

Research paper

Characteristic of Phenolated Moso Bamboo (*Phyllostachys pubesens*) Liquefied under Various Liquefaction Conditions

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【 Summary 】

Moso bamboo (*Phyllostachys pubesens*) was liquefied in phenol with sulfuric acid (H₂SO₄) or hydrochloric acid (HCl) as the catalyst. The effects of the weight ratio of phenol to bamboo and the kind and concentration of catalyst used on the characteristics of the liquefied bamboo were investigated. The results showed that liquefaction in phenol with H₂SO₄ had a better effect than with HCl as the catalyst. The former had less un-liquefied residue, more non-volatile matter, and more combined phenol than the latter. When comparing the liquefaction effect of the weight ratio of phenol to bamboo, a weight ratio of 3/1 had less un-liquefied residue and non-volatile matter, and a lower viscosity of liquefied products than with a weight ratio of 2.5/1. The FT-IR spectra showed that phenol reacted with the decomposed chemical components of bamboo and formed derivatives after liquefaction. The GPC analysis showed that liquefied bamboo had a weight average molecular weight of 1867~3550.

Key words: liquefaction, moso bamboo (*Phyllostachys pubesens*), phenolated bamboo.

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研究報告

不同液化條件酚液化孟宗竹之性質

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摘要

本研究以孟宗竹(*Phyllostachys pubesens*)為試材，酚為液化藥劑，H₂SO₄及HCl為催化劑進行液化處理，探討液化時採用之酚與竹材之重量比及使用之催化劑種類及添加量對所得液化竹材性質之影響。由試驗結果得知，孟宗竹在酚液體中進行液化處理時，以H₂SO₄為催化劑者之液化效果優於HCl，前者之液化產物有較低之殘渣率，較高之不揮發分及結合酚含量。比較液化時酚與竹材之重量比對液化效果之影響，液化時採用重量比3/1者其液化產物之殘渣率、不揮發分及黏度低於採用重量比2.5/1者。由FT-IR光譜分析顯示經液化處理後，酚與竹材之降解之化學組成成分產生反應並形成衍生物。GPC分析則顯示液化竹材之重量平均分子量介於1867~3550間。

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INTRODUCTION

Petroleum is a complex mixture containing various aliphatic and aromatic compounds. Nowadays, much of the chemical raw materials used in various industries predominantly come from the processing of petroleum such as distilling, separating, and refining. But petroleum is a non-renewable resource, and it will likely be exhausted in the near future. So, it is urgent that we find alternative resources. Plants are biomass materials with a renewable characteristic and are the most plentiful resources on earth. They are composed of natural polymers such as cellulose, hemicellulose, and lignin. But their solid nature restricts their application. If we can convert them from a solid to a liquid, the utilization of plants can substantially be extended.

Solvent liquefaction is one of the ways to convert the biomass of plants from a solid to a liquid. The liquefied products can be used to prepare various kinds of synthetic resins depending on the solvent used. If using phenol as the solvent, the liquefied products can be used to prepare phenolic resins. Alma et al. (1995a, b, 1996) liquefied birch wood in phenol with HCl as the catalyst, and the results showed that increasing the weight ratio of phenol to birch and the concentration of catalyst decreased the un-liquefied residue and increased the combined phenolics. They indicated that the phenolated wood blended with hexamethylenetetramine could be used as molding materials. Alma and Basturk (2006) liquefied grapevine cane with phenol in the presence of H₂SO₄ as the catalyst at 150°C for 2 h. The liquefied grapevine cane powder could be used to prepare the resol-type

phenol-formaldehyde resin (PF) when reacted with formaldehyde under alkaline conditions. Lee et al. (2000b, 2002) used phenolated corn bran to prepare novolak-type PF resins and phenolated wood to prepare resol-type PF resins.

