Influence of soaking temperature time on the ability prepared liquefaction of wood from cashew nut shell waste

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ABSTRACT

Liquefied wood is one of the phenolic resin. However, unlike commercial phenolic resins that are normally synthesized by the chemical reaction between phenol and formaldehyde, liquefied wood is usually produced by reacting phenol with wood-derived materials, and catalyst at 120-180°C. Depending on whether the catalyst is a base or a acid, the formed resin is a thermoset or a thermoplastic. In this study, wood liquefaction was prepared from a cashew nut shell waste (CNSW), phenol, and sulfuric acid catalyst. The cashew nut shell waste is taken from Binh Phuoc province -Vietnam and crushed to a size of less than 500 μ m. Phenol and sulfuric acid catalyst are chemical experiments. The powder of cashew nut shell waste, phenol, and sulfuric acid were mixed and reacted at 150°C for different soaking times. An optimal soaking temperature time was determined through a cashew nut shell waste residue content in wood liquefaction products. The wood liquefaction products also were determined by a number average molecular weight (M_n) and a weight average molecular weight (M_w) by Gel permeation chromatography method (GPC); the function groups by Fourier Transform Infrared method (FT-IR). The results showed that the formed resin is thermoplastic and the optimal soaking time to prepared liquefied wood is 180 minutes. This sample has a residual cashew nut shell waste ratio of 9.44%, a number average molecular weight of 7552, and a weight average molecular weight of 10640. The liquefied wood from cashew nut shell waste can be used as a binder in the manufacture of the medium density fiberboard (MDF) or as a material to promote the sintered process in the production of woodceramic materials. In addition, the liquefied wood can also be pyrolyzed to form carbon fiber. Carbon fiber can be applied as reinforcing materials for ceramic products.

Key words: phenolic resin, liquefied wood, cashew nut shell waste

INTRODUCTION

Phenolic resin is a resin synthesized from the phenol and formaldehyde. It was commercialized and was put into industrial production in the early 20th century. Phenolic resin has good thermal stability, capable of binding and high carbon content. When decomposed at high temperatures, the phenolic resin can transform into carbon fiber structures ¹. Carbon fiber can be applied as reinforcement materials ^{1,2}.

There are many methods for synthetic phenolic resin. Liquefied wood is one of the methods that have been studied ³. By this method, the wood will be transformed into a high-molecular material with a strong odor and dark black color by giving the phenol or multi-functional alcohol to react with a catalyst. This transformed process is complicated and has many products to form.

There have been many studies to fabricate wood lique-faction such as: using wood to react with polyethylene glycol/ glycerol/ polyhydric alcohol, and catalytic sulfuric acid at 150° C to create wood liquefaction $^{4-6}$. In

addition, ethylene glycol, sulfuric acid, and wood are also combined to make wood liquefaction ^{7–9}. Several studies have also used phenol and acid catalysts such as sulfuric acid, phosphoric acid to make wood liquefaction from the wood pulp at 120 - 180°C ^{10,11}. Regarding wood materials for fabricating wood liquefaction, studies often use agricultural waste to fabricate wood liquefaction. Types of agricultural waste can be mentioned as the fruit of oil palm ¹², the wood of Japanese cedar ¹³, waste paper ¹⁴, bagasse ¹⁵, corn stover ¹⁶.

It can be seen that these research directions consider wood liquefaction made from wood-based raw materials as renewable resources to replace fossiloriginated plastic. It contributes to addressing concerns about fossil fuel exhaustion issues and environmental pollution. In agriculturally developed countries like Vietnam, this research direction is even more important in the use of industrial waste products to make wood liquefaction into a product of economic value.

