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Research article

Effect of thermal processing on the synthesis of the phenolic resin from rice husk

Nguyen Cam Thuy[1,2,*], Huynh Ngoc Minh[1,2], Kieu Do Trung Kien[1,2,*]

ABSTRACT

Rice is a product that plays a vital role in our country's agriculture. In recent years, rice exports have contributed over 3.5 billion USD annually. However, the rice production industry also releases more than 9 million tons of rice husks annually. Part of the waste rice husk is processed by burning. Most of the remaining rice husks are not processed and are released into the natural environment. Disposing of rice husks has low economic efficiency and can cause environmental pollution. Therefore, taking advantage of this waste source for circular agriculture is a matter of concern. This study investigated the thermal processing for synthesizing phenolic resins from rice husks. The differential scanning calorimetry results show that the resin-forming reaction temperature is 150 ^oC. Different heat retention times (120, 150, 180, 210, and 240 minutes) at 150 ^oC were also investigated. The visual evaluation and determination of rice husk residue indicated that 180 minutes was the appropriate time to make phenolic resin from rice. husks at 150 ^oC. Under this condition, the formed plastic is solid and can be ground into fine particles; the remaining rice husk residue is low, accounting for 9.66% of the weight. The plastic reaction efficiency is almost unchanged if the heat retention time is longer than 180 minutes (210, 240 minutes). The Fourier Transform Infrared Spectroscopy spectrum analysis results show that the main functional groups of the resin are CH_n, C=C, C-H, OH, and aromatic rings. These functional groups are similar to the functional groups in the structure of commercial phenolic resins. Liquefied wood can be applied to treat rice husk waste in our country. This treatment method can also contribute to improving the value of rice trees.

Key words: Phenolic resin, liquefaction wood, rice husk, woodceramics

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INTRODUCTION

In the world's increasing food prices, rice is one of the essential agricultural products of our country. Every year, our country's rice export turnover accounts for over 3 billion USD. In addition to the benefits of rice grains, the farming and rice production industry also emits many waste rice husks. The rice husk is estimated to be about 9 million tons per year. Usually, this rice husk is used as fuel or burned. The burning process creates rice husk ash. A large amount of rice husk ash is discharged into the environment. However, burning often brings low economic efficiency and finds hidden risks to human health and the environment. To improve the value of rice and achieve circular agriculture, many studies have tried using rice husks as raw materials to produce higher economic value.

Rice husk has been studied for coking to turn it into a fuel with high use value. Coking rice husk has a high calorific value and low volatile matter. It can be applied as a clean fuel for high-temperature furnaces [1,2]. J. James et al. have also considered rice husk as a source of SiO_2 to make calcium silicate materials. Calcium silicate prepared by this method can be regarded as green cement to replace traditional Portland cement in the future [3]. Rice husk is also considered a precursor material for silicon in the reaction to create SiC [4]. SiC has many outstanding properties, such as heat resistance, electrical conductivity, and high mechanical properties [5–7]. SiC produced from rice husk ash has the advantage that it can be synthesized at low temperatures (only from 800 to 1300 ^oC) instead of above 1500 ^oC, like traditional methods [8]. As a result, the cost of SiC will be reduced. SiC from rice husks can be more easily accessible to consumers.

Phenolic resin is one of the plastics that are widely used in many different manufacturing and civil fields. It can also be used as a raw material to fabricate carbon fiber [9] and as a binder for woodceramics materials [10]. It is usually produced by polymerizing phenol and formaldehyde [11,12]. However, many studies have shown that liquefaction wood methods can ultimately make phenolic resins from wood-based materials [13,14]. Wood-based materials, whose main components are cellulose, lignin, and hemicellulose, can be raw materials for plastic synthesis when combined with phenol and acid/base catalysts [15]. Generally, the

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waste wood is mixed with phenol and additives and then heated at 130-250 ^{o}C to form phenolic resin [16]. Depending on the additive used, the resulting plastic can be thermoset [17] or thermoplastic [18].

An important research direction on liquefied wood is to use waste wood as the primary raw material source. Types of waste wood that have been used include wood powder [19], cashew nut shell [20], wood particleboard [21], sawdust [22], etc. The above studies show that liquefaction wood can be an effective treatment method for wood-based waste. This method can completely be applied to treat rice husk waste in our country.