In another liquefaction system, polyhydric alcohol is used as the solvent. The liquefied products can be used to prepare polyurethane (PU) and epoxy resins. Kurimoto et al. (2000, 2001b) liquefied Japanese cedar (*Cryptomeria japonica* D. Don.) using the co-solvents, polyethylene glycol-glycerol, as the reaction reagent in the presence of H₂SO₄. The liquefied woods obtained were blended with polymeric methylene diphenylene diisocyanate (PMDI) to prepare PU films. Lee et al. (2000a) indicated that liquefied corn bran can be used to prepare PU foams successfully. Tohmura et al. (2005) prepared isocyanate adhesives by mixing polyhydric alcohol-liquefied wood with PMDI, and they thought the resultant material had the potential to become a wood adhesive. Kobayashi et al. (2000, 2001) used the same liquefied products to prepare liquefied wood/epoxy resin. They found that liquefied wood was incorporated into the resin in the form of cross-linked polymer networks, and had a similar shear bonding strength to commercial epoxy resin.

The effects of liquefaction conditions on the characteristics of the liquefied products have been probed by some other researchers. Lin et al. (2001a, b, 2004) used β-O-4 lignin and cellobiose as model compounds to study the liquefaction mechanisms of lignin and cellulose in the presence of phenol with acid catalysts. They found that the yield

ratios of various compounds in the end reaction product greatly depended on the reaction conditions, i.e., the phenol/wood ratio, the concentration of the acid catalyst, the reaction temperature, and reaction time. Lee and Wang (2005) investigated the effect of water on wood liquefaction and the properties of phenolated wood. Pan et al. (2007) characterized the properties of the residue from wood liquefaction to understand some fundamental aspects of the liquefaction reaction and indicated that lignin dissolved prior to cellulose during liquefaction. Zhang et al. (2006) liquefied cellulose with phenol in the presence of H₂SO₄. They pointed out that the linkage fashion of bound phenol was governed by the liquefaction conditions, and the bound phenol did not take a phenoxy form but rather a hydroxyl benzyl form.

Many kinds of woody materials have been liquefied with the above solvent. Kurimoto et al. (1999, 2001a) indicated that the liquefaction reaction and re-condensation were strongly influenced by the wood species. Bamboo is one of the most important forest products in Taiwan. Nearly 60 species of bamboo are grown in about 150,000 ha. As bamboo shoots emerge from the ground, the diameter increases very quickly, and plants grow to their full height in approximately 60–70 d. These are renewable natural resources with enormous potential. Pu and Shiraishi (1993) investigated the liquefaction of bamboo with phenol as the solvent reagent, but that was the only published report we are aware of. Therefore, the objective of this article was to liquefy moso bamboo in phenol with H₂SO₄ or HCl as the catalyst, and investigate the influence of liquefaction conditions on the characteristics of the liquefied products.

MATERIALS AND METHODS

Materials

Moso bamboo (*Phyllostachys pubesens* Mazel ex H. de Len), approximately 5 yr old, was felled and collected from Lugu, Nantou, central Taiwan. All of the bamboo was air-dried and ground to a powder in a hammer mill with a 2-mm-diameter screen. The bamboo powder with dimensions between 20 and 60 mesh was dehydrated in a vacuum oven at 60°C. Chemicals such as H₂SO₄, HCl, phenol, methanol, acetic anhydride, and pyridine were reagent grade and were used without further purification. Tetrahydrofuran (THF) was HPLC grade, and was used as a solvent and eluant for gel permeation chromatography. Potassium bromide (KBr) was used for FT-IR spectrometry.

Liquefaction of bamboo in phenol

To liquefy moso bamboo, phenol and the acid catalyst were thoroughly premixed in a 1000-mL separable glass flask equipped with a stirrer, thermometer, and reflux condenser. The reagent was stirred and heated to 110°C by an electric heating mantle. Then the bamboo powder was gradually added to the reaction flask. After all of the bamboo powder had been added, the temperature of reactant was increased to the set temperature, and stirring and heating continued for 60 min to allow the liquefaction reaction to proceed. The liquefaction conditions are shown in Table 1.

