

Utilization of Rice Husk for Production of Multifunctional Liquid Smoke

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Abstract—Rice husk is an abundant and underutilized by-product of rice milling industries, especially in main rice producing countries like Indonesia. The current challenge is how to contribute value addition to the rice milling industries by maximizing utilization of the unwanted rice husk. Recently, there has been a growing interest in utilizing rice husk for production of multifunctional liquid smoke. However, limited studies have been done on this aspect. Previous published reviews are mostly focused on development of liquid smoke from different wood varieties/types or palm oil waste. The main objective of this review is to provide a comprehensive review and further research direction regarding potential development and applications of liquid smoke from rice husk based on the limited and scattered information currently available. This review covers production technology, characteristics, functional properties and potential applications of rice husk liquid smoke. The cited findings showed that there are at least four pyrolysis techniques that can be used for production of rice husk liquid smoke. In addition, there are several factors that may influence the quality of the final product. These include modes of pyrolysis (temperature, heating rate, residence time), biomass composition, and storage condition and period. Each technique and factor can be adjusted and exploited to extract and produce value-added liquid smoke by thermal processing. Rice husk liquid smoke has multifunctional properties, such as antioxidant, anti-inflammatory, antimicrobial and antidiabetic. Composition of major constituents in rice husk liquid smoke determines the magnitude of its functional properties and potential applications. Based on its functional properties, rice husk liquid smoke has a great potential for food and agriculture applications, such as food flavorings, food preservatives, biopesticides, and plant growth regulator. This review will be valuable for both industries and communities. Utilization of rice husk for production liquid smoke will not only benefit rice milling industries and farmers, but also the environment.

Index Terms—rice husk, by-product, liquid smoke, pyrolysis, utilization

I. INTRODUCTION

Rice (*Oryza sativa* L.) is one of staple foods for the world's population, especially in Asia, Africa, and Latin America. In 2017 world production of paddy is predicted to reach around 759 million tonnes. Indonesia is the third largest rice producing country in the world [1]. Indonesia

production of paddy in 2017 is estimated about 77 million tonnes (Ministry of Agriculture, The Republic of Indonesia) or around 10% of the world production. Rice husk is an abundant rice milling by-product accounting for 20% of the unhulled paddy [2], [3]. Annually, Indonesia produces nearly 16 million tonnes of rice husk that is mostly underutilized and becoming a serious problem for rice processing industries.

Rice husk can be classified as a complex lignocellulosic material. It contains cellulose (26-36%), hemicellulose (12-32%), lignin (15-23%), ash (13-23%), and crude protein (2-3%) [4]-[8]. However, Sung, Stone & Sun [8] studied the lignocellulosic composition of three rice husk varieties from USA and they found that the lignocellulosic composition was relatively similar among the three rice husk varieties. One of the most popular applications of rice husk in Indonesia is used as a heat energy source for drying. In this case, rice husk is incinerated and the heat generated is used to dry brick or unhulled paddy. During the incinerating and drying process, smoke generated usually goes to the surrounding air and may result in air pollution.

Separate previous studies showed that the smoke from rice husk incineration and residual rice husk ash may be utilized for production of new valuable products, i.e. rice husk liquid smoke [8]-[11]. Rice husk liquid smoke has been reported to have a number of functional properties, such as antimicrobial [6], [12], antioxidant [5], anti-inflammation [9], and antidiabetic [7]. Such multifunctional properties enable rice husk liquid smoke to be used for natural insecticide [10], [13], preservative [11] or disinfectant [12].

Recently, there has been a growing interest in utilizing rice husk liquid smoke for production of new valuable products. In addition, previous published reviews are mostly focused on development of liquid smoke from different wood varieties/types [14] or palm oil biomass [15]. On the other hand, reviews on development of rice husk liquid smoke and its derivative products have not been much studied. This article provides a comprehensive review and further research direction regarding potential development and applications of liquid smoke from rice husk. The described findings may have practical implications for food and agriculture sectors, including but not limited to healthier foods, improved human health, and more developed rice processing industries.