This study used rice husks as the raw material to synthesize phenolic resin using liquefied wood. The processing temperature for phenolic resin synthesis (including the temperature and reaction time) is the main object of study. Many studies on wood liquefaction have not mentioned the processing temperature for synthesizing phenolic resin. And research on synthesizing plastic from rice husks using the liquefied wood method.

MATERIALS AND METHODS

Raw materials

The primary raw material was rice husk (Long An province, Vietnam). Rice husks were collected, washed, and milled to a size less than 500 μm. The oxide composition of rice husk consists of 6.71% SiO_2, 0.38% Na_2O, 0.15% Al_2O_3, and 92.13% loss of ignition (wt.) [8]. The chemical elemental composition of rice husk consists of 45.91% C, 3.12% N, 6.12% H, 8.38% Si, 0.38% Fe, 0.19% Ca, 0.09% Na, 0.18% K, 0.09% Al, 0.11% P, 0.01% S, 0.02% Mn, and 92.13% others (wt.). X-ray fluorescence and Isotope Ratio Mass Spectrometry were used to analyze the chemical composition results. In addition to rice husk, phenol, and sulfuric acid were also used. Phenol and sulfuric acid are industrial chemicals with a purity of over 98%.

Methods

Rice husk, phenol, and sulfuric acid were mixed according to the composition given in Table 1. The mixtures, after mixing, were analyzed by differential scanning calorimetry (DSC) to choose an appropriate temperature to synthesize phenolic resin by the liquefied wood method.

The thermal effects of samples were determined by the differential scanning calorimetry method (Labsys Evo -Setaram). The analysis temperature was 30 to 300 ^{o}C, with a heating rate of 5 ^{o}C/min in the air environment. The mixtures were also reacted at high temperatures for different heat retention time to determine the right retention time for the resin formation. The retention times and sample symbols are also shown in Table 1. The heat retention time were evaluated through properties such as the visual evaluation, the composition of the residue, and the Fourier transform infrared spectroscopy (FTIR).

A digital camera was used to capture the visual images of the samples.

The rice husk residue was evaluated by the method of dissolving phenolic resin in ethanol solution [20]. The ethanol used was 40ml per 2.00 $\pm$ 0.01g of resin. The forming resins were dissolved in an ethanol solution. The residue after the filter was the remaining RH. The rice husk residue was calculated according to formula (1).

$$\%RH = (m_{rh}/m_{pr}).100\% \quad (1)$$

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 (%),

FTIR spectroscopy was used to evaluate the functional group composition of the formed phenolic resin (Nicolet 6700 – Thermo). The scanning range was 500 to 4000 cm^{-1}. The scan step was 0.96425 cm^{-1}. The spectrum was recorded as the transmittance spectrum.

THE RESULTS AND DISCUSSIONS

The mixture of rice husk, phenol, and H_2SO_4 solution, after being mixed with the ingredients as shown in Table 1, was analyzed by DSC to determine the reaction temperature of resin formation. Figure 1 is the result of the DSC analysis.

The DSC analysis result has five peaks corresponding to five thermal effects. Thermal effects include:

- The first endothermic effect occurs in the temperature range from 125 to 145 ºC, peaking at 138 ºC. This is the physical dehydration of the material [23].

- The second endothermic effect occurs in the temperature range from 145 to 160 ºC, peaking at 151 ºC. These are the physicochemical reactions to form phenolic resins [24].

- The third endothermic effect occurs in the temperature range of 170 to 182 ºC, peaking at 178 ºC. This is the range of phenol evaporation temperature that does not participate in the resin formation reaction [24].

- The subsequent exothermic effect occurs in the range of 185 – 205 ºC with a peak at 200 ºC. This effect corresponds to the stretch of the resin molecular chains [24].

Table 1: COMPOSITION OF RAW MATERIALS AND HEAT RETENTION TIME

Samples	Ratio (wt.)			Heat retention times
	Rice husk	Phenol	H_2SO_4(*)	
S120	1	2	5	120mins
S150	1	2	5	150mins
S180	1	2	5	180mins
S210	1	2	5	210mins
S240	1	2	5	240mins

