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## Short Communication

## Comparative study of liquefaction process and liquefied products from bamboo using different organic solvents

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## ABSTRACT

The effects of various solvents, including phenol, ethylene glycol (EG) and ethylene carbonate (EC), and different liquid ratios on the liquefaction of bamboo, have been studied systematically in this paper. The processes were catalyzed by hydrochloride acid at 180 °C in autoclaves for different reaction times. The results show that phenol is the optimum solvent for bamboo liquefaction with a yield up to 99%. The Fourier transform-infrared (FT-IR) analyses of the residues show that cellulose, hemicelluloses and lignin are almost decomposed when using phenol as solvent. The gel permeation chromatography (GPC) results of the liquid products show that the high molecular weight of bamboo decreases significantly to around 1800 g mol<sup>-1</sup> after liquefaction. The gas chromatography and mass spectrometry (GC-MS) analysis shows that low boiling point products of liquefied bamboo are similar regardless of the type of solvent used.

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## 1. Introduction

The depletion of non-renewable resources, such as petroleum, encourages the search for alternative renewable resources which are environmentally friendly and sustainable in nature. Biomass is a primary candidate as it is the only renewable source of fixed carbon, which is essential in the production of conventional hydrocarbon liquid transportation fuels and many consumer goods (Budija et al., 2009). Ever since Fierz-David (1925) converted wood and plant into liquid, there have been many studies on the liquefaction of bioresources. The primary objective has always been the conversion of renewable materials and residual biomass into bio-oil (Zheng et al., 2008). Typical liquefaction without a catalyst is conducted under drastic conditions, such as high pressures and temperatures (around 300–500 °C) (Wu et al., 2009). However, with a correctly selected solvent system, the biomass can be liquefied by using acidic catalysts at temperatures ranging from 120 to 180 °C (Wang et al., 2008a,b).

Various agricultural and forestry residues can be deemed as abundant and renewable such sources, but bamboo is the only biomass that is investigated in this paper. It is a fast growing species that does not require replanting as its vast root network continually sprouts new shoots. Bamboo is regarded as a potential bioenergy feedstock, yet remarkably little is known about this entire sub-family of tall graminaceous plants, despite its everyday utiliza-

tion for fibre and food (Scurlock et al., 2000). As bamboo is a very important renewable resource in Asia, it is therefore worthwhile to further investigate its applications. For instance, Sun and Li (2007) studied the liquefaction of bamboo with phenol and prepared adhesive from this phenol-liquefied bamboo with formaldehyde. Zhou et al. (2008) also studied the technique for preparing phenol-formaldehyde resin adhesive from bamboo tar.

There are existing studies on liquefaction of biomass through various solvents. However, the outcome of these studies is not suitable for comparison because there is discrepancy in the separation of products, defining the liquid products and difficulties in creating identical conditions to carry out the studies. These problems make it difficult to accurately measure the effects of solvents on the conversion rate, product distribution and residue properties. In the present paper, we will systematically report how organic solvents; namely, phenol, EG and EC, liquid ratio and reaction time, affect the bamboo liquefaction yield and the liquefied products. This study is a part of our ongoing attempts in creating a reliable liquefaction process which can generate desired products in a more selective and controllable manner.

## 2. Methods

## 2.1. Materials

*Phyllostachys angusta* bamboo (Moso bamboo) from the Guangdong province in Southern China was used as raw material. The air-dried sample was milled in a universal high-speed smashing

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machine and then sieved to give fractions with particle size ranges of 0.9–0.2 mm (20–80 mesh). Water, extractives, lignin and ash were determined by using the Chinese GB standard (GB/T 2677). The content of hemicellulose was tested according to a method introduced by [Yang et al. \(2006\)](#). The chemical composition of bamboo consists of lignin (24.66%), cellulose (36.11%), hemicellulose (33.16%), ethanol–benzene soluble (3.25%), ash (2.82%) and water (1.31%). The elemental composition of the bamboo sample was determined by an EA I4401 element analyzer. The oxygen content was estimated based on an assumption that the samples only contained the elements of C, H, O, N and S. The elemental composition of bamboo is C (46.19%), H (7.38%), O (45.41%), N (0.41%) and S (0.61%). Three different types of solvents; namely, phenol, EG and EC, were purchased from Oriental Chemicals & Lab. Supplies Ltd. (analytical grade) and used as received.