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II. PRODUCTION OF RICE HUSK LIQUID SMOKE

A. Processing

Rice husk liquid smoke is basically produced through a pyrolysis process. Pyrolysis of lignocellulosic biomass is a thermal degradation process of organic matter containing hemicellulose, cellulose and lignin through burning or heating process in the absence of air or other molecular oxygen containing gases [16]. Dried rice husk is incinerated and smoke is produced. The resulting smoke is collected and directed to a liquefaction pipe passing through a cooling tank. Once condensation occurs, the smoke is converted to a liquid form along the cooled pipeline. The final product, which is rice husk liquid smoke, has a smoky aroma and sugar-like odor. To separate insoluble materials, the liquid smoke could be left standing for about one week at room temperature. The supernatant is collected as the primary rice husk liquid smoke extract. In general, biomass pyrolysis involves decomposition, oxidation, polymerization, and condensation [16]. Biomass pyrolysis produces three types of products, which are (i) liquid (distillates of volatile condensable compounds), (ii) non-condensable gases, and (iii) solid char [14].

Generally, there are four types of pyrolysis, namely carbonization (slow pyrolysis), torrefaction, intermediate, and fast pyrolysis. The four main pyrolysis techniques have different indicating conditions and compositions of their major products (Table I). Carbonization is a conventional pyrolysis process characterized by a low heating rate (>1 °C/s) and a long solid residence time (hours until days) that favours the production of char. Appropriate slow pyrolysis may produce by-products such as acetic acid and tars (dark and oily material) [16]-[18]. Rice husk liquid smoke is commonly produced at farm level through this type of pyrolysis process, especially in Indonesia.

TABLE I. TYPES OF PYROLYSIS AND THE CORRESPONDING COMPOSITION OF MAJOR PRODUCTS

| Types | Composition (%) | | |
|---------------|-----------------|----------|-----|
| | Liquid | Solid | Gas |
| Carbonization | 30 | 33 Char | 35 |
| Torrefaction | 0-5 | 77 Solid | 23 |
| Intermediate | 50 | 25 Char | 25 |
| Fast | 75 | 12 Char | 13 |

Adapted from de Wild *et al.* [16]

Torrefaction is a pyrolysis type with a mild thermal treatment (~ 290 °C) with heating rate >1 °C/s and solid residence time ~ 30 min. During torrefaction, lignocellulosic biomass is transformed into a solid material. In comparison to the initial biomass, the solid material produced has a higher energy density, a better grindability and a lower moisture susceptibility. As a result, it can be used for an enhanced heat and power generation [16], [19]. Intermediate pyrolysis applies a thermal-conversion technique (400-500 °C) with heating rate 1-1,000 °C/s and hot vapor residence time ~ 10 -30s. The intermediate pyrolysis generates a higher amount of liquid product from biomass than carbonization and

torrefaction. Finally, fast pyrolysis is a thermal conversion-technique indicated by a relatively high temperature (500 °C), heating rate $>1,000$ °C/s and hot vapor residence time ~ 1 s.

The pyrolysis conditions facilitate conversion of biomass into a liquid product. The fast pyrolysis yields a higher fraction of the liquid product than the other three types of pyrolysis. The liquid product, which is also called pyrolysis-oil or bio-oil, can be used for both heat/power generation and a feedstock for chemicals [14], [16].

B. Factors Affecting Yield

Specific studies on factors affecting rice husk liquid smoke yield have not been done extensively. Therefore, there are limited published literatures. Previous studies reported that rice husk liquid smoke yield is influenced by biomass type [10] and pyrolysis temperatures [12]. Mustikawati *et al.* [10] found that slow pyrolysis of rice husk (160°C, 11h) yielded a lower amount of liquid smoke (10,80% of the rice husk weight) than that of coconut shell (13,12%) and wood (12,63%). The differences in liquid smoke yield may be due to differences in the composition of lignin, cellulose and hemicellulose (the main biomass constituents) in rice husk, coconut shell and wood. Then, according to Sukharnikov *et al.* [12] the yield of rice husk liquid smoke generally increased with the pyrolysis temperatures (Fig. 1). It is shown that The yield increased gradually when the liquid smoke generated at temperature below 400°C. The yield of rice husk liquid smoke increased drastically between 400°C and 500°C. There was a yield increase of 23% when the pyrolysis temperature was raised from 300°C to 500°C [12].