(*) According to the amount of phenol used

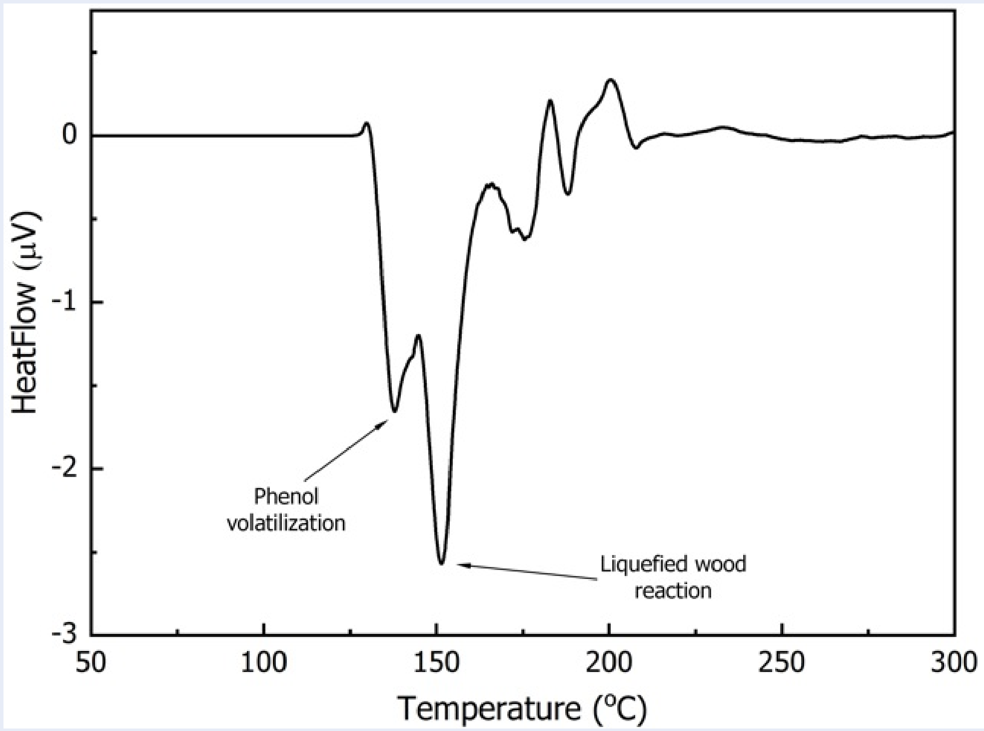

Figure 1: The result of the DSC analysis

- The final endothermic effect occurs in the temperature range from 205 to 215 °C, peaking at 208.31 °C. This is the resin decomposition temperature range[23].

The DSC results in Figure 1 show that 150 ^{o}C is the appropriate temperature to synthesize resin. The resin samples were synthesized at 150 ^{o}C for different heat retention times (120, 150, 180, 210, and 240 minutes). The results of the visual evaluation of the samples are presented in Figure 2.

The results of the visual evaluation of the samples are summarized in Table 2.

The visual results show that all samples have formed resin. However, the resin samples will boil if the heat retention time is too long (210 and 240 minutes). Thermal decomposition and breakdown of the plastic structure can occur if the thermal retention time is too long. So, less than 180 minutes is the appropriate retention time to synthesize resin at 150 ^{o}C.

The reaction efficiency of the resin process was also evaluated by determining the residue of rice husk remaining after the reaction. According to this principle, the lower the reaction efficiency, the more rice husks are left after the reaction. Figure 3 is the analysis results of the remaining rice husks of the samples.

The rice husk residue analysis results show that unreacted residue is relatively low under heat treatment