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In Vietnam, cashew is one of the important industrial trees. Due to exporting cashew nuts only, cashew nut shell has become one of the waste items in the cashew industry. Cashew nut shell is often pressed to get cashew oil. However, the cashew nut shell after pressing (often called the cashew nut shell waste) is still a waste to be treated. There have been some studies using cashew shell waste to fabricate ceramic ^{17,18}. Other studies have also used cashew shell waste to make wood liquefaction ^{19,20}. This research will study the influence of soaking temperature time on the ability to prepared wood liquefaction from cashew nut shell waste.

EXPERIMENTAL METHODS

Raw materials

CNSW was taken from Binh Phuoc. Chemical composition was shown in Table 1 and Table 2. CNSW was washed and crushed to a size of less than 500 μ m. Phenol (1.07 g/cm³, Merck) and 98% sulfuric acid (1.84 g/cm³, Merck) were also used to fabricate wood liquefaction from CNSW.

Phenol, CNSW were mixed with the ratio of 2/1 (wt.) and 5% sulfuric acid According to the amount of phenol used. The mixed mixtures were placed in the drying oven and soaked at 150 °C for different time periods to make wood liquefaction. The soaking temperature times are shown in Table 3.

Optimum soaking time was determined by analyzing properties such as vibrational units of functional groups by FT-IR method; Wood liquefaction reaction efficiency through assessment of residual CNSW ratio; The number average molecular weight (M_n) and the weight average molecular weight (M_w) by Gel chromatography (GPC) method.

Methods

Fourier-transform infrared spectroscopy (FT-IR):

The vibration of the functional groups of wood liquefaction was analyzed by Fourier-transform infrared spectroscopy (FT-IR – Model Nicolet 6700, Thermo, USA). The scanning step was 0.9642 with a scanning angle of 500 to 4000cm^{-1} . The binder was KBr. The test method was a transmittance spectrum.

Determine a residual CNSW:

Phenolic resin is crushed, filtered, and washed to remove residual phenol in the resin. The washed resin is dried at 70° C. The sample was then weighed 2,00 \pm 0.01 gram, contained in Erlen, added about 40 ml of ethanol, and swirled well. The sample is filtered

slowly through the filter paper and weighed to determine the mass of the filtered sample. After filtration is complete, the filter paper containing the residue is placed in a drying cup and dried to constant weight. The residual CNSW is determined from the following formula:

$$\%m_{gd} = \frac{m_s - m_{gl}}{m_o} \tag{1}$$

m_{ed} - the percentage of residual CNSW (%)

 m_s – the weight of filter paper and insoluble residue (gram)

m_{gl} - the weight of dry filter paper (gram)

m_o – the weight of the original phenolic resin (gram)

Gel permeation chromatography (GPC):

The number average molecular weight (M_n) and the weight average molecular weight (M_w) were determined by Gel permeation chromatography method (GPC - Model PL-GPC 50, POLYMERLAB, USA). The used soluble solvent is Dimethyl - Formamide (DMF). The wood liquefaction samples were dissolved in DMF solvent at a concentration of 0.10 mg/ml. The dosage for each sample injection was 50μ l.

THE RESULTS AND DISCUSSIONS

Vibrational units of functional groups by FT-IR spectrum

Wood liquefaction samples at different soaking times were determined functional groups by FT-IR method. FT-IR spectrums of samples were presented in Figure 1. FT-IR spectrum showed that the samples all had functional groups such as: CH_n (2929, 2850 cm⁻¹), C=O (1700cm⁻¹), C=C (1598 cm⁻¹), C-C (1513 cm⁻¹), CH₂ (1452 cm⁻¹), CH (1371 cm⁻¹), OH (1257 cm⁻¹), C-H (1092, 815, 754 cm⁻¹) (Table 4) 2,3,21,22 .

The results of the FTIR spectrum of the samples were similar. Comparison with the FTIR spectrum of previous studies showed that there was wood liquefaction formation in all samples ^{2,3,21,22}. In this case, it is difficult to estimate the optimal retention time for wood liquefaction generation at 150°C. Therefore, the determination of residual CNSW and the GPC method was used to evaluate this factor.

