Production and Characterization of Palm Fruit Shell Bio-Oil for Wood Preservation

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Abstract

Pyrolytic liquid (bio-oil), produced by pyrolyzing the shell of the palm fruit, was characterized, and its preservative properties were examined using drywood termites (*Cryptotermes* spp.) and blue stain fungi (*Ceratocystis* spp.). The yield from shell bio-oil production ranged from 35 to 37 percent relative to the mass of the raw material. The shell bio-oil had the following properties: specific gravity (ranging from 1.0365 to 1.0431), refractive index (ranging from 1.3594 to 1.3613), wood absorption level (ranging from 0.0088 to 0.0625 g/cm^3), and retention (ranging from 0.0022 to 0.0141g/cm^3). The shell bio-oil also had termicidal activity as shown by drywood termite mortalities: 25 percent between days 3 and 6, 50 percent between days 3 and 20, and 100 percent between days 5 and 49 in termite resilience tests. Using a fluorescence microscope, we demonstrated that the shell bio-oil–treated wood completely inhibited the growth of blue stain fungi on both pine (*Pinus merkusii*) and Sengon (*Paraserianthes falcataria*) wood.

The annual worldwide demand for palm oil products and derivatives has recently significantly increased. The US Department of Agriculture (USDA) reported that global palm oil consumption reached a record 37.3 million metric tons in 2006 and 2007, representing an increase of 13.2 million metric tons since 2001. This trend of strong increases in palm oil consumption is likely to continue in the coming decade (USDA 2006).

Although palm oil production provides economic advantages in some countries, it also produces problems, especially in developing countries. Production waste, such as fronds, fibers, mesocarps, kernel cakes, palm shells, and liquid waste require proper handling to avoid polluting the environment (Saono and Sastrapradja 1983).

Technology for recycling this biomass using pyrolysis could address such environmental problems and ensure the efficient use of natural resources. Pyrolysis appears to be the most promising technology because of its ease and low cost. Pyrolysis also results in at least two beneficial products: pyrolytic liquid (liquid smoke, vinegar, bio-oil, or tar) and charcoal, both obtained from one operation.

Liquid smoke or wood bio-oil, a product of pyrolysis, has been long used as a food preservative because of its antibacterial and antioxidant characteristics, its color, and its flavor (Gorbatov et al. 1971, Hollenbeck 1979). Wood bio-oil contains a large amount of acetic acid, formic acid, methanol, acetone, phenol, and neutral compounds required for wood preservation. Future uses of pyrolytic liquid are diverse, including as additives for combustion, and as adhesives, fuel enhancers, and specialty chemicals for fertilizers or preservatives (Girard 1992, Vasile and Brebu 2006). Freel and Graham (2002) found that bio-oil derived from wood exhibits properties required of a preservative.

This study focused on identifying and characterizing the physical properties of raw pyrolytic liquid (shell bio-oil) produced from the shell of the oil palm fruit. These properties can be used to address the need for environmental

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wood preservatives and for identifying the properties of pyrolytic liquid obtained from different sources. The shell bio-oil produced from oil palm fruit shells was tested for its usefulness as a wood preservative, and especially for preventing the termite and fungal attacks common in tropical countries. The effects of the preservative in preventing termite attack on wood destined for indoor use and in preventing blue stain fungal attack on freshly cut wood prior to being milled were assessed.

Materials and Methods

Materials

Air-dried shells of oil palm fruit were purchased from Sumatra Plantation (Indonesia). To control moisture content, the shells were stored for approximately 6 months at constant temperature (20°C) and relative humidity (50%) before testing; air-dried shells had a moisture content of 12 percent.

Pine (*Pinus merkusii*) and Sengon (*Paraserianthes falcataria*) logs were obtained from a community forest at Cangkringan village (Yogyakarta, Indonesia). The logs varied in diameter from 25 to 30 cm, and were 2 to 3 m in length. All logs were processed on the same day and were protected from fungal infection.