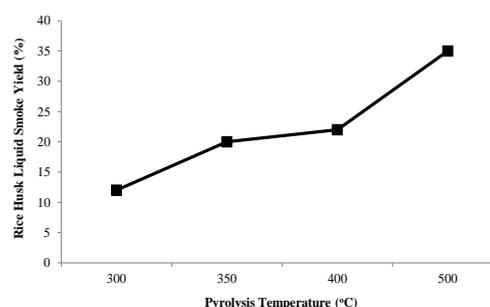


Figure 1. Effect of pyrolysis temperature on rice husk liquid smoke yield (Adapted from Sukharnikov *et al.* [12])

III. CHARACTERISTICS OF RICE HUSK LIQUID SMOKE

A. General Characteristics

General characteristic and composition of biomass liquid smoke are influenced by many factors, including modes of pyrolysis (temperature, heating rate, residence time), biomass composition, and storage period [8], [9], [16], [20], [21]. Chemical and physical characteristics of rice husk liquid smoke are presented in Table II. Previous studies showed that rice husk liquid smoke has a relatively low pH, ranging from 2,3 to 3,0 [8]-[10]. The pH of rice husk liquid smoke (pH 3.0) was even lower than that of coconut shell (pH 4,0) and wood (pH 3,8)

liquid smoke produced using the same pyrolysis mode and conditions [10]. Our unpublished work showed that the pH of rice husk liquid smoke decreased following distillation. This may be due to increased concentration of condensable acid fractions. Sung *et al.* [8] observed that the color of rice husk liquid smoke changed from initial light yellow to dark brown within two weeks of storage. This was shown by the decrease in lightness (L-value) with storage time. The color changes were consistent among three rice husk varieties studied [8] and the changes could be due to oxidation of phenolic compounds [22]. Then, a brown layer of precipitates is commonly settled following sedimentation/storage of rice husk liquid smoke. Presumably, the precipitate consists of lignin monomers and dimmers [23]. The pungent odor may come from phenol, methylphenol and 2,4-dimethylphenol present in the rice husk liquid smoke.

TABLE II. CHEMICAL AND PHYSICAL CHARACTERISTICS OF RICE HUSK LIQUID SMOKE

| Properties | Value/Description | References |
|------------------|---|------------|
| pH | 2.3 - 3.0 | [8]-[10] |
| Specific gravity | 1.007 | [9] |
| Fresh color | Light yellow | [8] |
| Aroma | Smoky | [9] |
| Odor | Pungent, burnt-sugar like | [8], [9] |
| Solubility | Partially soluble in water, highly soluble in alcohol, partially soluble in ether | [12] |

B. Composition

Table III summarizes main chemical composition of rice husk liquid smoke that has been published in different studies previously. In general, phenolic and furan compounds are the major volatile constituents of rice husk liquid smoke produced at pyrolysis temperature of 330 °C and 450-500 °C [8], [9]. The phenolic compounds account for about 85% of the main volatile constituents identified in rice husk liquid smoke [8]. Similarly, Guillen *et al.* [24] reported that the proportion of phenol derivatives in commercial liquid smoke was approximately 65%. However, Mustikawati *et al.* [10] found that rice husk liquid smoke produced at pyrolysis temperature of 160 °C contains mainly acetic acid, accounting for 76% of the major volatile constituents characterized. The proportion of acetic acid in rice husk liquid smoke was higher than that in coconut shell and wood liquid smoke (each around 53%). Meanwhile, rice husk liquid smoke had a lower proportion of phenol and phenol derivatives (12%) in comparison to coconut shell and wood liquid smoke (each about 20%) [10]. Such different results may be due to differences in the pyrolysis temperature set as well as the composition of main constituents in rice husks used. Lignin and cellulose are degraded at temperature of 200 °C or higher. Lignin and cellulose are the main source of phenolic and furan compounds [25]-[27]. Meanwhile, at pyrolysis temperature of 160 °C (below 200 °C) the predominant biomass constituent degraded is hemicellulose, which is the major source of acetic acid [15], [16]. Nevertheless,

appropriate thermal degradation may also transform hemicellulose into furan, furan derivatives [15], [28].