Properties of liquefied bamboo

After liquefaction, the un-liquefied residue, non-volatile matter content, and viscosity of the liquefied bamboos were measured, and the combined phenol and free phenol were calculated for the non-volatile matter content. To measure the un-liquefied residue, 5 g of liquefied bamboo was weighed and diluted with 100 mL of methanol, and then filtered through a G3 glass filter in a vacuum. The

Table 1. Liquefaction conditions and properties of liquefied moso bamboo

Code	Liquefaction conditions					Property				
	Phenol to bamboo (w/w)	Catalyst		Temp. (°C)	Time (min)	Residue ¹⁾ (%)	Visc. (cps)	Non-Volatile matter (%)	Combined phenol (%)	Free phenol (%)
		Kind	Rate (%)							
S1	3/1	H ₂ SO ₄	5	130	60	1.3	30,500	61.4	37.6	33.8
S2	2.5/1	H ₂ SO ₄	5	130	60	8.0	164,000	69.6	42.0	27.0
S3	2.5/1	H ₂ SO ₄	3.5	130	60	15.3	80,000	73.5	45.6	24.4
C1	3/1	HCl	10	120	60	18.5	2,750	42.2	19.5	48.7
C2	2.5/1	HCl	10	120	60	27.8	6,400	46.6	19.9	46.8
C3	3/1	HCl	10	110	60	24.4	2,250	41.1	18.4	49.8

¹⁾ Based on the weight of bamboo.

un-liquefied solid residue was dried in an oven at $103 \pm 2^\circ\text{C}$. The residue content was calculated based on the weight of the bamboo. The viscosity was measured using a Brookfield viscometer at 25°C . The non-volatile matter was measured using a rotary vacuum evaporator by heating at 180°C to remove the un-reacted phenol and was calculated by the following equation: $S(\%) = W_s / W_1 \times 100$; where W_s is the weight of the non-volatile matter after heat evaporation, and W_1 is the weight of the liquefied bamboo before evaporation treatment. Combined phenol and free phenol were used to show the phenol existing in the liquefied bamboo that was combined with the chemical components of the bamboo or was free in the system, respectively. Both the combined and free phenol contents were calculated from the non-volatile matter content of the liquefied bamboo. Combined phenol was calculated by subtracting the content of bamboo used in the liquefied system from the non-volatile matter content after liquefaction. Free phenol was calculated by subtracting the content of combined phenol from the content of phenol used in the initial liquefaction system.

FT-IR spectrum analysis

The liquefied bamboo was diluted with

methanol and filtered through a G3 glass filter. The filtrate was concentrated in a rotary vacuum evaporator at 180°C followed by oven drying to remove the methanol and un-reacted phenol. The residual solid ingredient was ground and blended with KBr powder at a weight ratio of 1:100. The FT-IR spectra were obtained in a Fourier transform infrared spectrometer (Mattson Genesis II, WI, USA), where using a diffuse reflectance accessory with a deuterated triglycine sulphate (DTGS) detector. The infrared scanning wave-number was $4000\text{--}400\text{ cm}^{-1}$ at a resolution of 4 cm^{-1} .

Molecular weight and weight distribution of liquefied bamboo

The molecular weight and weight distributions of un-acetylated and acetylated liquefied bamboo were determined using a Hitachi (Tokyo, Japan) L-6200A gel permeation chromatograph (GPC) equipped with a Phenomenex $5\ \mu\text{m}$ 100A column (CA, UAS) and monitored with a UV detector. The acetylation of the liquefied bamboo was conducted with acetic anhydride/pyridine (1/1; v/v). Samples were filtered through a $0.45\text{-}\mu\text{m}$ filter film to remove the residue followed by vacuum evaporation to remove the free phenol, un-reacted acetic anhydride, and pyridine. The dried liquefied bamboo was re-

dissolved in THF at a concentration of 0.1%. The injection volume of the sample was 20 μL . THF was used as the eluant with a flow rate of 1 mL min^{-1} . Monodispersed polystyrenes were used as standards for calibrating the molecular weight.

