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ABSTRACT

Coconut wood and coconut shell were subjected to destructive distillation to extract wood vinegar which can be utilized in agriculture, livestock farming, and even in the pharmaceutical industry. This study is centered on determining the physical and chemical characteristics of wood vinegar extracted from two different organic samples. Qualitative and Quantitative Analytical process were used to determine the composition and characteristics of wood vinegar from coconut wood and coconut shell. Results indicate that wood vinegar from coconut wood is orange to dark orange in color while yellowish to dark yellow was observed on coconut shell wood vinegar. The odors of the wood vinegars coming from the 2 samples were the same, according to most respondents which was copra-like odor. pH indicates high acidity on both samples and miscibility to most polar substances. Total Acid Content indicates a slightly higher percentage for coconut wood than coconut shell. Small amounts of soluble tar were observed on the two wood vinegar samples which indicates a high degree of purity and the moisture for coconut wood is lower than the coconut shell. Phenolic compounds were mostly characterized in the wood vinegar from the two samples with an almost similar composition but with varying concentrations, also, other trace chemicals were found on the wood vinegar samples indicating the complexity and varied uses of wood vinegar in different fields of agriculture and pharmaceuticals. It is therefore recommended that a pilot medium scale production of wood vinegar from coconut shell and coconut wood be conducted for agricultural and livestock use.

Keywords: coconut shell, coconut wood, wood vinegar, chemical properties, chemical composition.

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I. BACKGROUND OF THE STUDY

A recent trend in agricultural chemistry and green chemistry which is now utilized by some farmers in Southeast Asia is the Wood Vinegar. Wood vinegar comes from the destructive distillation of wood, wood tar and charcoals are also one of the products of this type of process involving wood.

Wood vinegar is produced when smoke from charcoal production is cooled by outside air while passing through a chimney or flue pipe. The cooling effect causes condensation of pyroligneous liquor, particularly when the temperature of smoke produced by carbonization ranges between 80 and 180°C (Nikhom, 2010 as cited by ECHO, 2012). This temperature is reached at the carbonization stage of exothermic decomposition and is indicated by the production of yellowish, acrid smoke (ECHO, 2012).

According to Payamara, J. (2011) wood vinegar production is a development of traditional process of charcoal burning or the burning of wood in an airless condition reducing it to a charcoal rather than a carbon dioxide, water vapor of firebricks is substituted for the mound of earth, and a device is added to collected and cool the vapors released to condense them.

Current research on wood vinegar states that it has beneficial effects to plants, soil and even on animal diet. Wood vinegar is made as an alternative to some mixture fertilizers which further contain numerous poisons that attach to plants and retain to the soil contaminating it.

Moreover, in the process of wood vinegar, there is only small information regarding the use of coconut shell and its trunk as a source of wood vinegar, hence this study will be conducted, which will in turn give additional source of wood vinegar, not only from true trees, but also from coconut tree.

II. OBJECTIVES OF THE STUDY

This research extracted wood vinegar from coconut shell and coconut trunk as a newer source of wood vinegar. Specifically, this study answered the following:

1. Determine the physical property of the coconut shell and trunk wood vinegar in terms of:
   a. Color
   b. Miscibility
   c. Odor
   d. pH
2. Determine the composition of wood vinegar generated from coconut shell and coconut wood in terms of:
   a. Acid content
   b. Water Content
   c. Soluble Tar Content
3. Determine the present Basic and Neutral substances in the wood vinegars.

III. METHODOLOGY

3.1 Research Design

This experimentally designed study focused on the extracting wood vinegar from coconut shell and wood which is basically used as fuel, and further determined its physical and chemical compositions of the extracted wood vinegar.

3.2 Preparation of Materials

Coconut shells and were collected from Borongan city market. Whereas, coconut wood was collected from the vicinity of Eastern Samar State University, Borongan City. The said components were then coursing and drying it in an open space to exclude water molecules from the samples and readied for destructive distillation.