## 2.2. Liquefaction procedure

The influence of the ratio between different solvents to bamboo (2:1, 3:1, 5:1, 10:1), and reaction times (2, 6, 10, 14, 18 h) on liquefaction yield (LY) was studied. One gram of bamboo mixed with a relevant mass of solvent and hydrochloric acid (5 wt% of the solvent) were loaded into 25 ml Teflon-lined stainless steel autoclaves. The temperature of the reaction was kept constant at 180 °C. The working pressures of different solvents at the reaction temperature were calculated from the steam tables ([Smith et al., 2006](#)). After the reaction, the residue was separated by filtration, then rinsed thoroughly with methanol and dried at 105 °C until the weight remained unchanged. LY is calculated with the following equation:

$$\text{Liquefaction yield (\%)} = (1 - \text{Mass of residue/Mass of raw material}) \times 100\% \quad (1)$$

## 2.3. Product analysis

The solvent to bamboo ratio of 10:1 was treated at 180 °C for 14 h and selected to investigate the effect of different solvents on the properties of liquefied products. The FT-IR spectra of bamboo residues with equal weights molded in KBr pellets were analyzed by a Perkin–Elmer 100 spectrometer. The molecular weight of the liquid products was analyzed by GPC on a Waters high performance liquid chromatograph (HPLC), equipped with a voltage detector and an Agilent MIXED-B column. The mobile phase was tetrahydrofuran (THF), which has a flow rate of 0.8 mL/min. The concentrations of the test samples were 0.5 wt% in THF, and the injection size was 20  $\mu$ L. The average molecular weight of the samples was calculated by using a calibration curve of monodisperse polystyrene ( $M_w = 1,290,000, 552,500, 66,000, 22,000, 970$ ).

The lower boiling point components in the liquid products were analyzed on an Agilent 7890/5973C GC–MS. The GC was fitted with a 30 m  $\times$  0.25 mm  $\times$  0.25  $\mu$ m fused quartz capillary column and coated with HP-5MS. Helium (99.999%) was used as the carrier gas with a constant flow of 1.0 mL/min. The oven was programmed with a 10 °C/min increase to a final temperature of 280 °C and held for 10 min. After a solvent delay of 5 min, full scan mass spectra were acquired from 50 to 650  $m/z$ . The injection size was 1  $\mu$ L. The identification of the peaks was based on computer matching of the mass spectra with the National Institute of Standards and Technology (NIST) 2005 library.

