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Original Article

Preliminary evaluation of production and characterization of wood vinegar from rubberwood

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Abstract

This study deals with a slow pyrolysis of rubberwood in a furnace at an atmospheric pressure to produce pyroligneous liquid or wood vinegar. Pyroligneous liquids produced were separated into two fractions based on temperatures in a furnace. The yields of wood vinegar were quantified. Also pH, SG and boiling ranges were determined. The optimum condition of pyrolysis was obtained at the heating rate of 1.4°C/min to the final temperature at 550°C/min with a yield of 27.45%. The pH and specific gravity were attained at about 2.9-3.83 and 1.009-1.027, respectively, depend on process conditions. Also the boiling ranges of rubberwood vinegar approximately were 96-108°C based on the ASTM-D86 method. However, no significantly changes were seen in the pH, specific gravity and boiling ranges of rubberwood vinegar regarding to hearting rates. For characterization, wood vinegar was fractionated by a distillation into three fractions. Both crude and distilled wood vinegars were analyzed by gas chromatography-mass spectroscopy and gas chromatography. Results showed that the wood vinegar is mainly composed of acetic acid. Also the rubberwood pyrolysis yields high methanol content compared to other woods.

Keywords: wood vinegar, pyrolysis, chemical analysis, rubberwood

1. Introduction

Wood vinegar or pyroligneous acid is a by-product of wood pyrolysis in the process of making charcoal. Wood vinegar is generally dark brown, viscous and composed of a very complex mixture of oxygenated hydrocarbons. It is useful for soil improvement, break seed dormancy, germination and especially in biological activity of fungal and termite attack (Kadota and Niimi, 2004; Nakai *et al.*, 2007; Samesshima et al., 2002). Wood vinegar compositions and yields depend on process conditions and compositions of starting material. Extensive studies on the chemical compositions and applications based on starting materials have been conducted with oak, sakura, green tea, bamboo, eucalyptus, mangrove plant and waste biomass (Nakai *et al.*, 2007; Kartel *et al.*, 2004; Loo *et al.*, 2007; Yatagai *et al.*, 2002; Mu *et al.*, 2003; Sakasegawa and Yatagai, 2005). However, there has been no systematic research in the advanced chemical analysis of rubberwood vinegar. It is necessary to clarify the characteristics of rubberwood vinegar to establish new uses; for example, coagulating agent for natural rubber sheets' production, preservative-treated wood and additives of rubber sheet and rubber block productions. (Ferreira *et al.*, 2005; Kartel *et al.*, 2004)

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Pyrolysis is one of the primary thermochemical conversion methods to convert wood/biomass into products of solid char, liquids and gases. Fast pyrolysis gives high liquid yields, but is energy demanding. Previous researches of fast pyrolysis have been investigated into many biomass types; corn stalk, wheat straw, rice straw, sugarcane bagasse, coconut shell (Tsai *et al.*, 2006). Although fast pyrolysis appears as the most promising method for wood vinegar yield, this research concerns only slow pyrolysis since wood vinegar production in Thailand is obtained as the by-product of charcoal production. Slow pyrolysis is the conventional method for charcoal production. These product yields depend on pyrolysis conditions such as pyrolysis pressure, heating rate, final temperature, reactor type, initial amount of the input and particle size (Bridgewater and Grassi, 1991). Although there are many operating factors for pyrolysis, it is found that heating rate and temperature are very important effected to product yields and chemical compositions. Previous studies concluded that higher final temperature favors gas formation while lower final temperature favors char formation. In another word, slow pyrolysis takes a long period of time and favors char yield whereas fast pyrolysis takes a short reaction times and favors high liquid yields. This implies that an optimal temperature and heating rate to produce wood vinegar must be determined. (Apaydin-Varon *et al.*, 2007; Antal and Varhegyi, 1995; Di Blasi *et al.*, 1999; Demirbas 2000; Goyal *et al.*, 2006, Kucuk and Demirbas; 1997, Putun *et al.*, 2007; Seebauer *et al.*, 1997).

In this study, rubberwood has taken as the biomass raw material to produce wood vinegar. Rubberwood or Para wood (*Hevea brasiliensis*) is one of the leading economic crops of Thailand which is widely planted in southern Thailand. Rotation period of this crop is 25-30 years, the time which yield drops and the trees are cut down for replantation. This implies rubberwood biomass could be claimed from 3-4% of the planting area annually. In general, 90% of the wood are residues, which comprise of 54% small branches, 32% are wastes at sawmills and 4% in furniture industries (Prasertsan and Sajjakulnukit, 2006).

