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Research Article

Effect of mixing phenol/rice husk to the ability form phenolic resin

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ABSTRACT

Rice is an essential plant that brings high economic value to Viet Nam. In addition to the main product, rice agriculture produces a significant amount of by-products each year. This waste is known as rice husk. The amount of rice husks discharged each year is estimated to be over 9 million tons. They are usually processed by burning. Another amount is released into the environment. These treatments are potentially harmful to the environment and bring low economic efficiency. Therefore, many ways of treatment were suggested and considered. In this study, rice husk was used to synthesize phenolic resin. The rice husk was ground to powder less than 500μm. The husk powder, phenol, and sulfuric acid were mixed with different ratios. The mixtures were heated to 150°C and soaked for 180 minutes. The efficiency of the reaction was evaluated through the remaining amount of rice husk. The C^{13} Nuclear Magnetic Resonance Spectroscopy (C^{13} - NMR) and Fourier Transform Infrared Spectroscopy (FTIR) were used to determine the structure and functional groups of the resin. A number average molecular weight (M_n) and a weight average molecular weight (M_w) were defined by using Gel Permeation Chromatography (GPC). The results show that phenolic resin was synthesized successfully from rice husk. The optimal ratio between phenol and rice husk powder is 2:1 with a 5% sulfuric acid additive. The efficiency of the reaction is determined at 90.34%. The results of structure prediction by FTIR and C^{13} - NMR show that the molecular formula of the formed resin is $(CH_2 - C_6H_4 - OH)_n$. It has the weight average molecular weight value of 11709 and the number average molecular weight of 7782. The synthesized phenolic resin can be applied as a binder or as one kind of heat-resistant plastic.

Key words: phenolic resin, liquefied wood, rice husk

INTRODUCTION

Phenolic resin is known as an important plastic that is widely produced and applied. Almost industrial phenolic resins are synthesized from phenol and formaldehyde. The synthesized resin can be categorized into two resins: thermoset and thermoplastic, depending on the additives used. With a base catalyst, M. Asim et al. has shown that the formed product is a thermoset resin after the reaction[1]. In contrast, with an acid catalyst, R. Zhou et al. produced a thermoplastic product[2]. Phenolic resin can maintain a stable structure despite being at high temperatures. Therefore, phenolic resin is currently consumed in large quantities and commonly used in several fields, such as heat-resistant materials, friction-resistant materials, adhesives, and the molding industry.

Due to the importance of phenolic resins, many research studies have been suggested to optimize the process and the efficiency of the phenolic resin production process[3]. On the other hand, finding alternative sources of raw materials for the production process of this plastic is suggested. And liquefied wood is one of the methods considered to be used. This method uses biomass materials to make phenolic resins[4]. Therefore, liquefied wood can also be applied to treat agricultural waste[5].

Many groups of authors have published their studies about synthesizing phenolic resins from different agricultural waste sources. H.M. Alma et al. prepared phenolic resins from pine and chestnut bark by mixing them with phenol and sulfuric acid and reacting at 130°C for 1 hour[6]. A. Ahmadzadeh et al. dissolved oil palm fruit with phenol at 105°C for 8 hours to produce phenolic resin[7]. S. H. Lee et al. also synthesized phenolic resin at 200°C in a pressure environment above 1atm from corn bran[8]. Moreover, phenolic resin has also been synthesized from other agricultural wastes such as waste paper[9], field corn plants[10], cashew shell waste[11], etc. Following all the above studies, it can be seen that Vietnam is one of the agricultural-producing countries; the liquefied wood method can effectively treat agricultural waste and valorize economic value for the farming industry. This study used rice husks, a by-product of rice production, to make phenolic resins by plasticized plant-

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based biomass method. In Vietnam, rice agriculture is one of the most important production industries, and every year this production field also generates a considerable amount of rice husk. It is known as rice husk and is considered a waste in this business. Many ways of treatment were suggested and considered. Rice husk is frequently treated by burning, which pollutes the environment and has low economic value. Therefore, synthesizing phenolic resin from rice husk can be an effective way to treat waste husk as well as improve the monetary value of the rice production.

EXPERIMENTAL METHODS

Raw materials

RH was taken from Long An Province. The chemical composition of RH was determined by XRF and IRMS methods. Table 1 shows the chemical composition. RH was washed and milled to a size of less than 500 μm. Phenol (1.07 g/cm^3) and 98% ulfuric acid (1.84 g/cm^3) were also used to make wood liquefaction from RH. The chemical composition of RH was determined by X-Ray Fluorescence (XRF) and Isotope Ratio Mass Spectrometry (IRMS). Table 1 shows the chemical composition of RH.