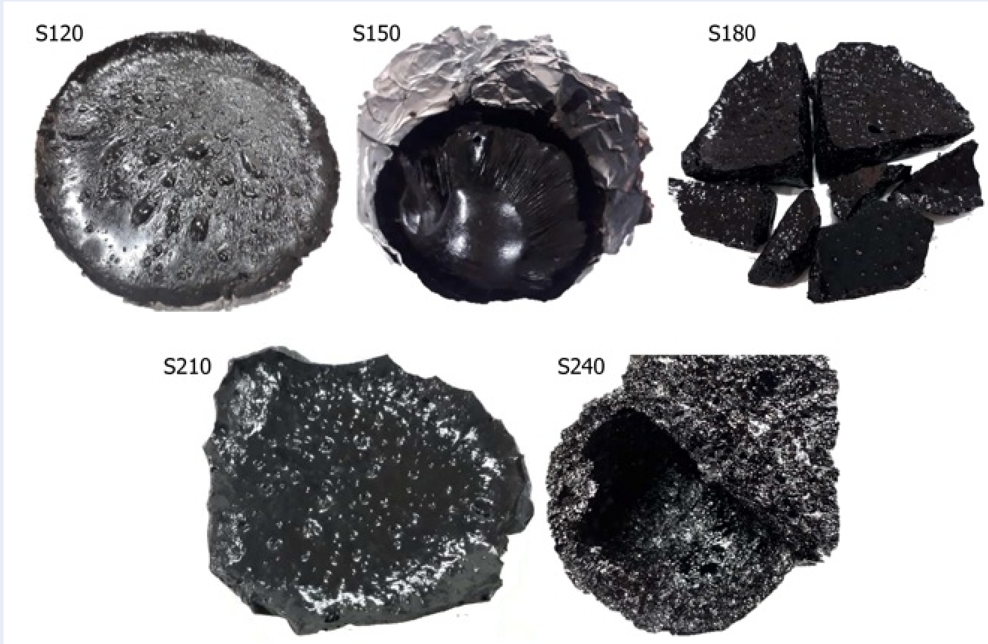

Figure 2: The visual images of samples

Table 2: the results of the visual evaluation

Samples	The results of the visual evaluation
S120	Samples are solid and plastic. When crushed for a while, the particles stick together. It is expected that due to the short heat retention time, the amount of phenol has not been fully reacted.
S150	
S180	The sample is solid. They can be ground into fine-sized.
S210	Samples are solid, porous, and fragile. They gradually lose the glossy color of the resin. It is predicted that the resin is thermally decomposed and creates pores due to the heat retention time being too long.
S240	

conditions. The longer the reaction time at 150^{o}C, the lower the rice husk residue. Sample S120, corresponding to 120 minutes of reaction time, has the highest residue of 13.28% (wt.). Sample S240 has the lowest residue of 9.39% (wt.). In other words, the reaction efficiency will increase by prolonging the resin-forming reaction time. Rice husk residue decreases rapidly when the reaction time is extended from 120 to 180 minutes (from 13.28% wt. to 9.66% wt.). However, if the reaction time is too long (over 180 minutes), the rice husk residue is not significantly reduced (from 9.66% wt. to 9.39% wt.). The results of determining the remaining RH show that the reaction efficiency of forming resin from RH is similar to previous studies on synthesizing phenolic resin by the liquefaction method from wood-based materials [18–20]. The visual evaluation and analysis of rice husk residue show that 180 minutes is the appropriate time to synthesize phenolic resin from rice husk at 150 ^{o}C.

Resin samples at different heat retention times were also analyzed for functional groups by the FTIR method. A commercial phenolic resin sample (symbol CP) was also investigated and considered a control sample to evaluate the resin-forming ability. Figure 4 shows the results of FTIR analysis of the resin samples from rice husk and the commercial sample.

The results of FTIR analysis in Figure 4 show that the plastic samples formed at different time intervals have the same spectral form as the CP sample. The wave number position 3400 cm^{-1} is character-