## 3. Results and discussion

### 3.1. Effect of solvent type on the LY

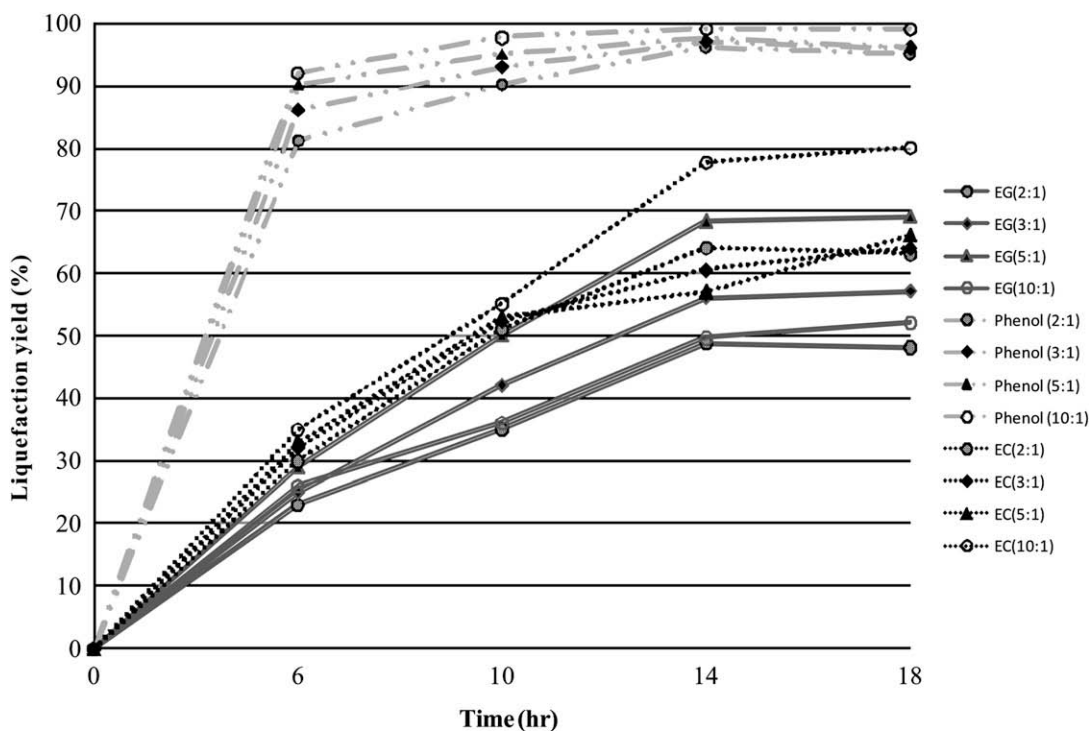
The influence of different solvents, liquid ratios and reaction times on the LY at a constant reaction temperature of 180 °C is shown in [Fig. 1](#). It can be seen that the LY increases when the reaction time is increased to 14 h. A further increase in the reaction time to 18 h might result in undesirable re-condensation and reduce the LY. Therefore, it was established that 14 h of reaction time is optimal in our system.

It was found that solvent types have a marked influence on the LY. Phenol is the optimal solvent for liquefying bamboo as the LY reaches 99% at the liquid ratio of 10:1, while the highest yields of EG and EC are only be 69% and 80%, respectively. The highest liquefaction efficiency of phenol can be attributed to its excellent solvolysis ability towards lignocellulose and higher working pressures at 180 °C. [Lin et al. \(2004\)](#) proposed the liquefaction mechanism of cellulose in the presence of phenol under acid catalysis. They suggested a glucose ring with consequent phenolation, dehydration, cleavage of the carbohydrate part, which leads to formation of various phenolated compounds. [Karagöz et al. \(2004\)](#) also showed that the intermediate product of liquefied lignocellulose contains a substantial amount of phenol derivatives. These intermediates have a similar polarity to phenol which can be dissolved efficiently and thus give a higher LY. On the other hand, the difference in liquefaction efficiency among EG and EC can be attributed to their dielectric constant values. The dielectric constants of EG and EC at 40 °C are 38.4 and 90.5, respectively. It is known that for an acid-catalyzed reaction in nonaqueous solvents, the acid potential depends on the permittivity of the solvent. A relatively high permittivity can lead to a high acid potential ([Yamada and Ono, 1999](#)). Higher dielectric constants of EC therefore result in a higher LY than that of EG.

The influence of the liquid ratio on the LY was also investigated. When using phenol as a solvent, the LY increased slightly with increases in the liquid ratio, which is consistent with the results from [Lee et al. \(2000\)](#). When EC was used as a solvent, the LYs showed no obvious differences among the liquid ratios of 2:1–5:1, but increased significantly at a ratio of 10:1. It can be concluded that the rate of liquefaction reaction is much faster than re-condensation at a liquid ratio of 10:1 ([Xie and Chen, 2005](#)). The optimal liquid ratio for EG is 5:1. Below this ratio, there is insufficient EG to liquefy bamboo and above this ratio, the surplus EG will conduct re-condensation reactions and lower the LY.