Termite (*Cryptotermes* spp.) cultures were provided by the Laboratory of Wood Preservation in the Department of Forest Products Technology (Gadjah Mada University, Indonesia).

Methods

Fungi culture preparation.—Blue stain infected sapwood was used as the fungal culture source. To isolate pure cultures, colonies were removed with a razor blade and washed by vortexing in tap water. This process was repeated three times. The fungal suspension was plated onto potato dextrose agar (PDA) medium and incubated at constant temperature (20°C) and humidity (80%) for several days. The resulting colonies were removed and replated onto fresh PDA medium. This process was repeated until a pure culture was obtained; the culture was maintained in an incubator. All specimens were examined microscopically to determine their condition and were used in the experiments below.

Pyrolysis of oil palm fruit shells and the physical properties of the product.—The fruit shells were pyrolyzed in a laboratory furnace at 300°C, 350°C, or 400°C. The temperature was ramped at various rates to reach the final temperature, which was maintained for 4 hours. An ice-cold water condenser trapped the condensate of the released gases. The collected pyrolytic liquid, commonly known as shell bio-oil or liquid smoke, was cooled overnight at room temperature (20°C). The specific gravity of the shell bio-oil was measured by following ASTM Standard D369-84 (ASTM International 2002), and an Atago hand-held refractometer was used to measure the refractive index of the solutions.

Wood sample treatment.—Wood samples were obtained from unseasoned sapwood of pine and Sengon wood. The following sample sizes were used for the different tests: 1 by 1 by 2 cm (absorption and retention under equilibrium conditions), 5 by 5 by 3 cm (resilience against drywood termite attack under equilibrium conditions), and 3 by 3 by 1 cm (resilience against fungal stain under fresh conditions). Wood was sampled randomly as required to provide the needed samples. All wood samples were treated with shell bio-oil either by soaking or dipping and then kept wet for a period of time sufficient to allow the preservative solution to penetrate and diffuse through the cell walls of the wood. The samples of wood for the termite resilience test were then dried, but the samples for the fungicidal test were kept wet. Further testing was conducted to determine preservation properties such as absorption and retention, which are important in determining the quality of a preservative treatment (Hunt and Garrat 1953, Fengel and Wegener 1984).

The absorption value of the pyrolytic oil preserver was calculated by

$$A = \frac{(\text{fww} - \text{iw})}{v} \quad (1)$$

where

A = absorption,
fww = final wet weight (g),
iw = initial weight (g), and
v = sample volume (cm^3).

This study required knowledge of the absorption value in order to identify the amount and depth of active compound penetrating the wood.

The retention value of the pyrolytic oil preserver was calculated by

$$R = \frac{(\text{adw} - \text{iw})}{v} \quad (2)$$

where

R = retention,
adw = air-dried weight (g),
iw = initial weight (g), and
v = sample volume (cm^3).

Termite attack resilience test.—Wood resilience tests against termites (*Cryptotermes* spp.) followed the American Wood Protection Association (AWPA) standards, with some modifications (AWPA 1997, Nakai et al. 2007). Fifty termites were confined with a glass barrier attached to a treated wood sample. For 3 months, termite mortality was calculated daily. The effectiveness of preservation was determined based on termite mortality percentages (25%, 50%, or 100%).

Stain fungi resilience test.—Isolates of fungi were inoculated on fresh wood samples (five replicates) maintained in an incubator at 20°C, and the samples were observed daily under an Olympus BX 41 microscope for 2 weeks to observe the growth and spread of the fungus on the fresh wood. Data obtained were subjected to one-way analysis of variance with completely randomized design and Tukey's multiple range test at a significance level of $P = 0.05$. Factorial design was chosen to examine the effect of each factor (temperature of pyrolysis, type of wood, and method of preservation) to the response variable (result of each type of experiments).