3.3 Instrument and Data Gathering procedure

Dry distillation of wood was done using procedures from Phywe (2017). The dry distillation (as seen on Figure 1) was assembled by the researchers for a faster rate of extracting wood vinegar. Experimental procedures were done under laboratory conditions, all in triplication to minimize errors. Extra care was also utilized in this experiment for explosive and toxic fumes are emitted during distillation.

Figure 1: Kiln for Extracting Wood Vinegar

3.4 Instruments of the Study

In the following figure (Figure 2) the instruments and reagents that were used in the study are specified:

Position No.	Material	Order No.	Quantity
1	Support rod, stainless steel, l=370 mm, d=10 mm	02059-00	1
2	Support base, variable	02001-00	1
3	Glass beaker DURAN®, short, 250 ml	36013-00	1
4	Test tube rack for 12 tubes, holes d= 22 mm, wood	37686-10	1
5	Boss head	02043-00	2
6	Glass tube,right-angled w.tip,10	36701-53	(1)
7	Protecting glasses, clear glass	39316-00	1
8	Rubber stopper, d = 22/17 mm, 1 hole	39255-01	2
9	Universal clamp	37715-00	2
10	Glass tubes,right-angled, 10	36701-59	(1)
11	Pipette with rubber bulb	64701-00	1
12	Rubber tubing, i.d. 6 mm	39282-00	1
13	Test tube, 180x18 mm,100pcs	37658-10	(1)
14	Test tube,180x20 mm,side arm,PN19	36330-00	1
15	Spoon, special steel	33398-00	1
16	Test tube,180x20 mm,DURAN, PN19	36293-00	1
	Butane burner f.cartridge 270+470	47536-00	1
	Butane catridge CV 300 Plus, 240 g	47538-01	1
	Glycerol, 250 ml	30084-25	1
	Sodium chloride 1000 g	30155-70	1
	Iron wool 200 g	31999-20	1
	Wood splints, package of 100	39126-10	1
	Indicator paper, pH1-14, roll	47004-02	1

Figure 2: Equipment and Reagents used in Dry distillation

3.5 Physical and Chemical Characterization

After extraction, the wood vinegar collected was placed in a dry opaque polyethylene (PET) bottle and placed standing for 24 hours to settle. Once this is achieved, the clear liquid (wood vinegar) from the crude extract was separated by pipetting off to exclude foreign chemicals which may interfere with the results. The pure wood vinegar was then subjected to physical and chemical tests as follows:

Color

The color of the wood vinegar extracted from two different coconut parts were determined by qualitative examination by 5 random respondents using their sense of sight. The perceived majority of color according to the respondents was recorded as the color of the wood vinegar.

Odor

The odor of the wood vinegar extracted from two different coconut parts were determined by qualitative examination by 5 random respondents using their olfactory sense. The perceived majority of odor according to the respondents was then recorded as the odor of the wood vinegar.

pH

The pH of the wood vinegars extracted was determined by using a pH paper. The tip of the pH paper dropped with 3 drops of vinegar samples and the paper was subjected to an indicator for numerical data and recorded. Testing was done in triplication.

Miscibility

Miscibility is the property of a liquid substance to mix in a given solvent. In this test, four (4) solvents namely; 96% ethyl alcohol, benzene, 10% H_2SO_4, and 10% NaOH was used as solvents to determine the solubility of the wood vinegars coming from different parts of the coconut tree. About 2 drops of sample was added to 1 mL of solvent and shaken. It was then allowed to stand for 10 minutes and the results were recorded. Three trials were done on each solvent.

3.6 Composition of Wood Vinegar

Composition of the wood vinegar which was extracted from coconut shells and coconut wood was determined using the procedures from Theapparat Y., Ponglimanont C. and Leelas-uphakul W. (2014).