### 3.2. The FT-IR spectra of the residues

The FT-IR spectra of the raw bamboo and solid residues after liquefaction with phenol, EG and EC were studied. Bamboo is mainly constituted of cellulose, hemicellulose and lignin. From its FT-IR spectra, the peak at 1638  $\text{cm}^{-1}$  can be attributed to unconjugated C=O bonds of xylans in hemicelluloses ([Pandey and Pitman, 2003](#)). The absorption at 1400  $\text{cm}^{-1}$  (benzene ring) represents the lignin, and absorption at 1165  $\text{cm}^{-1}$  corresponds to the  $\beta$  glycosidic bond vibration in cellulose ([Liu et al., 2008](#)). When using phenol as the solvent, all of these peaks (1638, 1400 and 1165  $\text{cm}^{-1}$ ) obviously diminish, implying that phenol decomposes cellulose, hemicellulose and lignin. On the other hand, it was found that the band at 1638 and 1400  $\text{cm}^{-1}$  almost disappears after liquefaction with EC. This result indicates that EC liquefies hemicelluloses and lignin prior to cellulose. After liquefaction with EG, a new band of 1707  $\text{cm}^{-1}$  indicates



Working pressures (MPa) for different solvents at 180 °C

Ratio	EG	Phenol	EC
2:1	0.487	0.527	0.432
3:1	0.549	0.587	0.497
5:1	0.646	0.686	0.601
10:1	0.86	0.903	0.806

Fig. 1. The influence of the ratio between different solvents and bamboo, and reaction times on LY at a reaction temperature of 180 °C.

that ketones are produced in the process of liquefaction. These ketones could be attributed to lignin derivatives and further reaction of sugars which come from the degradation of cellulose and hemicellulose. The peaks that appear at 1510 and 1462  $\text{cm}^{-1}$  represent benzene derivations that are decomposed from lignin.

### 3.3. Gel permeation chromatography (GPC) of liquid products

GPC is applied to obtain the molecular weight distribution of the liquid products. In our study, the molecular weight is evaluated through the retention time by referring to the calibration curve deduced from standard samples. This calibration curve reflects a relationship between retention time and molecular weight. The GPC results showed that the molecular weight distribution curves are very similar among the three different solvents and can be divided into two major peaks; one with a shorter retention time, which corresponds to higher molecular weight and the other one that is sharp and narrow, contributes to a smaller molecular weight, which can consist of the unreacted solvent and the smaller molecules produced by in-depth decomposition of the lignocellulose of bamboo. After liquefaction, the molecular weights show a significant drop-off. The average molecular weight ( $M_w$ ) of the first peak of the liquid products from EC, EG and phenol are 1877, 1815, and 1786  $\text{g mol}^{-1}$ , respectively and the dispersion is 1.0015, 1.00116, and 1.009, respectively. These products have high molecular weight partially due to the lignin degraded products and it is hard to be detected by GC and GC-MS (Zhang et al., 2007).

### 3.4. Gas chromatography and mass spectrometry (GC-MS) of liquid products

From the GPC results, many small molecular weight compounds which are less than 970  $\text{g mol}^{-1}$  can be observed after the liquefaction of bamboo. GC-MS was used to obtain more detailed information of the liquefied products with lower boiling points.

All of the components found by GC-MS can be divided into aromatic and non-aromatic compounds. Despite the different solvents used, several components with the same compositions were obtained, including benzoic acid methyl ester ( $RT = 7.366$ ), 1,4-Benzenedicarboxylic acid, dimethyl ester ( $RT = 13.27$ ) and 1,3-Benzenedicarboxylic acid, dimethyl ester ( $RT = 13.426$ ). These results indicate that resemblances in the liquefaction mechanism exist among the liquefaction processes although different kinds of chemicals are used as solvents.