Determine residual CNSW

Results from the graph in Figure 2 showed that the residual CNSW of liquefied wood decreased as the soaking time increases. When the retention time increased, the reaction time for creating wood liquefaction was prolonged, the reaction performance also increased. The residue of CNSW was reduced rapidly

Table 1: The composition of the chemical elements (% wt.) of CNSW 20

	<u> </u>					
Sample	С	N	Н	О	Others	
CSNW	53.6	9.5	8.8	26.8	1.2	

Table 2: The composition of chemical compounds (% wt.) of CNSW

Sample	Cellulose	Hemi cellulose	Lignin	Anacardic acid	Cardanol	Cardol	2-Methyl Cardol
CSNW	53-56	11-12	16-17	10-15	1-2	2-4	<1

Table 3: The soaking temperature times

Sample		Ratio (wt.)			Soaking time
	Phenol	CNSW	Sulfuric acid(*)	(°C)	(minutes)
ST90	2	1	5%	150	90
ST120	2	1		150	120
ST150	2	1		150	150
ST180	2	1		150	180
ST210	2	1		150	210

^{*} According to the amount of phenol used

Table 4: Vibration of functional groups

Wavenumbers (cm ⁻¹)	Function groups	Ref.
3351	Vibrate of OH group of phenolic and methylol	21
2929, 2850	Vibrate of CH2 group of aliphatic	2,21
1700	Vibrate of C=O group of carboxylic	3
1598	Vibrate of C=C group of benzen	22
1513	Vibrate of C-C in aromatic ring	3
1452	Vibrate of -CH2 group	1
1371	Vibrate of -CH group	21
1257	Vibrate of OH group	1
1092	Vibrate of C-H group inside the aromatic ring	1,21
815	Vibrate of C-H group of benzen	1
754	Vibrate of C-H group outside the aromatic plane	1

when extending the retention time from 90 to 180 minutes corresponding to the model ST90 to ST180 (from 29.68% to 9.44%). If the soaking time was prolonged, the residue of CNSW would be difficult to reduce further (from 9.44% to 9.02%).

When the heat retention time was prolonged, the chemical reactions between phenol, CNSW, and the acid catalyst would take place more thoroughly. As a result of this process, the amount of CNSW involved in the reaction to create the resin was more, the amount of CNSW remaining after the reaction

also was less. However, besides the resin-forming reaction, there was also the thermal decomposition of CNSW 23 . Therefore, when the heat retention time was longer than 180 minutes, the result of CNSW residue decreases very little. The CNSW residue result showed that at 150^o C the optimal soaking time was 180 minutes.

Determine \mathbf{M}_n and \mathbf{M}_w by GPC

The results of the GPC analysis of the samples were presented in Table 5 and Figure 3. According to the

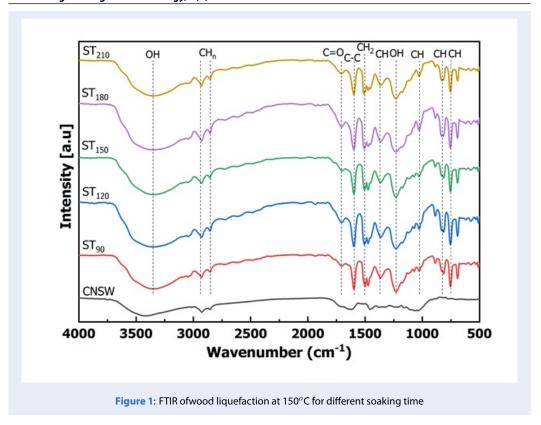


Table 5: Effect of soaking time to residual CNSW ratio

Sample	Temperature (°C)	Soaking time (minute)	Residual CNSW ratio (% .wt)
ST90	150	90	29.68 ± 1.45
ST120	150	120	15.25 ± 1.36
ST150	150	150	11.95 ± 0.91
ST180	150	180	9.44 ± 1.15
ST210	150	210	9.02 ± 1.24

data in Table 5 and Figure 3, the samples all had the presence of two characteristic distribution areas. The first region (region 1) had the number average molecular weight (M_n) in the range of 6997 - 7552 and the weight average molecular weight (M_w) in the range 10152 - 10849. The second region (region 2) had M_n index in the range 1275 - 1325 and M_w in the range of 1545 – 1755. This region was the distribution of oligomers.