The objective of this study was to investigate the optimal conditions for wood vinegar production of rubberwood. In addition, the fractional distillation was employed as an advanced separation to obtain three distilled wood vinegar fractions and determined their chemical compositions. Although pyrolysis products consist of three parts; solid char, gases, and wood vinegar, this research is reported only wood vinegar results.

2. Experimental procedures

2.1 Preparation of biomass

Rubberwood logs as starting materials were obtained from the local sawmill in Songkhla Province, Thailand. Only parts of slab cuts from logs were used.

2.2 Chemicals

All chemicals used for analysis were AR or GC grades obtained from Merck, Fluka, and Aldrich.

2.3 Pyrolysis production

Only parts of slab cuts from rubberwood logs (length 30 cm) were carbonized in a horizontal-cylindrical furnace. It was 45 cm in internal diameter and 80 cm in length. The furnace was filled with wood logs of approximately 35 cm organized carefully in a horizontal position such that smaller wood logs were placed at the bottom. By opening and covering the orifice of the furnace, the entrance of air was controlled and effected to heating rate in the pyrolysis. Parameters indicative of combustion that comes with practice were the color and the smoke volume emitted from the furnace. The blue smoke indicates the process of wood carbonization. The furnace was heated from room temperature to the end of smoke collection under various heating rates. The smoke released from materials was cooled and collected in a condenser as shown in Figure 1. Smoke collection was started at the beginning of carbonization and continued until smoke is clear. The wood vinegar was collected dividing into two parts with the rising of temperatures from room temperature to 400°C (RW-1) and 400°C up (RW-2).

2.4 Fractional distillation method

Separation of compounds from crude wood vinegar was achieved by means of fractional distillation. The 1.5 L of crude wood vinegar (RW-1 and RW-2) was fed into a 3.5 L, round bottom still pot of fractional distillation and then heated from room temperature to 105°C at 3°C/min and held until at the end of liquid collection in a receiving flask. The column contains a packing material of semi- ring. Top condenser is set at the top of column which provides reflux ratio of 4:1. Moreover, the conduct tube of the second condenser

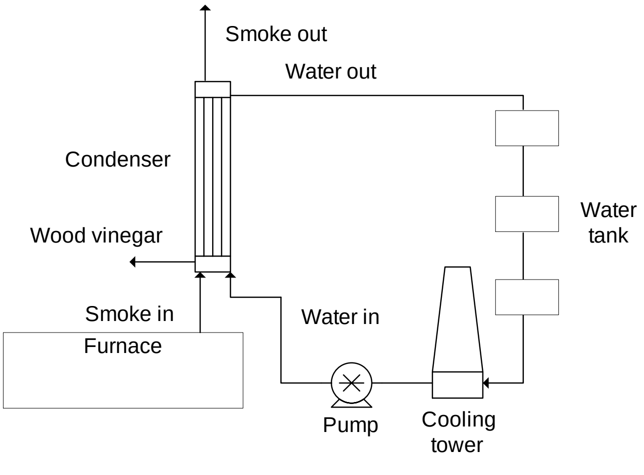

Figure 1. Rubberwood pyroligneous acid production

was set for increasing an efficient of a condensed process. The vapor formed in a still pot is continuously removed and condensed in a two consecutive condenser to produce a distillate. Distillates were collected dividing into three fractions with the rising of the heater temperatures from room temperature to 95°C (DW-1), 95-105°C (DW-2), and then the rest in a still pot (DW-3). The target temperature of 105 °C was maintained until no distillate came out in a collector.

2.5 Physical characteristics

pH of wood vinegar was measured with a pH meter (Mettler Toledo). Specific gravity (SG) was measured with a pycnometer. Boiling range was measured by the ASTM D86 distillation apparatus. Yields were calculated as the weight ratio of wood vinegar to rubberwood fed into a furnace.