The aromatic compounds undoubtedly originated from the deposition of lignin, in which the benzoyl unit was the most common resultant, such as benzoic acid methyl ester, 1,3-benzenedicarboxylic acid dimethyl ester, benzenepropanoic acid, 3,5-bis(1,1-dimethylethyl)-4-hydroxy-, methyl ester, which were decomposed from the basic unit of lignin-phenyl propane. During the liquefaction process of phenyl propane, the  $\beta$ -aryl was broken and a new benzylether bond was produced (Karagöz et al., 2005). The reactions between these resultants finally led to the formation of phenolic compounds. Furthermore, the phenolic compounds were not stable and decomposed to form benzendiol derivatives (Karagöz et al., 2004).

Meanwhile, the non-aromatic compounds can be separated into two parts according to the formation mechanism. One part consists of propanoic acid and its derivatives that contribute to further decomposition of the furan derivatives (Wang et al., 2008a,b), such as 2-furancarboxaldehyde and 5-methyl-2-furan-carboxaldehyde, which originate from the decomposition of cellulose (Karagöz et al., 2005).

The other part is composed of a long straight-chain (carbon number arranged from C<sub>10</sub> to C<sub>20</sub>) consisting of alkane, ester, alco-

hol and aldehyde. The components are different from the results of other studies by acid-catalyst liquefaction (Zhang et al., 2007), but similar to those reacting at higher temperatures and pressures (Wang et al., 2008a,b; Yuan et al., 2007). This result might be attributed to the use of the autoclave as a reactor, which can provide a higher pressure that improves pyrolysis to form long straight-chain compounds.

**Table A1**

The components and relative content of compounds liquefied from phenol.

Pk	RT	Refs.
1	5.299	Phenol
2	5.501	Carbamic acid, phenyl ester
3	5.595	Dodecane, 4,9-dipropyl-
4	5.68	Dodecane, 4,9-dipropyl-
5	5.737	Dibenzazepine, 10,11(5H)-dihydro-10-hydroxy-
6	5.807	Octadecane, 3-methyl-
7	6.052	Thiazole, 4,5-dimethyl-
8	6.156	3',4'-Difluoroacetophenone
9	6.561	Benzo thiophene-3-carboxylic acid,4,5,6,7-tetrahydro-2-amino-6-ethyl-, ethyl ester
10	6.81	1,1'-Biphenyl, 3,4-diethyl-
11	7.116	Styrene, 2-nitro-3'-[4-methylphenoxy]-
12	7.366	Benzoic acid, methyl ester
13	8.133	3,7-Dithiocyanophenothiazine
14	8.92	Ethanol, 2-(2-butoxyethoxy)-
15	8.995	7-Oxabicyclo[2.2.1]hept-2-ene-2,3- dicarboxylic acid, dimethyl ester
16	9.937	Benzoic acid, 3-chloro-, methyl ester
17	11.439	Benzoic acid, 4-cyano-
18	12.051	Diphenyl ether
19	13.27	1,4-Benzenedicarboxylic acid, dimethyl ester
20	13.426	1,3-Benzenedicarboxylic acid, dimethyl ester
21	13.496	Dodecanoic acid, methyl ester
22	13.967	1,7-Octadiene, 2-methyl-6-methylene-
23	15.813	Methyl tetradecanoate
24	16.882	Cyclopentanedicanoic acid, methyl ester
25	17.904	Hexadecanoic acid, methyl ester
26	18.149	Benzenepropanoic acid, 3,5-bis(1,1-dimethylethyl)-4-hydroxy-, methylester
27	19.811	Octadecanoic acid, methyl ester

**Table A2**

The components and relative content of compounds liquefied from EC.