Kensuke Naka pointed out that polymer had a molecular weight of more than ten thousand and oligomer had a molecular weight of several thousand or less 23 . GPC results showed that region 1 with M_w value over 10000 was the distribution region of the polymer and region 2 with M_w value from 1545 to 1755 was the distribution region of the oligomer. The W_n was used

to determine the formation of the liquefied wood at different thermal retention time. When holding the heat at 150^{o} C from 90 minutes to 180 minutes (ST90 – ST180 samples), the oligomers linked together to form polymers. The W_n value of the polymer region would increase and the oligomer region would decrease. Conversely, if the holding time was too long (ST210 sample), the W_n value of the polymer region would decrease and the oligomer region would increase. This result demonstrated that prolonging the soaking time also would sever the molecular circuit of the newly formed polymer.

This result combined with the residual CNSW ratio (Figure 1) and the relative intensity ratio of the peaks on the FT-IR spectra (Figure 2) showed that the formed wood liquefaction at different soaking times

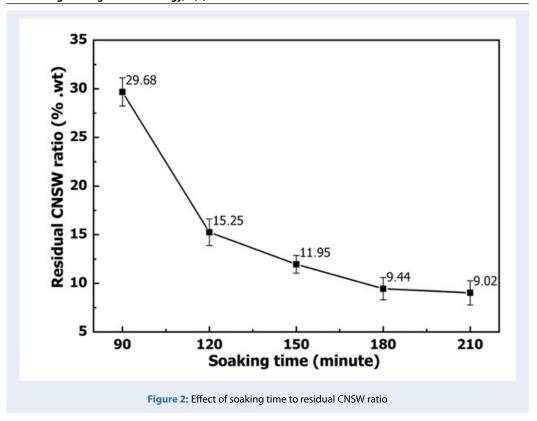


Table 6: Gel permeation chromatography of wood liquefaction samples

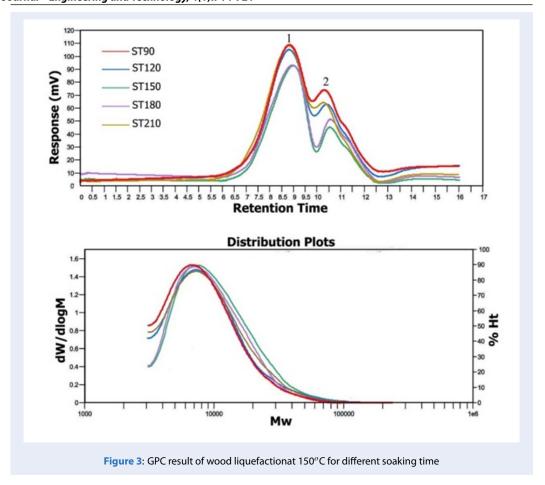
Sample	Soaking time (minutes)	Region	M_n	M_w
ST ₉₀	90	1	7056	10152
		2	1325	1679
ST ₁₂₀	120	1	7141	10845
		2	1295	1610
ST ₁₅₀	150	1	7410	10401
		2	1282	1755
ST ₁₈₀	180	1	7552	10640
		2	1275	1545
ST ₂₁₀	210	1	6997	10509
		2	1323	1629

had a similar structure and 180 minutes was the optimal time to fabricate wood liquefaction from CNSW at 150^{o} C.