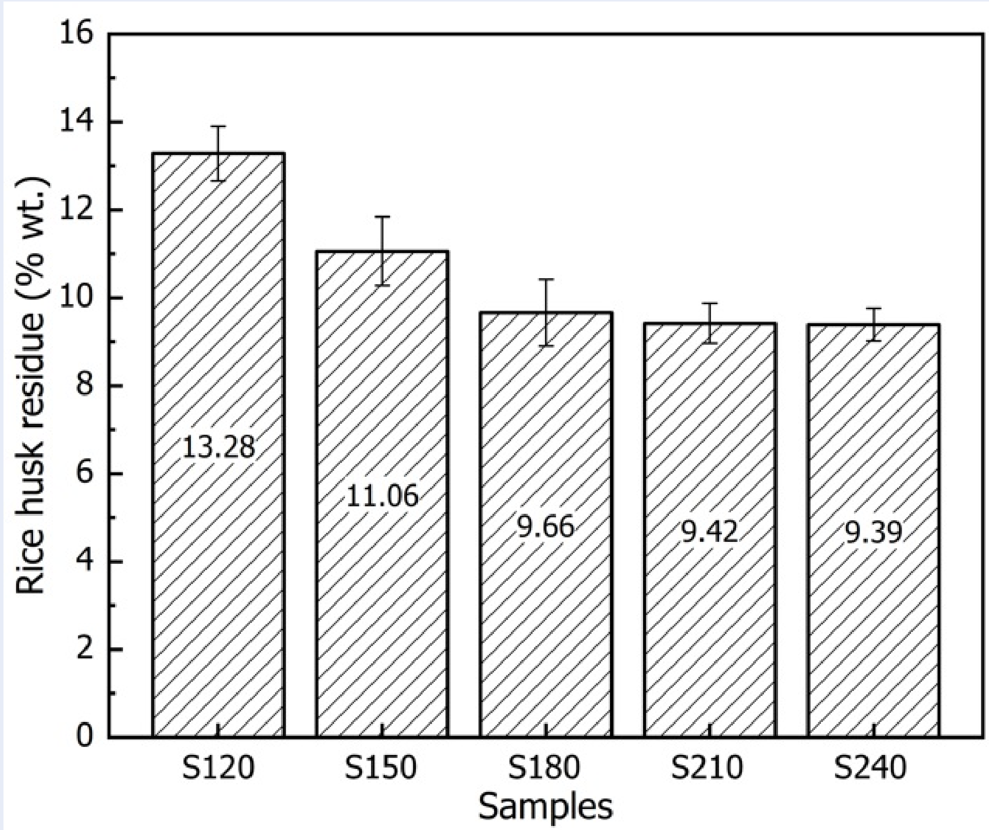

Figure 3: The rice husk residue of samples (%wt.)

istic of the stretching vibration of the -OH group in phenolic and methylol[25]. At wavenumber 2920, 2850 cm^{-1} represents the longitudinal oscillation of the CH_{n-} [26–28] group. At the position of wavenumber 1600, 1510, 1440 cm^{-1}, it is typical for the vibration of the C=C group in the aromatic ring[26–29]. The position of wave number 1355 cm^{-1} characterizes the C-H group[25–27]. The position of wave number 1220 cm^{-1} represents the -OH group[25–27]. At position wavenumber 1020, 825, 760, and 695 cm^{-1}, it is typical for stretching vibration of the C-H group in the benzene ring[25–27]. At the position of wave number 570 cm^{-1}, it is typical for the bending vibration of the out-of-plane aromatic ring[25–27].

The FTIR results can prove that the liquefied wood method has produced phenolic resin from rice husk. Phenolic resin from rice husk has a functional group composition similar to commercial phenolic resin.

CONCLUSION

This study used phenolic resin from rice husk, phenol, and catalyzed sulfuric acid by liquefied wood. With the composition of phenol/rice husk being 2/1 and 5% acid catalyst (wt.), the results of differential thermal analysis show that 150 ^oC is the suitable temperature to synthesize resin. After the reaction, the visual evaluation and the residue of the rice husk's remaining results show that 180 minutes is the appropriate reaction time at 150 ^oC. The results of the FTIR comparison of rice husk resin and commercial phenolic resin samples show that their functional group is similar. Liquefied wood from rice husk can be considered an effective method to treat rice husk waste, improving economic efficiency for rice.