RESULTS AND DISCUSSION

Properties of liquefied bamboo

Table 1 shows the liquefaction conditions and properties of liquefied moso bamboo. Results indicated that liquefaction with H_2SO_4 as the catalyst produced a better liquefaction effect than with HCl as the catalyst. The un-liquefied bamboo residue of the former was 1.3, 8.0, and 15.3% based on the weight of bamboo used for S1, S2, and S3, respectively, while those of the latter were 18.5, 27.8, and 24.4% for C1, C2, and C3, respectively. Comparing among the various liquefaction conditions, liquefaction with a weight ratio of phenol to bamboo of 3/1 had the residue lower than 2.5/1. This trend was the same as our previous studies for the liquefaction of Taiwan acacia, China fir, and Japanese cedar, but liquefied bamboo had a higher un-liquefied residue than those 3 woods (Lee et al. 2003, 2004, 2006). This may have been caused by bamboo having a lower lignin content than wood. But lignin is easier to liquefy than carbohydrates (Pan et al. 2007). So, wood should have a lower un-liquefied residue. The effect of liquefaction temperature on the residue content can be compared between C1 and C3: the former was liquefied at 120°C and had less residue than the latter processed at 110°C , indicating that increasing the temperature can promote the liquefaction reaction.

The viscosity of liquefied bamboo predominantly depended on the kind of catalyst. As described previously, bamboo liquefied with H_2SO_4 as the catalyst had less residues than

that with HCl, but bamboo processed with H_2SO_4 as the catalyst had a significantly higher viscosity. This might be because the decomposed lignin undergoes a re-condensation reaction under acid liquefaction conditions (Kurimoto et al. 1999). The stronger acidity of H_2SO_4 promoted greater re-condensation than HCl, resulting in a higher viscosity. In addition, cellulose decomposes to glucose and is then transformed to 5-hydroxymethylfurfural, that further provides levulinate and polymerized derivatives which increase the viscosity at the last stage of liquefaction (Zhang et al. 2006). This phenomenon can be proven by the viscosities of liquefied bamboo of S2 and S3. These were liquefied with the same weight ratio of phenol to bamboo but had different dosages of H_2SO_4 . S2 liquefied with a 5% catalyst had a viscosity of 164,000 cps, but S3 liquefied with only 3.5% H_2SO_4 had a viscosity lower than that of S2. These results indicate that the liquefaction of lignocellulose should be carefully controlled to avoid a re-condensation reaction.

Figure 1 displays the viscosity variation of S1 and S2 liquefied bamboo during the period of liquefaction. The weight ratios of phenol to bamboo for S1 and S2 were 3/1 and 2.5/1, respectively. As shown in Fig. 1, the viscosity of S1 slightly increased during the period of liquefaction, but that of S2 drastically increased at reaction times of between 30 and 45 min. Such a result indicates that less solvent in the liquefaction system makes it more difficult to control the reaction.

The non-volatile matter contents shown in Table 1 were measured using a vacuum evaporator heated to 180°C to remove the unreacted phenol. At the initial stage of liquefaction, the weight percentages of bamboo were between 23 and 26% for various liquefaction conditions. But as shown in Table 1, all of the non-volatile matter contents after liquefaction

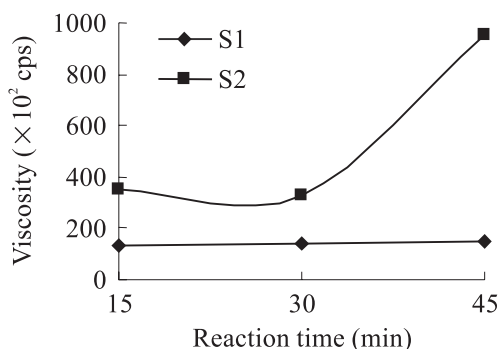


Fig. 1. Viscosity variation of S1 and S2 during the period of liquefaction. S1 and S2 were liquefied with the weight ratios of phenol to bamboo of 3/1 and 2.5/1, respectively, and 5% H_2SO_4 as the catalyst.