Acid Content

The total acid content (TAC) and ionization characteristics was evaluated using titration with 0.1 N NaOH whose content was calculated from the volume of the titrated NaOH at pH 8.15 at the termination point of acetic acid, a major organic acid in the wood vinegar (Mun et al., 2007). The NaOH concentration was standardized with 0.1 N potassium hydrogen phthalate.

3.7 Soluble Tar Content

The total soluble tar content was evaluated by transferring a portion (0.5 g) of crude wood vinegar to a calibrated vial and heating at 105 °C overnight to remove any volatile components. The obtained residues were used for calculation of the tar content.

3.8 Water content determination

Titration was used to determine the amount of water in the wood vinegar samples. The titrator was calibrated with dry methanol. Then, 200 µL of wood vinegar was dropped in a container and titrated with the Karl Fischer reagent until reaching the end point. The results were the mean water content ± the relative standard deviation (measured as the percent milligrams of water per milliliter of sample) of three different measurements of each sample.

3.9 Basic and Neutral substances

Qualitative and Quantitative analyses were done using Gas Chromatography and Mass Spectrophotometry (GC/MS) to determine the present and enumerate the substances found in the crude wood vinegar extracted from coconut shell and coconut wood.

IV. RESULTS AND DISCUSSION

Acetic acid is, among other substances, produced by the dry distillation of wood (Phywe, 2017).

The following results were obtained after the testing of crude wood vinegar extracted using the procedures from Phywe (2017) in which the acetic acid (as primary acid) is formed. Also, methanol is formed just as the same time as acetic acid which is from the methoxy groups of the Iignins. The acetic acid content of the distillate is approximately 6%, which is about three times higher than that of methanol.

Also, it was observed that coconut shell when subjected to dry distillation gives more wood vinegar in terms of actual yield than coconut wood, all of which the two samples where thoroughly dried under sunlight for 6 hours.

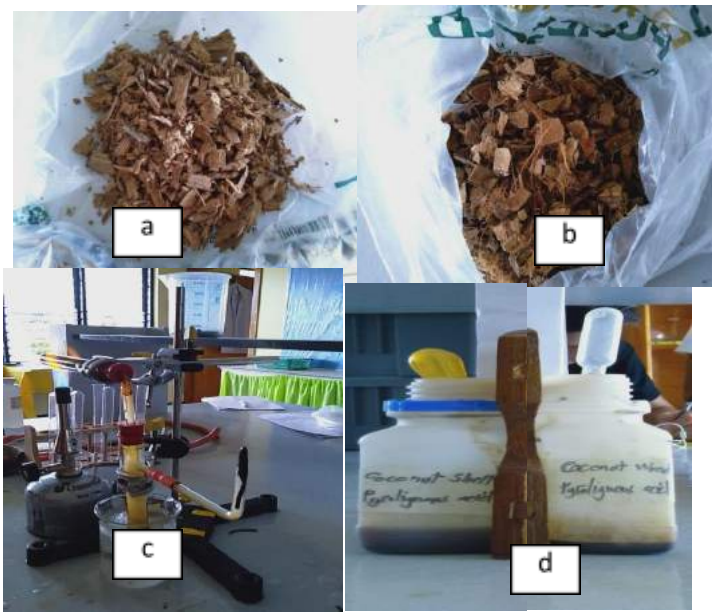

Figure 3: (a) coconut wood; (b) coconut shell; (c) dry distillation; (d) wood vinegars

Physical Properties

The physical property results are herein numerated:

Color and Odor

Table 1: Color and Odor of Wood Vinegars extracted from two (2) samples

	Physical Properties			
	CW Wood Vinegar		CS Wood Vinegar	
	Color	Odor	Color	Odor
R_1	Orange	Smells like burnt wood	Reddish orange	Burnt paper
R_2	Dark orange	Copra-like odor	Yellowish brown	Copra-like odor
R_3	Brown orange	Woody odor	Orange brown	Copra-like odor
R_4	Orange	Copra-like odor	Reddish orange	Copra-like odor
R_5	Dark orange	Copra-like odor	Yellowish brown	Copra-like odor

**CW – coconut wood; CS – coconut shell*

The majority of respondents indicated the color of the wood vinegar extracted from the coconut wood at a range of orange to dark or brown orange. Also, in terms of its odor, the wood vinegar from CW smells like copra, as described by most of the respondents.