RH, phenol, and sulfuric acid were mixed in different ratios and reacted at 150°C in the electrical furnace, soaked at 180 minutes to make phenolic resin. Different ratios of the raw materials are shown in Table 2.

Methods

The optimized ratio was determined by analyzing properties such as the efficiency of the synthesized process through the remaining quantity of husk after reaction; FTIR was used to define the presence of organic and inorganic compounds in the synthesized; The Number Average Molecular weight (Mn) and the weight average molecular weight (Mw) by GPC analysis.

The efficiency of the resin-synthesized reaction:

The efficiency of the reaction is defined through the remaining husks, and the experimental procedure has two steps. Firstly, determine the amount of RH remaining in the sample by dissolving synthesized resins in ethanol (Only resins were dissolved in ethanol, whereas RH remained unreacted). The mixing ratio was 2 grams of resin/40 ml of ethanol. The mixture was stirred for 30 mins. The insoluble mixtures were filtered and washed carefully then the percentage of the non-reacted RH was defined by the formula (1). In step two, the efficiency was determined by the formula (2). The result was the average of three measurements.

$$\%RH = \frac{m_{rh}}{m_{pr}}.100\% \tag{1}$$

$$\%H = 100\% - \%RH \tag{2}$$

Where,

m_{rh} – the amount of RH remaining in the sample (gram),

m_{pr} – the amount of phenolic resin (gram),

RH(%) -the percentage of RH remaining in the sample (%),

H - the efficiency (%).

The functional groups and a molecular formula of synthesized resins:

The functional groups which exist in the samples were determined by the FTIR technique. The wavelength range used in this study is from 500 to 4000 cm^{-1}, and the step is 0.9642.

^{13}C-NMR was used to define the molecular formula of synthesized wood liquefaction samples. The solvent used in the experiment is Methanol (MeOD) (100 -150mg of each synthesized resin will be dissolved in 0.6ml solvent)

The Number Average Molecular weight (M_n) and the weight average molecular weight (M_w):

The Number Average Molecular weight (M_n) and the weight average molecular weight (M_w) were determined by Gel Permeation Chromatography. The soluble used solvent is Dimethyl - Formamide (DMF). The wood liquefaction samples were dissolved in DMF solvent at 0,10 mg/ml. The dosage for each pump is 50μl.

THE RESULTS AND DISCUSSIONS

Figure 1 shows wood liquefaction samples after being synthesized from different ratios of raw ingredients. At first sight, the PN1 sample had a solid form without the typical gloss of regular plastic. The sample was porous, and the structure seemed like separate particles and heterogeneous. Sample PN1.5 was still in solid form, not glossy and porous. Sample PN2 had a solid form. However, unlike the two past samples, the PN2 sample was in a block shape and had the typical gloss-like plastic products. Sample PN2.5 was a flexible block with a standard gloss that seemed plastic. Sample PN3 seemed liquid and glossy. According to the above assessment, it is possible to predict that the ratios fitted with samples PN2, PN2.5, and PN3

Table 1: **Chemical composition (% wt.) of RH** [12]

Sampl	C	N	H	Si	Fe	Ca	Na	K	Al			Mn	Others
Rice husk	45.91	3.12	6.12	8.38	0.38	0.19	0.09	0.18	0.09	0.11	0.01	0.02	35.4

Table 2: **Different ratios (%wt.) of the ingredients**

Sample	Ratio (%wt.)			Temperature	Soaking time
	Phenol	RH	Sulfuric acid*	(oC)	(mins)
PN1	1	1	5%	150	180
PN1.5	1.5	1		150	180
PN2	2	1		150	180
PN2.5	2.5	1		150	180
PN3	3	1		150	180