exceeded 26%. This result means that some of the phenol had combined with the bamboo, been transformed as a derivative, and could not be removed by heating. Comparing among all of the liquefied bamboos, those liquefied with H_2SO_4 as the catalyst had higher non-volatile matter than those with HCl. The non-volatile contents were then used to calculate the phenol that had combined with the chemical components of the bamboo (referred to as combined phenol) or free in the liquefied bamboo (referred to as free phenol). As shown in Table 1, combined phenol for liquefied bamboo using H_2SO_4 as the catalyst was 37.6~45.6%, but was only 18.4~19.9% for those using HCl as the catalyst. This result further indicates that using H_2SO_4 as the catalyst had a better effect for liquefying bamboo in phenol.

FT-IR analysis of liquefied bamboo

Figure 2 shows the FT-IR spectra of moso bamboo and liquefied bamboo in the range of 400 to 4000 cm^{-1} . As shown in the spectra, bamboo liquefied in phenol with H_2SO_4 (S1) and HCl (C1) as the catalyst had similar spectra, suggesting they had the same

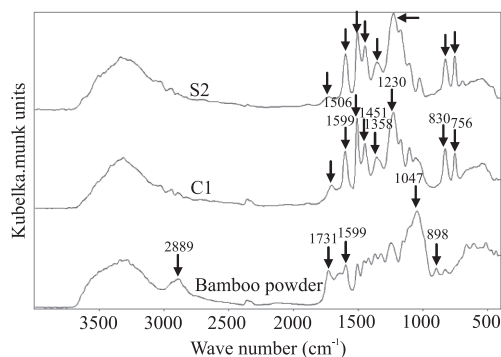


Fig. 2. FT-IR spectra of liquefied bamboo and bamboo powder. C1, Liquefied with a weight ratio of phenol to bamboo of 3/1 and 10% HCl as the catalyst. S2, Liquefied with a weight ratio of phenol to bamboo of 2.5/1 and 5% H_2SO_4 as the catalyst.

products. However, they had obvious differences with untreated bamboo, indicating that the structure of the chemical components of the bamboo had been changed by liquefaction. Untreated moso bamboo had a strong absorption band at 2889 cm^{-1} , which was attributed to C-H and CH_2 stretching vibrations of aliphatic hydrocarbons in cellulose and hemicellulose. But this absorption band was not obvious for liquefied bamboo, indicating that much of the carbohydrate components had been destroyed by liquefaction. The absorption band at 1731 cm^{-1} was attributed to the C=O stretching vibration that is characteristic of un-conjugated ketones, aldehydes, and carboxyls of lignin and hemicellulose. This absorption was stronger for untreated bamboo than liquefied bamboo, indicating that some of the lignin and hemicellulose had been degraded. The absorption bands at 1599, 1506, and 1451 cm^{-1} were attributed to aromatic ring stretching vibrations. These signals were much stronger in liquefied bamboo than untreated bamboo. In addition, liquefied bamboo had a new absorption band which appeared at 1358 cm^{-1} that was attributed to the in-plane

deformation of phenolic OH, and absorption bands at 830 and 756 cm^{-1} attributed to benzene derivatives. All of these results indicate that phenol was introduced into the structure of phenol-liquefied bamboo. Boeriu et al. (2004) indicated that the vibration originating from carbohydrates was associated with other vibrations in the region of the spectrum between 1000 and 1300 cm^{-1} . After liquefaction, the C-O-C symmetrically stretching vibration at 1047 cm^{-1} became weak, but a strong and obvious absorption band appeared at around 1230 cm^{-1} . This result indicated that the structure of the chemical components of bamboo had been destroyed, but had reacted with phenol to form a derivative with new ether bonds. The absorption band at 898 cm^{-1} was attributed to the C-H deformation vibration of the cellulose β -anomeric linkage. It disappeared after liquefaction, indicating that cellulose had been degraded.