Results and Discussion

Shell bio-oil characteristics

The pyrolytic liquids were heterogeneous and separated into a lower heavy oil phase and an upper aqueous phase;

these phases were not isolated and were studied together as bio-oil. The raw material, palm fruit shell with 12 percent moisture content, yielded between 35.98 and 38.95 percent shell bio-oil (ratio of the weight of bio-oil to the weight of the raw material) following pyrolysis, similar to other recent reports (Phan et al. 2008, Carrier et al. 2010). Carrier et al. (2010) reported that slow pyrolysis at around 450°C, achieved at a heating rate of 15°C min^{-1}, provided good yields, superior quality of the char product (>30%, wt/wt), and optimum yields of bio-oil.

Statistical analysis indicated no significant differences in the yield and specific gravity of shell bio-oil produced at the three temperatures tested for pyrolysis (Table 1) and significant effect on refractive index of the bio-oil (Table 2).

The specific gravity of shell bio-oil ranged from 1.0365 to 1.0431, fulfilling the AWPA Standard P1 for wood preservatives, which requires a specific gravity of not more than 1.025. The refractive index of the shell bio-oil ranged from 1.3598 to 1.3610, fulfilling the Acceptance Criteria for Organic Wood Preservatives Systems (Annex A) of International Code Council–Evaluation Service (ICC-ES) 2007, which is applied in the United States (ICC-ES 2009). Thus, palm shell bio-oil offers promising prospects as a wood preservative.

Application of shell bio-oil for wood preservation

Absorption and retention of shell bio-oil.—The shell bio-oil penetrated and diffused into the two types of wood differently (Table 3). Generally, pine (softwood) showed higher absorption than Sengon (hardwood). Water-soluble and water-based preservatives quickly and easily penetrate the vessels of hardwood by diffusing through cell walls. The penetration of preservatives through softwood cells is limited by the presence of a dotted boundary, which results in decreased permeability of preservatives. Thus, the preservative requires more time to penetrate pine wood. Longer soaking times will allow more preservative to be absorbed by the wood. The tracheid valve structure of pine wood resulted in the preservative residing longer in pine than in Sengon wood. The difficulty of water-soluble preservatives to impregnate resin channels will limit their value for treating pine wood (Freeman et al. 2002) but likely explains why pine wood absorbed more shell bio-oil than Sengon wood in the dipping experiments.

Long-term effective wood preservation requires adequate retention, so various chemical preservatives and treatment methods are used in accordance with the properties of each type of wood. Based on the test result, although only intended for short-term preservation, this bio-oil has shown good retention value, especially for specific types of wood (pine and Sengon) and uses (AWPA 1997). In this study, the average retention value of both preserved pine and Sengon wood was 0.005917 g/cm^3, although that of Sengon wood (0.006126 g/cm^3) was overall higher than that of pine wood (0.005707 g/cm^3). Statistically, the absorption value was influenced by type of wood, and the retention values were only influenced by the temperature of pyrolysis used in preparing the preservatives (Table 4).

Table 2.—Comparisons of refractive index means at different temperatures of pyrolysis.

Pyrolysis temp (°C)	Mean difference	SE	*P*	95% CI
300 and 400	−0.0013167[a]	0.0002	0.000	−0.00193 to −0.00071
350 and 400	−0.0009333[a]	0.0002	0.003	−0.00154 to −0.00032

[a] The mean difference is significant at the 0.05 level.

We found that shell bio-oil retention on yellow pine is comparable to that of other waterborne preservatives such as copper naphthenate 1 percent (5.278 kg/m^3), chromated copper arsenate 1 percent (5.626 kg/m^3), or ammoniacal copper quat type C 1 percent (8.439 kg/m^3) (Freeman et al. 2002). Generally, retention level varies depending on the species, assay zones, climatic conditions, and exposure conditions. The retention levels obtained in this study are average for preservative retention levels issued by AWPA Commodity Standards (AWPA 1997).

Termiticidal effect of shell bio-oil.—The termiticidal test indicates wood resilience, reflected by the mortality of drywood termites. In this study, the wood protection properties of shell bio-oil were determined by dipping or soaking the entire wood sample in shell bio-oil. All three shell bio-oil samples exhibited clear and similar termiticidal activity (Table 5). The observed drywood termite mortalities were approximately 25 percent between days 3 and 6, 50 percent between days 3 and 20, and 100 percent between days 5 and 49. Statistically, there was no significant difference in the results between/within the temperature of pyrolysis, type of wood, and method of preservation observed in this study.

