower pollution as well as cost of formulation compared to petroleum-based types.

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