Figure 3 shows the FT-IR spectra of the un-liquefied solid residue in the liquefied bamboo. Some of the characteristic absorption bands which appeared in bamboo can

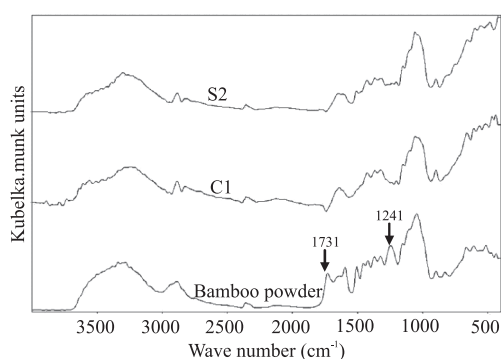


Fig. 3. FT-IR spectra of the residue of liquefied bamboo and bamboo powder. C1, Liquefied with a weight ratio of phenol to bamboo of 3/1 and 10% HCl as the catalyst. S2, Liquefied with a weight ratio of phenol to bamboo of 2.5/1 and 5% H_2SO_4 as the catalyst.

also be seen in the un-liquefied residue, such as around 3300 cm^{-1} attributed to OH groups, 2890 cm^{-1} attributed to C-H and CH_2 , 1200~1000 cm^{-1} attributed to the pyranose ring, and 898 cm^{-1} attributed to the C-H deformation vibration of the cellulose β -anomeric linkage. But the absorption band at 1731 cm^{-1} attributed to the carboxyl or carbonyl C=O stretching vibration and 1241 cm^{-1} attributed to the C-O stretching vibration of the methoxy group of guaiacyl ring (Hassan 2003) disappeared in the un-liquefied bamboo residue, suggesting that the acetyl groups of hemicellulose and the methoxy groups of lignin had been broken during liquefaction. By comparing between liquefied bamboo and the residue, the characteristic absorption attributed to the aromatic ring or phenolic ring appearing in the liquefied wood was not clear as that in the un-liquefied residue. This indicates that the main component of the residue still belonged to wood, not to phenolated wood.

Molecular weight and distribution of liquefied bamboo

The chemical components of bamboo include linear polymers such as cellulose and hemicellulose and network polymers such as lignin. The molecular chains of cellulose and hemicellulose, although they have a linear structure, are held together via intermolecular and intramolecular hydrogen bonds. Therefore, they are hard to disperse and dissolve in a solvent under normal conditions. In this study, bamboo was liquefied in phenol using H_2SO_4 or HCl as the catalyst. Consequently, the components of bamboo were degraded to a small segment followed by reaction with phenol, transformation as phenolic derivatives, and dissolution in free phenol. The molecular weight and molecular weight distribution of acetylated and un-acetylated liquefied bamboos were determined by the GPC an-

alysis. Williams et al. (1983) used GPC to measure the molecular weight of condensed tannin, but they indicated the result was influenced by hydrogen bonds caused by the strong polarity of hydroxyl groups in tannin. Chen and Hatano (1990) indicated that the measured molecular weight of the unmodified bark extractive was lower than its true value. They thought that the high-molecular weight portion of the bark extractive had not really dissolved in THF and was excluded at the filtration stage.

Table 2 shows the weight-average molecular weight (Mw), number-average molecular weight (Mn), and molecular weight polydispersity (Mw/Mn) of various acetylated and un-acetylated liquefied bamboo samples examined in this study. The Mw of acetylated and un-acetylated liquefied bamboo samples were 1867~3550 and 1872~3028, respectively. The former was slightly higher than the latter. The Mw/Mn of acetylated liquefied bamboo (1.1~1.6) was higher than that of un-acetylated liquefied bamboo (1.0~1.4). As shown above, all of the liquefied bamboo had an average molecular weight of oligomers not polymers. This suggests that the chemical components of bamboo had been drastically degraded by the liquefaction reaction. However, these molecular weights were lower

than those of liquefied Japanese cedar in our previous study (Lee et al. 2006).