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CONFLICT OF INTEREST

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

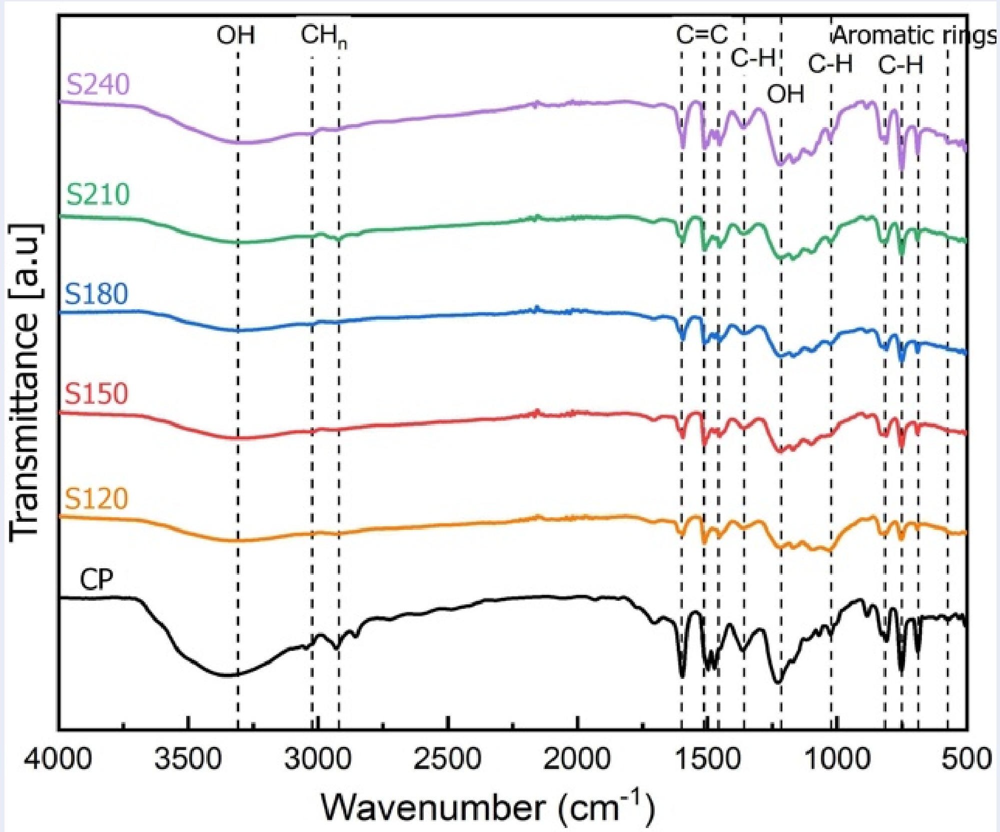

Figure 4: The FTIR spectrum of samples

AUTHOR'S CONTRIBUTION

The authors confirm contribution to the paper as follows: study conception and design: Kieu Do Trung Kien; data collection: Kieu Do Trung Kien; analysis and interpretation of results: Huynh Ngoc Minh; draft manuscript preparation: Nguyen Cam Thuy. All authors reviewed the results and approved the final version of the manuscript.

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Tạp chí Phát triển Khoa học và Công nghệ – Engineering and Technology 2024, 6(SI3):18-25

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Ảnh hưởng của thời gian xử lý nhiệt đến quá trình tổng hợp nhựa phenolic từ vỏ trấu

Nguyễn Cẩm Thùy[1,2,*], Huỳnh Ngọc Minh[1,2], Kiều Đỗ Trung Kiên[1,2,*]

TÓM TẮT

Lúa gạo là sản phẩm giữ vai trò quan trọng trong nền nông nghiệp của nước ta. Trong những năm gần đây, xuất khẩu gạo luôn đóng góp trên 3.5 tỉ USD mỗi năm. Tuy nhiên, hằng năm ngành sản xuất lúa gạo cũng thải ra hơn 9 triệu tấn vỏ trấu mỗi năm. Một phần trấu thải được xử lý bằng phương pháp đốt. Phương pháp này thải ra hàng triệu tấn tro trấu mỗi năm. Phần lớn vỏ trấu còn lại không được xử lý và bị thải ra môi trường tự nhiên. Phương pháp xử lý này mang lại hiệu quả kinh tế thấp và có thể gây ô nhiễm môi trường. Vì vậy, tận dụng nguồn chất thải này hướng đến nền nông nghiệp tuần hoàn là vấn đề đang được quan tâm. Trong nghiên cứu này, chế độ nhiệt để tổng hợp nhựa phenolic từ vỏ trấu đã được nghiên cứu. Kết quả phân tích nhiệt vi sai cho thấy nhiệt độ phản ứng tạo nhựa là 150 ^oC. Các thời gian lưu nhiệt khác nhau (120, 150, 180, 210, và 240 phút) ở 150 ^oC cũng đã được khảo sát. Kết quả đánh giá ngoại quan, xác định dư lượng trấu chưa phản ứng, phổ biến đổi hồng ngoại đã chỉ ra được 180 phút là thời gian thích hợp để tạo nhựa phenolic từ vỏ trấu ở 150 ^oC. Trong điều kiện này, mẫu nhựa tạo thành dạng rắn và có thể nghiền thành hạt mịn, lượng trấu thải còn lại thấp chiếm 9.66% khối lượng. Nếu thời gian lưu nhiệt lâu hơn 180 phút (210, 240 phút), hiệu suất phản ứng tạo nhựa gần như không đổi. Kết quả phân tích phổ FTIR cho thấy các nhóm chức chính của nhựa là CHn, C=C, C-H, OH, và vòng thơm. Các nhóm chức này tương tự như nhóm chức trong cấu trúc của nhựa phenolic thương phẩm. Vì vậy, nhựa hóa gỗ có thể được xem như là một phương pháp để xử lý chất thải vỏ trấu ở nước ta. Phương pháp xử lý này cũng thể góp phần nâng cao giá trị của cây lúa.

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

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