CONCLUSIONS

In this study, the effect of soaking time at 150^{o} C on the ability to prepared wood liquefaction was investigated. The FTIR results showed that there was LW formation in all samples at different soaking

times. When increasing the soaking time, the residual CNSW ratio in resin decreased. However, when increasing the retention time of more than 180 minutes, the residual CNSW ratio did not decrease significantly. M_w and M_n values in the GPC results showed that the samples had coexistence of the polymer and oligomer and 180 minutes was the optimal time to fabricate wood liquefaction from CNSW at 150^{o} C. The ST180 sample had the residual CNSW ratio of



9.44%, the number average molecular weight (M_n) of 7552, and the weight average molecular weight (M_w) of 10640.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interest regarding the publication of this article.

AUTHOR'S CONTRIBUTION

The authors confirm contribution to the paper as follows: study conception and design: Do Quang Minh, Kieu Do Trung Kien; data collection: Nguyen Vu Uyen Nhi; analysis and interpretation of results: Huynh Ngoc Minh; draft manuscript preparation:

Kieu Do Trung Kien. All authors reviewed the results and approved the final version of the manuscript.

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Ảnh hưởng của thời gian lưu nhiệt đến khả năng tạo gỗ hoá nhựa từ nguồn nguyên liệu bã thải vỏ điều

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TÓM TẮT

Gỗ hoá nhưa là một trong những sản phẩm nhưa phenolic. Tuy nhiên, không giống như các loại nhựa phenolic thương phẩm thường được tổng hợp từ phản ứng hoá học giữa phenol và formaldehyde, gỗ hoá nhựa thường được tổng hợp từ phản ứng giữa phenol với một loại nguyên liệu gốc gỗ và xúc tác tại nhiệt đô 120 – 180°C. Tuỳ thuộc vào xúc tác sử dụng là bazơ hoặc axít, nhưa tạo thành có thể là nhựa nhiệt rắn hoặc nhiệt dẻo. Trong nghiên cứu này, gỗ hoá nhựa được tổng hợp từ bã thải vỏ điều, phenol và xúc tác axít sulphuric. Bã thải vỏ điều được lấy từ tỉnh Bình Phước – Việt Nam và được nghiên đến kích thước hạt nhỏ hơn 500 μ m. Phenol và xúc tác axít sulphuric sử dụng là các hoá chất thí nghiệm. Bột bã thải vỏ điều, phenol và axít sulphuric được trộn với nhau và được cho phản ứng ở 150°C trong những thời gian lưu nhiệt khác nhau. Thời gian lưu nhiệt thích hợp được xác định thông qua dư lượng bã thải vỏ điều còn lại sau phản ứng tạo nhựa. Ngoài ra, sản phẩm gỗ hoá nhựa cũng được các định số lượng phân tử số trung bình (M_n) và phân tử khối trung bình (M_w) bằng phương pháp sắc ký thẩm thấu gel (GPC). Nhóm chức tạo thành được xác định bằng phổ biến đổi hồng ngoại (FTIR). Kết quả chỉ ra rằng nhựa tạo thành là nhựa nhiệt dẻo và thời gian lưu nhiệt thích hợp để tạo gỗ hoá nhựa là 180 phút. Mẫu nhựa này có dư lượng bã thải vỏ điều còn lại sau phản ứng là 9.44%, phân tử số trung bình là 7552, phân tử khối trung bình là 10640. Gỗ hóa nhựa từ bã thải vỏ điều có thể được sử dụng làm chất liên kết trong sản xuất tấm ván gỗ ép (MDF) hoặc làm nguyên liệu thúc đẩy quá trình kết khối trong sản xuất vật liệu gốm gỗ. Ngoài ra, gỗ hoá nhựa cũng có thể được nhiệt phân để tạo thành sợi cacbon. Sợi cacbon có thể được ứng dụng làm vật liệu gia cường trong sản xuất một số loại gốm.

Từ khoá: nhựa phenolic, gỗ hoá nhựa, bã thải vỏ điều

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