Figure 4 is the molecular weight distribution of the S1 and C1 liquefied bamboo. The molecular weight distribution of the liquefied bamboo exhibited the characteristic of polydispersity which could be distinguished into several regions. For acetylated liquefied bamboo, 3 major regions were evident: molecular weights < 1600, 1600~1800, and > 1800. These 3 molecular weight distribution regions might have been caused by the different liquefaction capabilities of cellulose, hemicellulose, and lignin, but this hypothesis was not proven in this study. In addition, as shown in Fig. 4, the molecular weight distribution of acetylated liquefied bamboo had shifted to a higher region than un-acetylated liquefied bamboo. This might be attributed to 2 reasons. First, after acetylation, hydroxyl groups in the liquefied bamboo were substituted by acetyl groups, which have much greater molecular weight than hydrogen atoms. Second, acetylation would improve the solubility of the high-molecular-weight component in THF, and they would not be excluded at the filtration stage. So, a higher molecular weight was obtained.

Compare the influence of liquefaction conditions on the molecular weight as shown

Table 2. Average molecular weight and dispersion of liquefied bamboo

Liquefied bamboo	Acetylated			Unacetylated		
	Mn ¹⁾	Mw ²⁾	Mw/Mn ³⁾	Mn ¹⁾	Mw ²⁾	Mw/Mn ³⁾
S1	1863	2047	1.1	1938	2004	1.0
S2	2086	2386	1.1	2024	2238	1.1
S3	2084	3049	1.5	1931	2404	1.2
C1	2023	2867	1.4	1835	1872	1.0
C2	2025	2140	1.1	1908	2105	1.1
C3	2210	3550	1.6	2122	3028	1.4

¹⁾ Mn, number-average molecular weight.

²⁾ Mw, weight-average molecular weight.

³⁾ Mw/Mn, the molecular weight polydispersity.

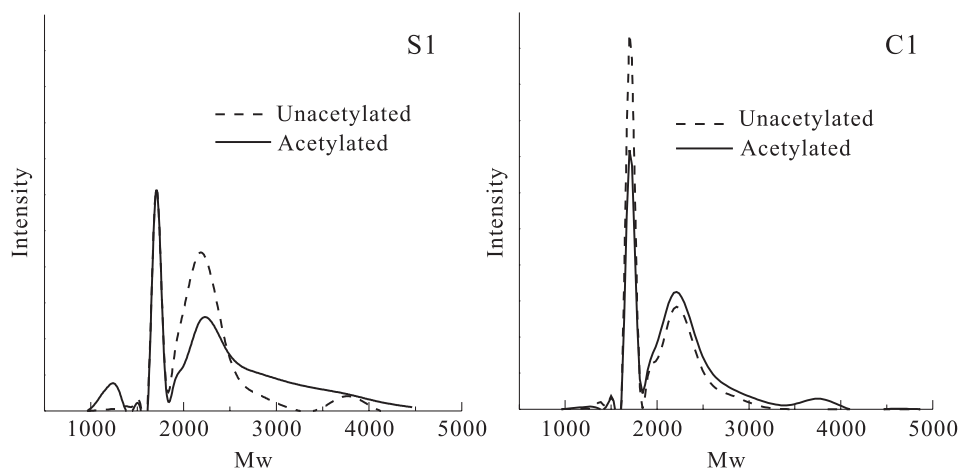


Fig. 4. Molecular weight distribution of liquefied bamboo of S1 and C1. S1, Liquefied with a weight ratio of phenol to bamboo of 3/1 and 5% H_2SO_4 as the catalyst. C1, Liquefied with a weight ratio of phenol to bamboo of 3/1 and 10% HCl as the catalyst.