* According to the amount of phenol used

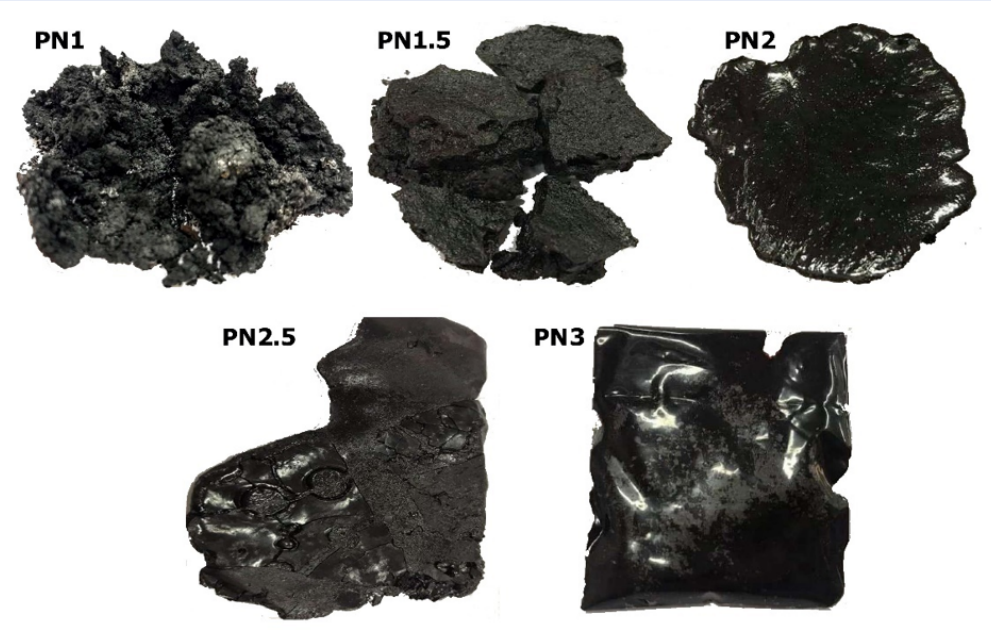

Figure 1: Synthesized phenolic resins from PN samples

for better plastic-forming ability than the remaining samples.

To more precisely determine the ability to synthesize resins of sample mixtures, the reaction efficiency was calculated by dissolving the resin with ethanol solution. Figure 2 are the results of calculating the efficiency from formulas (1) and (2).

As shown in Figure 2, the resin-forming ability increases with the amount of phenol. The reaction efficiency increased sharply when the phenol/RH ratios rose from 1/1 to 2/1. When the amount of phenol increased, the efficiency increased from 67.22% to 90.34%. However, if we keep increasing the phenol content in the mixture (with samples PN2.5 and PN3), the reaction efficiency does not change significantly (91.33% with sample PN2.5 and 94.08% with sample PN3). Compared with the observed shape of the synthesized, the PN2.5 and PN3 samples also have a plastic and liquid state. This case has also been mentioned in previous studies. When the amount of phenol is excessively used, the non-reacted phenol will reduce the phenolic resin's viscosity [13]. The error bar of each