Whereas, the wood vinegar from coconut shell was described by the respondents to be reddish brown to yellowish brown in color range, also, in terms of odor, the copra-like odor was the majority of description as stated by the respondents. This states that, certain chemicals that specify the distinctive color and smell of coconut wood vinegar are present.

Miscibility

In terms of the wood vinegars' miscibility, the following results were obtained:

Table 2: Miscibility of the extracted wood vinegar from the two (2) samples

	CW Wood Vinegar			CS Wood Vinegar		
	T1	T2	T3	T1	T2	T3
96% Ethanol	M	M	M	M	M	M
Benzene	IM	IM	IM	IM	IM	IM
10% H_2SO_4	M	M	M	M	M	M
10% NaOH	M	M	M	M	M	M

**M – miscible; IM – immiscible **CW – coconut wood; CS – coconut shell*

The table above show that the wood vinegar extracted from CW and CS are miscible to basic, acidic and alcoholic medium, but in contrast, the wood vinegar samples are immiscible to non-polar organic solvents. This indicates that one of the best methods for administering wood vinegar is the use of polar organic or inorganic solvents for various agricultural procedures.

pH

pH pertains to the acidity or alkalinity of the substance, the following table indicates that pH of the wood vinegar samples:

Table 3: pH of the extracted wood vinegar from the two (2) samples

	CW Wood Vinegar	CS Wood Vinegar
T_1	2.0±0.28868	2.5±0.28868
T_2	2.5±0.28868	2.0±0.28868
T_3	2.0±0.28868	2.0±0.28868
Average	**2.1667**	**2.1667**

**CW – coconut wood; CS – coconut shell*

Table 3 above show that the wood vinegar extracted from both coconut wood and coconut shell has a highly acidic pH. This acidity can be attributed to the presence of acetic acid and other pyroglinous acids that are products of destructive distillation of wood. This result is highly off the standard pH of wood vinegar which is between 2.5 to 3.0.

Acid Content

Acid content is the amount of acid that is present in the wood vinegar. It pertains to all acidic substances other than acetic acid which is one of the main chemical substances present in wood vinegar. According to Phywe (2017), acetic acid is approximately 6% of the total acid present in all wood vinegar samples. Acid content is herein shown below:

Table 4: Total Acid Content (TAC) of extracted wood vinegar from the two (2) samples

	CW Wood Vinegar (% by weight)	CS Wood Vinegar (% by weight)
T_1	6.47±0.21385	6.98±0.18520
T_2	6.61±0.21385	6.63±0.18520
T_3	6.89±0.21385	6.91±0.18520
Average	**6.6567**	**6.8400**

Average wood vinegar TAC was between 6.65 and 6.84 percent by weight as calculated at pH 8.15 using 0.1 N Sodium Hydroxide as titrant. This indicates that acid content of both wood vinegars from coconut wood and shell are slightly higher than the expected TAC present in most wood vinegars which is 6% by weight.

4.1 Soluble Tar Content

Soluble tar is the number of organic tar dissolved in the wood vinegar matrix. Soluble tar is herein tabulated in the subsequent table:

Table 5: Total Soluble Tar (TST) of extracted wood vinegar from the two (2) samples

	CW Wood Vinegar (% by weight)	**CS Wood Vinegar** (% by weight)
T_1	0.28±0.01528	0.31±0.01155
T_2	0.30±0.01528	0.33±0.01155
T_3	0.27±0.01528	0.31±0.01155
Average	**0.2833**	**0.3167**

Total Soluble Tar (TST) is the amount of soluble tar that is dissolved in the wood vinegar. It can be observed that the two samples of wood vinegars contain small amounts of soluble tar which is an indication of the degree of purity of the extracted wood vinegar. Also, this result coincides with the Japanese standard that dissolved tar content should not be more than 3% (Wada, 1997).