in Table 2. With a higher weight ratio of phenol to bamboo, there was a lower Mw, i.e., $S1 < S2$ and $C1 < C2$. Moreover, the greater the amount of catalyst used, the lower the Mw was ($S2 < S3$). And the higher the reaction temperature used, the lower the Mw was ($C1 < C3$). All of these results suggest that more solvent and catalyst in the liquefaction system and a higher reaction temperature promoted the liquefaction reaction and induced a lower molecular weight of the liquefied bamboo. These results are similar to those of Lin et al. (1997) who liquefied birch wood in phenol using H_2SO_4 as a catalyst under various liquefaction conditions.

Figure 5 shows the molecular weight distribution of acetylated liquefied bamboo under various liquefaction conditions. Comparing liquefied bamboo of S1 and S2, which had the same dosage of H_2SO_4 but a different weight ratio of phenol to bamboo, both of them had similar molecular weight distribution curves. But S1 had a higher intensity in the lower-molecular-weight region, suggesting that liquefaction with a higher weight ratio of phenol to bamboo was advantageous

for the decomposition reactions. Comparing liquefied bamboo of S2 and S3, both of them had the same weight ratio of phenol to bam-

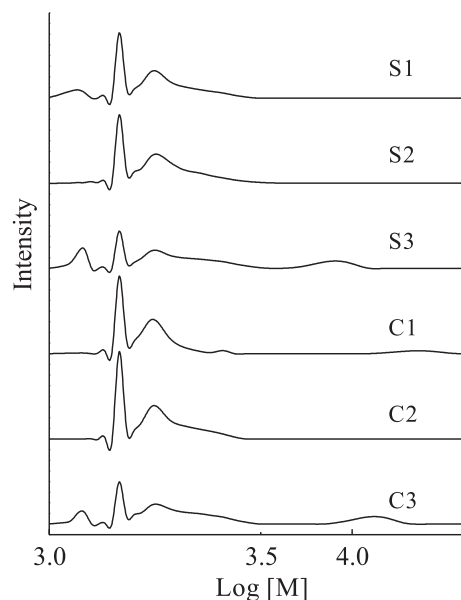


Fig. 5. Molecular weight distribution of various acetylated liquefied bamboos. S1, S2, and S3 were liquefied with H_2SO_4 as the catalyst. C1, C2, and C3 were liquefied with HCl as the catalyst.

boo but the dosage of H_2SO_4 for S3 was less than that for S2. The GPC analysis curve for S3 shows a high-intensity signal for molecular weights of < 1600 and another signal at molecular weights of > 5500 , but those were not evident for S2. This result suggests that the molecular weight of liquefied bamboo with a lower dosage of acid catalyst during liquefaction would be non-uniform, and have a broader range of molecular weight distribution.

When HCl was used as the catalyst for liquefaction, both C1 and C2 had the same liquefaction conditions except the weight ratio of phenol to bamboo, and these produced similar molecular weight distributions. But C1 and C3 were liquefied at different temperatures and had greatly different molecular weight distributions. C3 which was liquefied at a temperature of $110^\circ C$ had components with molecular weights of < 1600 and > 10000 . This suggests that a temperature of $110^\circ C$ is not sufficient to obtain uniform liquefaction.

CONCLUSIONS

Moso bamboo was liquefied in phenol with H_2SO_4 or HCl as the catalyst and with the weight ratios of phenol to bamboo of 2.5/1 and 3/1 at temperatures of 110 to $130^\circ C$ for 60 min. The results showed that liquefaction with H_2SO_4 as the catalyst had a better effect than HCl under the same reaction conditions. The FT-IR spectra showed that phenol reacted with the bamboo and formed derivatives after liquefaction. The GPC analysis showed that liquefied bamboo had weight average molecular weights of 1867~3550. But the weight ratio of phenol to bamboo, the dosage of catalyst, and the temperature of liquefaction all influenced the molecular weight distribution of the liquefied bamboo.

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