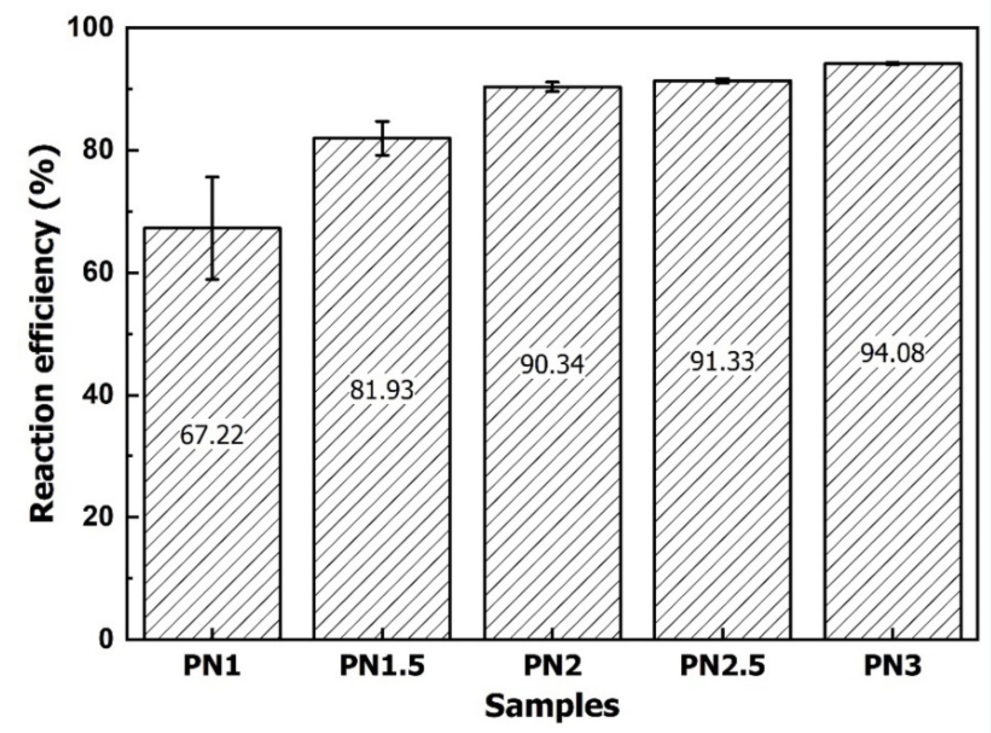

Figure 2: Reaction efficiency changed depending on different samples

sample in Figure 2 also shows that the less amount of phenol used, the higher the uncertainty of the formula is. The change in the error bar can be explained that when the amount of phenol is low, it is not dispersed evenly in the RH solid phase, leading to an unstable resin reaction. Therefore, the results of determining the reaction efficiency at different times will vary greatly. From figures of actual samples and efficiency of reaction, it is shown that the composition ratio of sample PN2 is the optimal composition to create phenolic resin from RH under experimental conditions. Moreover, if we keep increasing the amount of phenol used, it not only cost much but also not improve the efficiency considerably. On the other hand, increasing the phenol used will result in a large amount of residual phenol in the product. Therefore, sample PN2 will be selected for structural analysis through FTIR, NMR and GPC methods.

The PN2 resin sample was analyzed by Gel permeation chromatography (GPC) to determine the number average molecular weight (M_n) and the weight average molecular weight (M_w). The results of the GPC analysis are shown in Table 3. Table 3 illustrates the M_n and M_w values of the PN2 sample. GPC analysis results show that the sample has two distinct regions. Region 1 has the number average molecular weight of 7782 and the weight average molecular weight of 11709. Region 2 has the number average molecular weight of 1334 and the weight average molecular weight of 1660. The values indicate that region 1 has a high molecular mass, which is typical in polymer formation. In contrast, the molecular mass defined in region 2 is smaller, representing oligomers' formation[14]. The GPC results again demonstrated that phenolic resin was formed from RH through the wood liquefaction method.

The result of the FTIR analysis is shown in Figure 3. Table 4 illustrates the functional groups present in samples depending on wave number positions. According to the FTIR analysis result, the structure of the phenolic resin corresponding to the sample PN2 appears to have functional groups such as OH (3321, 1230, 1170 cm^{-1}), CHn (3050, 2940 cm^{-1}), C-O (1700 cm^{-1}), C=C (1600 cm^{-1}), C-C (1510 cm^{-1}), CH_2 (1450 cm^{-1}), CH (1350, 1100, 1020, 814, 753, 692 cm^{-1}). These above functional groups are commonly found in the structure of phenolic resins. NMR spectroscopy was used to determine the molecular formula of the synthesized phenolic resin from PN2 compositions.

Figure 4 shows the result of the ^{13}C NMR analysis of the result PN2 resin. The results of the NMR anal-