2.6 Gas chromatography-mass spectroscopy (GC-MS) and gas chromatography (GC)

The samples were analyzed by gas chromatography-mass spectroscopy (GC- MS) using an HP GC 5890 and MSD 5972 (Hewlett-Packard, Palo Alto, CA) equipped with a column 30m x 0.32mm i.d. x 0.5-mm film thickness (polyethylene glycol), J&W Scientific Innowax. The oven temperature was programmed from 40 to 250°C at a heating rate of 2°C/min such that it was maintained at 40°C for 10 min, then the temperature was raised to 250°C and held at 250°C for 30 min. As a carrier gas, helium was used. For GC, HP 6850 series II and HP 6890 series gas chromatographs (Hewlett- Packard, Palo Alto, CA) equipped with a flame-ionization detector (FID), a sniffing port, and a splitless injector was used.

3. Results and Discussion

3.1 Yield, pH, SG and boiling range

Crude wood vinegar obtained from this work is separated by two fractions of RW-1 and RW-2. These two fractions are significantly different in colors. The RW-1 is radish brown whereas the RW-2 is dark brown. This is because wood destructive reactions in high temperature tend to increase tar formation compared to the RW-1 which collected at lower temperature. In similar, DW-1 is the clear liquid while DW-2 is light yellow and DW-3 is deep dark brown. Table 1 shows the crude wood vinegar yields in relation to the heating rate. It was found that higher heating rate tends to favor wood vinegar yields. Moreover, RW-2 quantities tend to increase in high heating rate process. Yields of crude wood vinegar ranges from 23.27-29.01%.

The pH values of crude wood vinegar are acidic with the increasing acidic of RW-1 compare to RW-2 as shown in Table 1. This is because acidic chemicals during wood destructive of hemicellulose and cellulose are formed in the temperature range of 200-350°C. In making comparisons, the wood destructive at high temperature tends to favor phenolic and its derivatives instead of carboxylic acid. Therefore, RW-2 is less acidic. Also the pH results in this work are in agreement with Lim, 2002. The SG of crude wood vinegar is also shown in Table 1. Little difference was observed in the SG of the RW-1 and RW-2. Compare to other works, the SG in this research are in good agreement.

Table 2 shows pH of distilled wood vinegar obtained from both RW-1 and RW- 2 as feeds. Also in making these comparisons we note that the pH of distilled wood vinegar are lower than those of the crude wood vinegar because of the loss of water during distillation. Fraction DW-2 showed strong acidity. It was found that DW-2 tends to be the highest acidic property compared to other fractions based on their boiling points of acidic chemicals. Moreover, pH of distilled wood vinegar from RW-2 seems to be systematic higher than those obtained from RW-1. This is in agreement with the previous pH results of RW- 1 and RW-2 as shown in Table 1.

Table 3 shows SG of distilled wood vinegar. It was found that the SG of DW-1 is the lowest since the fractional distillation separates based on its chemical boiling point. This is in agreement with the results of DW-3 such that DW-3 provides the highest SG since chemicals in DW-3 are having higher boiling ranges and tends to have complicate molecular structures. It was also concluded that the heating rates do not effect to the SG.

Table 4 shows the boiling ranges of crude wood vinegar. No significantly change was seen in the boiling ranges of wood vinegar regarding to heating rates. It was found that the average boiling range is approximately at 96-108°C which shows a very narrow range and approximately closed to the boiling point of water at atmospheric pressure. This is because the main component of wood vinegar is water. Although wood vinegar contains many chemicals, the concentrations of these chemicals are only small amounts. Therefore, the boiling range of wood vinegar is overshadowed by water which its boiling point is at 100°C at atmospheric pressure.

3.2 Chemical compositions of wood vinegar analysis

Table 5 shows examples of chemicals found in crude and distill wood vinegar obtained from GC-MS. Chemicals were found in many groups such as carboxylic, alcohol, ketone, aldehyde and chemicals from lignin destructive; namely, phenol, guaiacol and phenolic derivatives. This is because wood destructive depends on temperature such that hemicellulose was started at temperature of 100-260°C, then followed by the destructive of cellulose at temperature of 240-350°C. Finally lignin was changed at temperature of 280-500°C. (Fengel and Wegener, 1984; Chen *et al.*, 2001; Kartel *et al.*, 2004) The most chemical species of RW-1 and RW-2 have found to be the same. This is because wood pyrolysis involves concurrent and consecutive reactions based on wood chemical compositions. Therefore, wood destructives

Table 1. Yields* of overall crude wood vinegar from furnace, pH and SG of crude wood vinegar of RW-1 and RW-2