Pk	RT	Refs.
1	5.501	Pentanedinitrile, 2-methylene-
2	5.595	4-Azatricyclodecane-3,5-dione, 4-phenyl-
3	5.675	S-Methyl n-propylphosphonamidothioate
4	5.737	Ethyl chloride
5	5.812	Propanoic acid, 2-chloro
6	6.556	2-Ethylacridine
7	6.622	2-Ethylacridine
8	6.806	2-Bromo-4,6-dimethylbenzamide
9	7.116	Isloxazolo[4,3-a]phenazine, 1-phenyl-
10	7.366	Benzoic acid, methyl ester
11	7.422	Benzoic acid, methyl ester
12	8.129	6-Oxo-3-methoxy-N-methyl-4,5,7,8-diepoxy morphine
13	8.92	Ethylene glycol diglycidyl ether
14	8.99	7-Oxabicyclo[2.2.1]hept-2-ene-2,3- dicarboxylic acid, dimethyl ester
15	10.681	2-Propen-1-ol, 3-phenyl-
16	11.778	2-Propenoic acid, 3-phenyl-, methyl ester
17	13.271	1,4-Benzenedicarboxylic acid, dimethyl ester
18	13.374	Phenol, 2,4-bis(1,1-dimethylethyl)
19	13.426	1,3-Benzenedicarboxylic acid, dimethyl ester
20	13.492	Dodecanoic acid, methyl ester
21	15.813	Decanoic acid, methyl ester
22	17.904	Hexadecanoic acid, methyl ester
23	18.149	Benzenepropanoic acid,3,5-bis(1,1-dimethylethyl)-4-hydroxy-,methylester
24	19.806	Octadecanoic acid, methyl ester
25	22.127	1,2-Benzenediol, 3,5-bis(1,1-dimethylethyl)-
26	24.651	2-(Acetoxymethyl)-3-(methoxycarbonyl)biphenylene



**Table A3**

The components and relative content of compounds liquefied from EG.

Pk	RT	Refs.
1	5.506	1-(3-Hydroxy-4-methylphenyl)-1,3,3,6-tetramethylindan-5-ol
2	5.591	4,9-dipropyl-Dodecane
3	5.68	Decane
4	5.732	Eicosane
5	5.812	3-methyl-Octadecane
6	6.622	Oxalic acid
7	6.806	2,5-dimethyl-4'-nitro-1,1'-Biphenyl
8	7.112	5-methyl-2-Heptano
9	7.366	Benzoic acid, methyl ester
10	9.362	Indeno[2,1-c]pyridine, 3,7-dimethyl-6-nitro-9-phenylimino-
11	9.504	2,4(1H,3H)-Pyrimidinedione, dihydro-5,6-dihydroxy-5-methyl-
12	11.773	2-Propenoic acid, 3-phenyl-, methyl ester
13	11.834	2-Nonanol
14	11.929	1,2-Benzenedicarboxylic acid, 2-ethoxy-2-oxoethyl methyl ester
15	12.051	Diphenyl ether
16	12.706	Dimethyl phthalate
17	13.271	1,4-Benzenedicarboxylic acid, dimethyl ester
18	13.369	Phenol, 3,5-bis(1,1-dimethylethyl)
19	13.426	1,3-Benzenedicarboxylic acid, dimethyl ester
20	13.497	Dodecanoic acid, methyl ester
21	13.572	Benzoic acid, 4-ethoxy-, ethyl ester
22	13.967	Pyrido[2,3-d]pyrimidine, 4-phenyl-
23	14.683	1H-Indole, 5-methyl-2-phenyl-
24	15.813	Methyl tetradecanoate
25	17.692	Hexadecanal, 2-methyl-
26	17.904	Hexadecanoic acid, methyl ester
27	18.149	Benzenepropanoic acid, 3,5-bis-(dimethylethyl)-4-hydroxy-, methylester
28	19.806	Octadecanoic acid, methyl ester

#### 4. Conclusions

In the present paper, we have systematically reported how organic solvents (phenol, EG and EC), the liquid ratios and the treatment times affect the bamboo LYs and the liquefied products. It is found that phenol is the best solvent for bamboo liquefaction. The FT-IR analyses of the residues show that the major peaks of bamboo diminish when using phenol and EC as the solvents while ketone, ester and benzene derivations are produced in the process of liquefaction with EG. The GPC results show that after liquefaction, the high molecular weight products of bamboo decrease significantly to around 1800 g mol<sup>-1</sup>. The GC-MS analysis shows that low boiling point products of liquefied bamboo are similar regardless of the type of solvent used.

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#### Annex

See Tables A1–A3.

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