Table 3: **GPC analysis result**

Sample	Region	M_n	M_w
PN2	1	7782	11709
	2	1334	1660

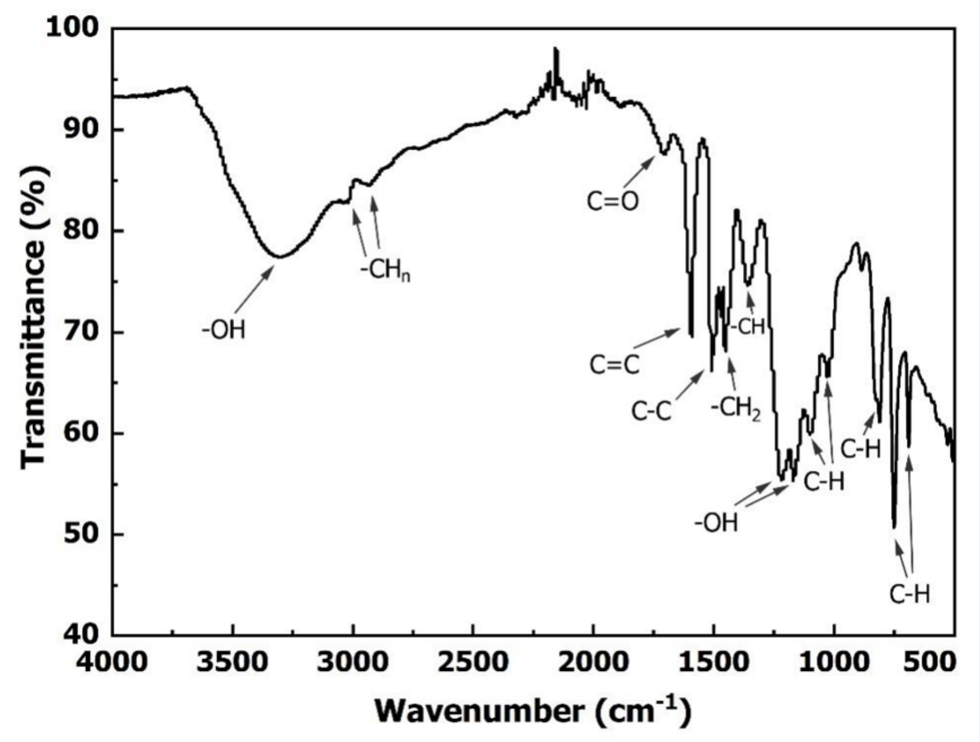

Figure 3: FTIR analysis result of PN2 sample

Table 4: **Functional Groups' Vibration**

Function Groups	Wavenumber (cm^{-1})	Reference
Vibrate of OH group of phenolic and methylol	3321	[15]
Vibrate of CHn group	3050, 2940	[16]
Vibrate of C=O group	1700	[17]
Vibrate of C=C group	1600	[18]
Vibrate of C-C in aromatic ring	1510	[19]
Vibrate of -CH$_2$ group	1450	[20]
Vibrate of -CH group	1350	[15]
Vibrate of OH group	1230, 1170	[20]
Vibrate of C-H group inside the aromatic ring	1100, 1020	[15]
Vibrate of C-H group of benzene	814	[20]
Vibrate of C-H group outside the aromatic plane	753, 692	[18]