Heating rate (°C/min)	Overall crude wood vinegar yields (%)	pH		SG	
		RW-1	RW-2	RW-1	RW-2
0.6	23.27	3.20	3.16	1.011	1.009
0.7	24.45	2.90	3.83	1.014	1.016
0.9	24.91	3.48	3.54	1.012	1.010
1.4	27.45	3.00	3.31	1.012	1.015
2.0	29.01	3.15	3.37	1.024	1.027

* % by weight of wood vinegar to weight of rubberwood fed into a furnace

** Other reported work of rubberwood wood vinegar was as follows: Lim *et al.*, 2002: pH 2.98 and SG 1.014.

Table 2. pH of distilled wood vinegar of DW-1, DW-2 and DW-3

Heating rate (°C/min)	RW-1 (as feed)			RW-2 (as feed)		
	DW-1	DW-2	DW-3	DW-1	DW-2	DW-3
0.6	2.93	2.42	3.01	2.98	2.67	3.10
0.7	2.85	2.46	3.01	2.93	2.81	3.25
0.9	2.77	2.60	2.99	2.76	2.63	3.26
1.4	2.91	2.64	3.12	3.00	2.57	3.04
2.0	3.09	2.79	3.52	3.02	2.81	3.61

Table 3. SG of distilled wood vinegar

Heating rate (°C/min)	RW-1 (as feed)			RW-2 (as feed)		
	DW-1	DW-2	DW-3	DW-1	DW-2	DW-3
0.6	0.984	1.002	1.034	0.991	1.003	1.042
0.7	0.992	1.002	1.022	0.989	1.005	1.035
0.9	0.976	1.004	1.028	0.983	1.006	1.047
1.4	0.984	1.007	1.035	0.981	1.003	1.036
2.0	0.983	1.003	1.109	0.982	1.004	1.082

Table 4. Boiling ranges of crude wood vinegar from a furnace

Heating rate (°C/min)	Boiling range (°C)
0.6	97-108
0.7	96-106
0.9	96-105
1.4	98-107
2.0	99-106

of hemicellulose, cellulose and lignin occur at the same time such that while parts of wood started to carbonized, other parts of that wood have already carbonized. Therefore, combustion gases from these reactions came out at the same time.

Table 6 shows major chemical compositions of wood vinegar. The most abundant compound was acid, especially

Table 5. GC-MS results of crude and distilled wood vinegar

Substance	RW-1 (crude)	RW-2 (crude)	RW-1 (Distillation)			RW-2 (Distillation)		
			DW-1	DW-2	DW-3	DW-1	DW-2	DW-3
Acetic acid	✓	✓	✓	✓	✓	✓	✓	✓
ProPionic acid	✓	✓	✓	✓		✓	✓	
Formic acid	✓	✓	✓	✓	✓	✓	✓	✓
Butyric acid	✓	✓	✓	✓	✓	✓	✓	✓
Methanol	✓	✓	✓	✓	✓	✓	✓	✓
Acetone	✓	✓	✓	✓		✓	✓	
2-CycloPenten-1-one				✓			✓	
2-Methyl-2-cycloPenten-1-one			✓	✓		✓	✓	
2,3-DimethylcycloPent-2-en-1-one			✓	✓		✓	✓	
2,3-Dimethyl-2-cycloPenten-1-one			✓	✓		✓	✓	
2-Hydroxy-2-cycloPenten-1-one					✓			✓
2-Hydroxy-3-methyl-2-cycloPenten-1-one					✓			✓
1,4-Dimethoxybenzene			✓	✓		✓	✓	
4-Methoxy-2-methyl-1-(methylthio)benzene					✓			✓
Maltol					✓			✓
Furfural	✓	✓	✓			✓		
Furfuryl alcohol			✓	✓	✓	✓	✓	
2-Furylmethylketone			✓			✓		
Guaiacol	✓	✓		✓			✓	
Phenol	✓	✓			✓			✓
o-Cresol			✓	✓	✓	✓	✓	✓
m-Cresol				✓	✓		✓	✓
P-Cresol			✓	✓	✓		✓	
2-Methoxy-4-methylPhenol			✓	✓		✓	✓	
4-Ethyl-2-methoxyPhenol			✓	✓		✓	✓	
o-EthylPhenol			✓	✓		✓	✓	
m-EthylPhenol			✓	✓		✓	✓	
2-Ethylfuran			✓			✓		
2,6-DimethylPhenol			✓	✓	✓	✓	✓	✓
2,5-DimethylPhenol			✓			✓		
2,6-Xylenol			✓			✓		
2,5-Xylenol			✓			✓		
2,6-DimethoxyPhenol					✓			✓
Phloretin					✓			✓
4-Hydroxy-3,5-dimethoxybenzaldehyde					✓			✓
Pyridine					✓			✓
1-Hydroxy-2-butanone					✓			✓
Dihydro, 2(3H)-Furanone					✓			✓
2-Furanone					✓			✓