It was previously reported that bio-oil (wood bio-oil) has little to no termiticidal activity and that it is weak compared with synthetic termiticides (Yamanoi 1993). In contrast, the results of our study strongly suggest that shell bio-oil has the potential to be a low-cost and environmentally low-impact wood preservative. Yatagai et al. (2002) also showed that acetic acid, which is the largest component of the wood bio-oil from carbonization of wood, exhibited high termiticidal activity against *Reticulitermes speratus*.

Fungicidal activity of shell bio-oil.—The preservation of wood by shell bio-oil against attack by blue stain fungi (*Ceratocystis* spp.) was measured. Using a fluorescence microscope, we demonstrated that wood treated with shell bio-oil completely inhibited the growth of blue stain fungi on both pine wood and Sengon wood (Table 6). Fungi only

Table 1.—Mean and standard deviation of yield, specific gravity, and refractive index of shell bio-oil.[a]

Pyrolysis temp (°C)[b]	Yield	Specific gravity	Refractive index
300	0.3750 (2.950E−02)	1.043117 (2.80638E−02)	1.359750 (2.25832E−04) A
350	0.3500 (2.828E−02)	1.041967 (2.88972E−02)	1.360133 (3.20416E−04) A
400	0.3578 (3.545E−02)	1.036467 (2.51339E−02)	1.361067 (5.85377E−04) B

[a] In the same column, means followed by different letters are significantly different (Tukey's test, 0.05 α level). Means followed by no letters are not significantly different (statistically similar).
[b] Pyrolysis temperature caused significant differences in refractive index of bio-oil.

Table 3.—Average absorption and retention properties of shell bio-oil.

Wood	Pyrolysis temp (°C)	Soaking time (h)	Absorption (g/cm^3)[a]	Retention (g/cm^3)[b]
P. merkusii	300	Dipped	0.0097	0.0039
		24	0.0359	0.0066
		48	0.0625	0.0080
	350	Dipped	0.0088	0.0037
		24	0.0540	0.0066
		48	0.0701	0.0110
	400	Dipped	0.0103	0.0045
		24	0.0487	0.0035
		48	0.0465	0.0034
P. falcataria	300	Dipped	0.0119	0.0058
		24	0.0244	0.0069
		48	0.0271	0.0022
	350	Dipped	0.0102	0.0048
		24	0.0247	0.0064
		48	0.0295	0.0141
	400	Dipped	0.0103	0.0061
		24	0.0235	0.0049
		48	0.0232	0.0038

[a] Absorption was influenced by type of wood. Mean (standard deviation) = 0.0295 (0.0203).

[b] Retention was influenced by pyrolysis temperature: pyrolysis at 350°C provided significantly different results compared with pyrolysis at 400°C. Mean (standard deviation) = 0.0059 (0.0040).

grew well at the neutral pH of the control samples. Therefore, fungi cannot thrive and may die if the timber has an extreme (e.g., acidic) pH. Rather than merely stopping fungal growth, shell bio-oil appears to act as a fungicide and kills spores and hyphae.

To develop a shell bio-oil useful against fungi, it is also essential to conduct wood resilience tests against decay fungi. It has been noted that wood bio-oil exhibits significant activity against white rot, brown rot, and several decay fungi (Freel and Graham 2002, Mazela 2007, Mohan et al. 2008). Freel and Graham (2002) found that bio-oil from wood is more effective against white rot fungi than against brown rot decay fungi. However, bio-oil affords nearly total protection against decay fungi at retentions in excess of 13 lb/ft^3 (Freel and Graham 2002, Mazela 2007).