4.2 Water Content

Water content is the amount of moisture or water molecules present in the wood vinegar samples. The water content of wood vinegar samples from coconut wood and coconut shell are herein tabulated:

Table 6: Water Content of extracted wood vinegar from the two (2) samples

	CW Wood Vinegar (% by weight)	CS Wood Vinegar (% by weight)
T_1	80.2±0.45826	90.3±0.20000
T_2	80.8±0.45826	89.9±0.20000
T_3	81.1±0.45826	90.1±0.20000
Average	**80.7**	**90.1**

It was observed that the coconut shell has higher water content than coconut wood. This result is higher than two commercially produced wood vinegar which possesses less water content at a range of 80.49 and 79.18%, respectively (Theapparat, Yongyuth & Chandumpai, Ausa & Leelasuphakul, Wichitra & Laemsak, Nikhom & Ponglimanont, Chanita., 2014).

4.3 Basic and Neutral substances

Other substances were also found present in the wood vinegar. Here are the some of the many chemical compounds found in the two (2) samples:

Table 7: Other Substances found in Wood Vinegar Samples

	Chemical Compounds	% Concentration on CW WV	% Concentration on CS WV
1	Alkyl aryl ether	34.045	32.665
2	Phenol	30.224	28.799
3	Nitro-2-methyl-2-butane	10.006	11.120
4	2-methyl propyl ester butanoic acid	7.988	9.098
5	2-methoxyphenol	6.879	5.006
6	2,6-dimethoxyphenol	6.541	7.074

Percent concentration is the total concentration of other substances in the remaining 4-5 % of the unknown compounds present in the wood vinegar samples. Other trace chemicals are also found such as 9-octadecenoic acid (Z)-tetradecyl ester-(Oleic acid, tetradecyl ester)-$C_{32}H_{62}O_2$, 2-lauro-1,3-docecoin-$C_{35}H_{66}O_6$, dedecanoid acid, 1,2,3-propanetriyl ester (glyceryl tridodecanoate) -$C_{39}H_{74}O_6$, octanoic acid, Syringol, 1-(4-hydroxy-3,5-dimethoxyphenyl)-ethanol and 1-(4-hydrox y)-4-hydroxytoluene. These chemicals can be further utilized for agriculture, aquaculture, soil fertilizer, antimicrobial, antioxidant, flavoring, latex coagulant, sheet additive, wood preservative, pesticide, plant growth enhancer, antifungal, feedstuff and even repellent or insecticide.

V. CONCLUSIONS

Based on the results of the study, the following conclusions are herein drawn:

1. Clear to dark orange is the color of wood vinegar extracted from coconut wood, while yellowish to dark yellow orange was observes on wood vinegar from coconut shell, both have a copra-like odor as perceived by most of the respondents.
2. High pH was observed on both wood vinegar samples and its miscibility was observed to be high on most polar solvents, but is immiscible to non-polar solvents.
3. Slightly high TAC was recorded on both the wood vinegar samples which are slightly higher than the allowable percentage for wood vinegar which is 6%.
4. In terms of soluble tar, a very low percentage was observed on both the samples which are <1% which coincides with the Japanese standard that dissolved tar content should not be more than 3%.
5. For water content, wood vinegar from coconut shell has a higher percentage presence than wood vinegar from coconut wood at 80.7 and 90.1 percent, respectively indicating a high percent of moisture presence in the extracted wood vinegar samples.
6. In terms of basic and neutral substances, Alkyl aryl ether, Phenol, Nitro-2-methyl- 2-butane, 2-methyl propyl ester butanoic acid, 2-methoxyphenol, 2,6-dimethoxyphenol were the most common substances found in the two samples with varying percentage composition, also other trace chemicals were observed using GC-MS analysis.

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