P: found sPecific chemical

acetic acid. Acetic acid is derived from acetyl groups during pyrolysis. Fenkel and Wegener (1984) reported that hemicellulose thermal degradation provides acetic acid, methanol, furfural, aldehyde and ketones. The increasing in acidicity of wood vinegar was caused by the removal and leaching of organic acids from wood (Kartel *et al.*, 2004). Generally, acids are formed from the destructive reactions of cellulose, hemicellulose and lignin. This is in agreement with the chemical composition of rubberwood itself which contains hemicellulose and cellulose almost 70% by weight. As seen, acids and phenols are dominated compared to other chemicals and contributed to acidic pH values. For phenol and its derivatives, it is derived from the lignin pyrolysis. Generally, phenolic compound quantities increased as pyrolysis temperature increased. It is likely that differences in the concentrations of phenolic compounds in these fractions were caused by their boiling points of these chemicals. However the higher concentrations of phenolic compounds in RW-2

Table 6. Compositions of crude and distilled wood vinegar (% by weight of component in a stream)

Substance	RW-1	RW-2	RW-1 as feed			RW-2 as feed		
			DW-1	DW-2	DW-3	DW-1	DW-2	DW-3
Acetone	0.027	0.021	0.679	0.010	-	0.429	0.037	-
Acetic acid	4.524	4.259	3.302	5.125	2.525	2.795	5.568	2.413
Formic acid	0.132	0.124	1.116	0.095	0.062	1.653	0.129	0.004
Propionic acid	0.250	0.220	0.378	0.365	-	0.720	0.433	-
Butyric acid	0.122	0.182	0.014	0.016	0.006	0.220	0.021	0.008
Methanol	0.215	0.249	6.774	0.047	0.008	8.746	0.022	0.007
Furfural	0.102	0.132	0.077	0.089	0.130	0.130	0.123	0.138
Guaiacol	0.029	0.040	-	0.008	0.077	-	0.026	0.763
Phenol	0.015	0.026	0.012	0.016	0.014	0.030	0.031	0.012
p-Cresol	0.010	0.025	0.007	0.009	0.013	-	0.390	-
m-Cresol	0.080	0.072	-	0.077	0.087	-	0.069	0.079

(compared to RW-1) were caused by a pyrolysis temperature.

It was found that the chemical distributions in RW-1 and RW-2 themselves and distilled wood vinegar obtained from RW-1 and RW-2 are the same trends but different in quantities. The results show that phenol and phenolic compounds in RW-2 were higher than RW-1 because of chemical reactions at high temperatures. Acetic acid was found to be the most chemical quantity (except water) in both RW-1 and RW-2. Acetone and methanol were found only in DW-1. Acetic acid is the most component in fraction DW-2.

Quantities of phenolic compounds in DW-3 seems to be higher than DW-2. These results are in good agreement with the physical results as previously shown and explained in Sec.3.1. For chemicals (guaiacol, cresol and some of phenolic derivatives) for wood preservative groups, we found these chemicals in DW-2 and DW-3. This suggested that it is possible to use a fractional distillation to separate major groups of chemicals in wood vinegar. This will be useful to advanced separations such that only some chemicals needed can be extracted using an advanced distillation. The most significant results found in this study that methanol quantities was found to be higher than many report researches of other crops such as bamboo and Eucalyptus. This is because rubberwood is the latex crops which tend to provide methanol formation from thermo-conversion reactions.

4. Conclusion

The main target of this work is to produce wood vinegar in the slow pyrolysis. The possibility of obtaining the wood vinegar with the yield up to 29% by mass at atmospheric pressure and temperature approximately 500-550^{o}C has been shown. Under conditions of increasing in heating rate, yield of wood vinegar seems to be increased. Heating rate appeared to have no significant effects on the pH, SG and boiling ranges of wood vinegar. Qualitative observations also indicated the chemical compositions. Also phenol and phenolic compounds increased with the pyrolysis temperature increased. Fractional distillation can be used as an advanced separated wood vinegar.

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