The development of an effective and safe wood preservative generally requires extensive laboratory and field tests, including leaching studies. Determining the amount of leaching of a biocide preservative is important for predicting the long-term efficacy of treated wood and for determining potential environmental contamination. It is also the first step in finding additives or modifications to reduce preservative leaching (Mourant et al. 2009). Leaching studies should demonstrate the amount of leaching that may occur under field conditions. Information about

Table 4.—Comparison of retention means at different temperatures of pyrolysis.[a]

Pyrolysis temp (°C)	Mean difference[b]	SE	*P*	95% CI
350 and 400	0034056	0.0011149	0.011	0.00068–0.006131

[a] Tukey's test for absorption was not performed because type of wood only contained two parameters.

[b] The mean difference is significant at the 0.05 level.

Table 5.—Average mortality of drywood termites on shell bio-oil–treated wood.[a]

Wood	Pyrolysis temp (°C)	Soaking time (h)	Achieving mortality (d)[b]		
			25%	50%	100%
P. merkusii	300	Dipped	4.33	12	41.67
		24	3.33	4.67	12.67
		48	3	3.33	15
	350	Dipped	5.33	13	41.67
		24	3.67	4.67	26.33
		48	2.67	3.67	5.67
	400	Dipped	4.33	13	41.67
		24	3.33	4	7.67
		48	2	2.33	4.33
	Control		52.33	69.33	88.67
P. falcataria	300	Dipped	3.67	5	17.67
		24	3	4.33	22.67
		48	3.33	6.33	26.67
	350	Dipped	7.33	19.67	49
		24	3.33	5	30.33
		48	3.33	6	22
	400	Dipped	4	10.67	41.67
		24	3.33	5.33	19.33
		48	3.33	3.67	18.33
	Control		56	68	92

[a] No factors (temperature of pyrolysis, type of wood, and method of preservation) significantly influenced termite mortality. Tukey's tests were not performed because there were no significant differences between factors.

[b] Means (standard deviations) = 5.83 (3.35) for 25 percent mortality, 15.56 (11.43) for 50 percent mortality, and 32.67 (11.18) for 100 percent mortality.

*Table 6.—Average of fungal (*Ceratocystis *spp.) growth on shell bio-oil–treated wood.*[a]

Wood	Pyrolysis temp (°C)	Soaking time (h)	Fungal growth
P. merkusii	No preservative treatment	Control 1	Growth
		Control 2	Growth
		Control 3	Growth
	300	Dipped	No growth (died)
		24	No growth (died)
		48	No growth (died)
	350	Dipped	No growth (died)
		24	No growth (died)
		48	No growth (died)
	400	Dipped	No growth (died)
		24	No growth (died)
		48	No growth (died)
P. falcataria	No preservative treatment	Control 1	Growth
		Control 2	Growth
		Control 3	Growth
	300	Dipped	No growth (died)
		24	No growth (died)
		48	No growth (died)
	350	Dipped	No growth (died)
		24	No growth (died)
		48	No growth (died)
	400	Dipped	No growth (died)
		24	No growth (died)
		48	No growth (died)

[a] No factors (temperature of pyrolysis, type of wood, and method of preservation) significantly influenced fungal growth.

leaching must be evaluated with respect to exposure conditions and product type. Previous scientists have highlighted the need for further research to address the effects of exposure to different environments, such as fresh water, seawater, and highly organic environments, and the need to monitor the overall environmental fate of leached wood preservative components (Lebow 1993, Hingston et al. 2001, Obanda et al. 2008).

Conclusions

The yield of shell bio-oil produced from palm shell oil ranged between 35 and 37 percent of the raw material. The following properties of shell bio-oil were determined: specific gravity (ranged from 1.0365 to 1.0431), refractive index (ranged from 1.3594 to 1.3613), wood absorption level (ranged from 0.0088 to 0.0625 g/cm^3), and retention (ranged from 0.0022 to 0.0141 g/cm^3). Shell bio-oil has potential as a wood preservative for preventing attacks by drywood termites and blue stain fungi. The following drywood termite mortalities were achieved: 25 percent between days 3 and 6, 50 percent between days 3 and 20, and 100 percent between days 5 and 49. Blue stain fungi did not grow on shell bio-oil–treated wood.

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