TABLE III. FIVE MAJOR COMPOUNDS IN RICE HUSK LIQUID SMOKE FROM DIFFERENT STUDIES

| Pyrolysis Temperature (°C) | Number of Compounds Specified | Five Major Compounds | References |
|----------------------------|-------------------------------|--|------------|
| 450 - 500 | 161 Volatiles | Phenol; 2,4,-dimethyl-3-(methoxycarbonyl)-5-ethylfuran; 2,6-dimethoxyphenol; 16-keto-tetrahydrosolasodine; benzenecarboxylic acid | [9] |
| 330 | 8 Major Volatiles | 2-Methoxy-4-methylphenol (4-Methylguaiacol); 2-Ethylphenol; 4-Ethyl-2-methoxyphenol (4-Ethylguaiacol); 4-Methoxyphenol; 2-Mehtoxy-4-vinylpneonl | [8] |
| 160 | 16 Major Volatiles | Acetic acid (CAS) Ethylic acid; Phenol, 2-methoxy-(CAS) Guaiacol; Acetic acid, Anhydride with Formic Acid; 2-Methoxy-4-methylphenol; Phenol, 3-ethyl-(CAS) m-Ethylphenol | [10] |

The proportion of major volatile compounds in rice husk liquid smoke is also influenced by the pyrolysis temperatures [8]. This may be due to the fact that hemicellulose, cellulose and lignin (the main biomass constituents) transform in different ways at various temperatures and thus they produce unique volatile compounds [16], [29]. Fig. 2 shows the effect of different temperatures on the proportion of three major volatile compounds in liquid smoke produce from the same rice husk. The percentage of the 2-ethylphenol increases consistently with temperatures. Conversely, percentage of the 2-Methoxy-4-methylphenol (4-methylguaiacol) decreases drastically from 250 °C to 330 °C. The 4-methoxy-phenol content is relatively stable at 200 °C and 250 °C, but the increases gradually at 330 °C. Fenner & Lephardt [30] found that maximum formation of guaiacol and 2-methoxy-4-alkyl-substituted phenols took place at temperature of 385 °C. As a result, the proportions of the three major volatiles in rice husk liquid smoke at 200 °C and 330 °C are different. The data suggest that generation temperatures can be used to control concentrations of the desired constituents present in the rice husk liquid smoke.

The effect of rice husk varieties, storage, and the presence of oxygen or air on the composition of rice husk liquid smoke has also been reported. Sung *et al.* [8] demonstrated that the main volatile compounds present in liquid smoke did not vary with the rice varieties of the original rice husk. Although this may be due to the same or similar composition of lignin, cellulose, and hemicellulose in the rice husk varieties tested. In other

words, different results may be obtained when the compositions of lignin, cellulose, and hemicellulose in the rice husk varieties are widely different. The composition of rice husk liquid smoke remained the same after a 6-week refrigerator storage. If adequate air is present during the preparations, oxygenated compounds such as vanillin, vanillic acid, guaiacol, and acetovanillone would be formed and present in the rice husk liquid smoke [8].

IV. FUNCTIONAL PROPERTIES AND APPLICATIONS OF RICE HUSK LIQUID SMOKE

A. Functional Properties

Functional properties of rice husk liquid smoke have been the subjects of various studies recently. Rice husk liquid smoke has been reported to have antioxidant [5], [31], anti-inflammatory [9], antimicrobial [6], [12] and antidiabetic properties [7], [32]. Salanti *et al.* [5] tested the radical scavenging activity of acidolysis and alkaline enzymatic lignins from rice husk, expressed as IC₅₀ (the half-maximal inhibitory concentration). The results showed that IC₅₀ values of the lignin samples were found to be overall similar to those of reference antioxidants (quercetin, rutin, BHA, BHT). This suggests that lignin derivatives from rice husk exhibited antioxidant activity. Then, Huang *et al.* [31] examined the antioxidant activities of three prominent methoxyphenols of liquid smoke flavouring, from rice hull, which are 4-Methoxyphenol (4-MP), 4-ethyl-2-methoxyphenol (EMP) and 4-propenyl-2-methoxyphenol (isoeugenol). EMP displayed the highest inhibitory effects on radical production and biomolecule oxidation in the acellular systems. The three smoke flavouring phenols also decreased nitric oxide production. The results showed the antioxidant activity of the three phenols.

The anti-inflammatory property of rice husk liquid smoke was examined by Kim *et al.* [9]. The extract from rice husk liquid smoke was administered through topical application of 1% of the extract to skin inflammation of mice or feeding mice with a standard diet with 1% extract for two weeks. Administration of the extract significantly reduced the expression of biomarkers associated with the TPA-induced inflammation. The results showed the anti-inflammatory activities of the rice husk liquid smoke extract.