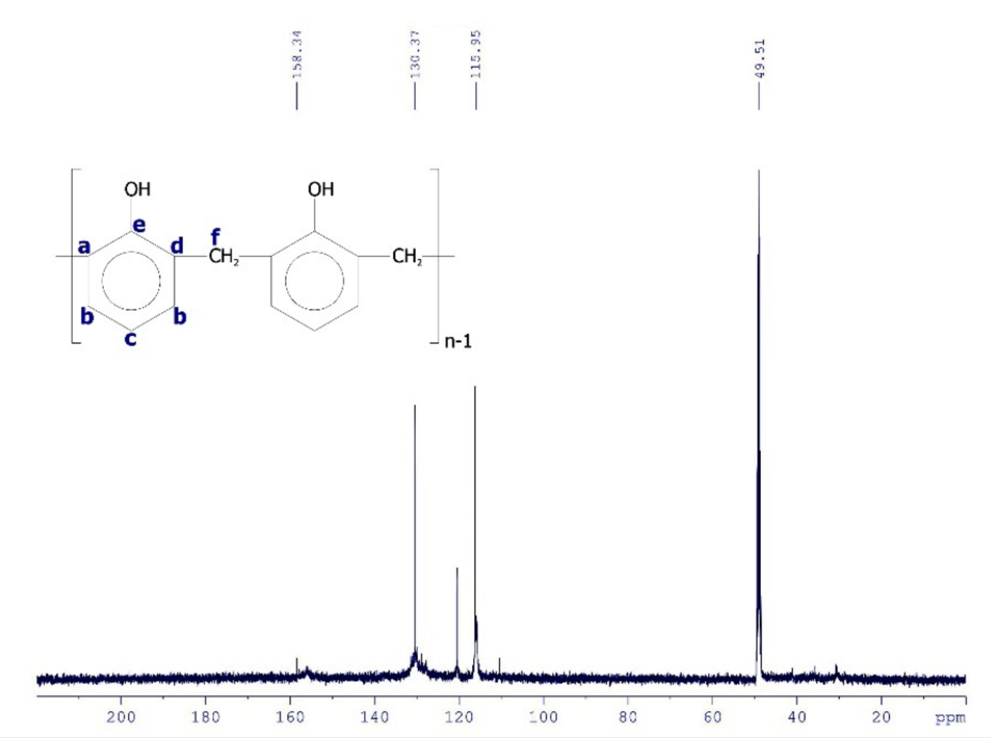

Figure 4: The ^{13}C-NMR analysis results

ysis showed that the characteristic peaks for carbon appeared at different positions of the benzene ring. Specifically, the C_e of the ring is directly attached to the OH radical (158.34ppm), the C_a is adjacent to the C_e position at a fixed peak (130.37 ppm), the C_b was defined at the peak corresponding to 120.48 ppm peak, the C_c is at the para position (115.95 ppm), and C_d is linked to CH_2 (110.35 ppm), C_f of CH_2 attached to the ring at the ortho position (49.51 ppm)[21]. With the above determinations, it can be predicted that the molecular formula of the synthesized phenolic resin is $(CH_2\text{-}C_6H_4\text{-}OH)_n$, as shown in Figure 4.

CONCLUSIONS

In this study, phenolic resin was successfully synthesized from rice husk material through the wood liquefaction method. By determining the synthesized process's efficiency, the phenol/rice husk ratio is 2/1, and 5% acid used as an additive brings an optimal composition to produce plastic at 150°C and then soaked in 180 minutes. With the above ratio composition, the resin-forming reaction efficiency is 90.34%. The resulting sample has a solid form and typical gloss, as seen in almost resins. The results of GPC analysis showed that a polymer structure was formed with the weight average molecular weight value reaching 11709 and the number average molecular weight reaching 7782. Besides that, analysis of functional groups and ^{13}C - NMR result has also shown that the synthesized resin formed as a phenolic resin with the molecular formula $(CH_2\text{-}C_6H_4\text{-}OH)_n$.

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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: Ong Dieu Hanh, Kieu Do Trung Kien; data collection: Nguyen Cam Thuy; analysis and interpretation of results: Nguyen Hoang Thien Khoi; draft manuscript preparation: Le Huynh Tuyet Anh. All authors reviewed the results and approved the final version of the manuscript.

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Bài nghiên cứu

Ảnh hưởng của tỉ lệ phenol/vỏ trấu đến khả năng tạo nhựa phenolic

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TÓM TẮT

Lúa gạo là loại nông sản mang lại giá trị kinh tế cao cho Việt Nam. Bên cạnh sản phẩm chính, nền nông nghiệp trồng lúa cũng thải ra một lượng lớn chất thải hằng năm. Chất thải này là vỏ trấu. Lượng trấu thải mỗi năm ước tính là hơn chín tấn. Chúng thường được xử lý bằng phương pháp đốt. Một lượng khác thì được thải trực tiếp ra môi trường. Những cách xử lý này tiềm ẩn nguy hại đến môi trường và mang lại giá trị kinh tế không cao. Trong nghiên cứu này, vỏ trấu đã được sử dụng để tổng hợp nhựa phenolic. Vỏ trấu đã được nghiền với kích thước hạt nhỏ hơn 500 μm. Bột vỏ trấu, phenol và xúc tác axit sulphuric đã được trộn với các tỉ lệ khác nhau. Các hỗn hợp sau trộn được gia nhiệt ở 150^{o}C và lưu nhiệt trong 180 phút. Khả năng phản ứng tạo nhựa đã được đánh giá thông qua xác định lượng trấu còn lại chưa phản ứng. Phương pháp cộng hưởng từ hạt nhân C^{13} (C^{13} – NMR) và phổ biến đổi hồng ngoại Fourier (FTIR) cũng đã được sử dụng để xác định cấu trúc và nhóm chức của nhựa. Số lượng phân tử trung bình (M_n) và trọng lượng phân tử trung bình (M_w) đã được xác định bằng phương pháp sắc ký thẩm thấu gel (GPC). Kết quả chỉ ra rằng đã tổng hợp được nhựa phenolic từ vỏ trấu với tỉ lệ phenol/trấu là 2/1 và 5% xúc tác axit sulphuric. Hiệu suất phản ứng tạo nhựa là 90.34%. Kết quả dự đoán cấu trúc bằng FTIR và C^{13}-NMR cho thấy cấu trúc phân tử của nhựa hình thành là $(CH_2 - C_6H_4 - OH)_n$. Nhựa có trọng lượng phân tử trung bình là 11709 và số lượng phân tử trung bình là 7782. Nhựa phenolic được tổng hợp có thể ứng dụng như là chất kết dính hoặc một loại nhựa chịu nhiệt.

Từ khoá: nhựa phenolic, nhựa hóa gỗ, vỏ trấu

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