The rice husk liquid smoke have been tested for antimicrobial activities in previous studies. The phenols, acids and alcohol fractions of rice husk liquid smoke showed bactericidal and fungicidal activities. The phenol fraction exhibited the highest activities, followed by alcohol fraction and acid fraction [12]. The rice husk liquid smoke also assayed for bactericidal activity against the foodborne pathogen, *Salmonella Typhimurium*. The bactericidal activity was tested *in vitro* and *in vivo* using a Salmonella-infected Balb/c mouse model. The results showed that the rice husk liquid smoke extract exhibited inhibitory activity to the bacterial growth, comparable to that of the medicinal antibiotic vancomycin. The most effective way for the antibacterial activity of rice husk liquid smoke extract was 4 consecutive administrations at

12-h intervals that reduced bacterial growth by 75.0%. The inhibitory activity of rice husk liquid smoke extract to the bacterial growth may result from the low pH value. *In vivo* study using mice infected with lethal doses of the Salmonella showed that the use of 1.0% v/w of rice husk liquid smoke extract protected the liver tissues against Salmonella-induced pathological necrosis lesions and thus decreased mice mortality [6].

The antidiabetic effect of rice husk liquid smoke was investigated using mice fed with a high-fat diet. Dietary administration of 0.5 or 1% of the liquid smoke lasted for 7 weeks. The results showed that dietary administration of the rice husk liquid smoke restored (i) glucose-regulating molecule activities to normal control levels, (ii) necrotic damage in the liver to normal levels, and (iii) the reduced insulin-producing β -cell population of the pancreas associated with a high-fat diet intake to nondiabetic normal control levels [7], [11], [32].

B. Potential Applications

Based on its functional properties, rice husk liquid smoke has a great potential for food and agriculture applications. Application of liquid smoke for food uses, such as flavorings, is currently gaining acceptance in food industries since it has a number of advantages over the traditional smoking practices. Liquid smoke can be prepared and applied much easier with more consistent results [11]. Foods flavored with liquid smoke contain a much lower concentration of polycyclic compound [33]. Liquid smoke contributes a more uniform food flavor and color, enables to control smoke intensity in foods [34], and reduce smoke pollution. Because of such advantages, the wood-derived liquid smoke has been used as flavorings in foods [11]. In this case, the rice husk liquid smoke may be used as a new source of liquid smoke flavorings for food applications. However, since the chemical constituents of rice husk liquid smoke may vary widely and the compounds responsible for the observed bioactivity are still not fully identified, evaluation of the all safety aspects of rice husk liquid smoke needs to be done prior to its recommendation for food uses.

In agriculture sector, application of rice husk liquid smoke as an insecticide to soybean plant was demonstrated by Mustikawati *et al.* [10]. In this study, the efficacy of liquid smoke generated from rice husk, coconut shell and wood on soybean pests, armyworm (*Spodoptera* sp.), pod borer (*Etiella* sp.), pod sucking (*Nezara viridulla*, *Riptortus linearis*, and *Piezodorus hybneri*) was compared. The results showed that application of three types of liquid smoke could decrease pest attack significantly and they were equally effective in combating the soybean pests. The percentage of pest attack on soybean plant with liquid smoke treatment was significantly lower as compared to that without liquid smoke treatment. The efficacy of the three types of liquid smoke was comparable to that of the commercial/synthetic pesticide used in the study. The efficacy of the rice husk liquid smoke may result from the bioactives acting as antimicrobial or toxicants for the insect pests of Lepidoptera. Previous data also show that rice husk liquid smoke produced at temperature

160 °C consists mainly of acetic acid. According to Yatagai *et al.* [18], acetic acid play an important role in accelerating the plant growth. Therefore, rice husk liquid smoke can be used for both insecticides and plant growth regulator. Each major volatile compounds in the rice husk liquid smoke may have different antimicrobial or efficacy to insect pests or fungi. These aspects merit further study for development of bioinsecticides or fungicides from rice husk liquid smoke.

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Dr. Hoerudin and his colleagues have developed isolation and purification techniques for cellulose nanofiber from agro-waste materials, silica nanoparticles from rice husk. His major research interests are (i) nanoencapsulation of vitamins, minerals, dan bioactives for development of functional foods, (ii) isolation, modification,

characterization, and utilization of cellulose nanofibers for food-related applications, (iii) nanoencapsulation of bio-based agrochemicals, and (iv) applications of silica nanoparticles from rice husk for agricultural, food and industrial applications.



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