

# Characterization compound chemical from cocoa waste as acetic acid and phenol

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## Characterization compound chemical from cocoa waste as acetic acid and phenol

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**Abstract.** Potential Cocoa bean and derivative products consist compound of polyphenols and different potential levels of antioxidants. The greater content of polyphenols provides benefits to human health, cosmetic and food functional. Pyrolysis compounds under go decomposition into lignin, hemicellulose, and cellulose in the biomass waste that produce liquid smoke, tar, charcoal and other. The results of produce liquid smoke, tar and charcoal. Analysis of the lignin content of the cocoa pod husks from Barru District was 44.98%, cellulose was 60.03%, hemicellulose 15.54%, while the lignin content analysis of cocoa pod husks from Sidrap was 43.17%, cellulose was 43.26%, hemicellulose is 3.13%, and other is 31.3%. At a pyrolysis temperature of 514 °C, the acetic acid content was 8.94% and the phenol content was 1.23% for the liquid smoke of the fruit skins of Barru Regency, while the acetic acid content was 4.37% and the phenol content was 9.34% for liquid smoke Sidrap Regency, From the separation of chemical compounds from the liquid smoke of cocoa pod skin in Barru Regency, it turns out that the highest content of acetic acid is 42.61% and phenol by 5.56% compared to other chemical compounds. From the separation of chemical compounds from the liquid smoke of the cocoa pods in Sidrap Regency, it turned out that the highest content of acetic acid was 42.51% and phenol by 5.54% compared to other chemical compounds.

### 1. Introduction

Potential biomass waste to be used as liquid smoke has gained attention in recent years, which is produced by pyrolysis process. Pyrolysis compounds under go decomposition into lignin, hemicellulose, and cellulose in the biomass waste that produce liquid smoke, tar, charcoal and other. Differences in the composition of the components of the raw material is expected to affect the composition chemical and type of compound pyrolysis results.

Biomass waste content in cellulose, hemicellulose and lignin and the pyrolysis conditions are the primary factors that pyrolysis reactions and resulting products. Several typical wood biomass contains 25%–35% hemicelluloses, 40%–50% cellulose, and 10%–40% lignin [1]. The pyrolysis process can be conducted at low temperature (300 °C–450 °C), medium temperature (450 °C–600 °C) or high temperature (>600 °C) [2]. Thermochemical processes pyrolysis is considered as the most promising and important technology for liquid smoke and gaseous fuels and also solid char. Observed in Pyrolysis technology for biomass can be used for various products including electricity, chemicals, fertilizers, and

bio charcoal [3]. In addition, the pyrolysis technology also can be divided into moving bed, fluidised bed, rotary kiln and fixed bed pyrolysis processes according to different pyrolysis devices[4].

Cocoa pod husk represents between 40 to 75% of the whole cocoa fruit weight where each ton of cocoa fruit will produce between 700 to 750 kg of cocoa pod husks (5). Many concerns point out to the need to use of renewable feedstock, composting, fertilizer, and replacing as much as possible the fossil fuels; among them could be mentioned the depletion of fossil oil reserves, constant uncertainties as far as price is concerned, unsecured supplies, and environmental pollution [6]. this research will use two types of pyrolysis of waste biomass derived from cacao shell district Barru and Sidrap Testing of physical and chemical properties of cacao waste determine compression test and depend ability and long burning. The main objectives of this research were (1) to get the yield of liquid smoke and charcoal on pyrolysis process, (2) identification of the fractions of potential chemical components of liquid smoke cacao shell district Barru and Sidrap (3). Determine the characterization of the morphological structure of the cocoa pod husk from Barru and Sidrap.

## 2. Methods

### 2.1. Manufacture of Liquid Smoke

Samples consisting of cacao shell district Barru and Sidrap put into the kiln is made of stainless steel which is equipped, Burning carried out at a temperature of until 515°C for 5 hours for two sample. Increase in temperature of tero smoke issued again. Liquid smoke or ter separated from the condensate by precipitation for 24 hours. Analysis was conducted on the liquid smoke yield (% w/ w), calor value, and acetic acid levels.

### 2.2. Identification of Chemical Compounds Liquid Smoke

Chemical compounds of each fraction liquid smoke temperature in the identification using GC-MS (Gas Chromatography Mass Spectrometry), Further chemical constituents were identified by GC-MS a length of 50 m and a diameter of 0. Of 125°C, and gas flow rate of 0.6 mL/min and injection volume of 0.2 mL. Analysis GC-MS results of the chemical components of the calculation in the form of acetic acid concentration of each fraction liquid smoke.

### 2.3. Characterization

The surface morphology of the charcoal cocoa shell were visualized in a JEOL JSM-6380LA analytical Scanning Electron Microscopy (SEM). The materials were treated with 8% NaOH solubility to remove impurities and placed in vacuum dry oven at 60°C for overnight. A small piece of specimen (0.5 cm<sup>2</sup>) was placed into double-sided tape of the specimen stub. The specimen was lightly pressed by release paper and coated with a thin layer of gold-palladium film before submitted to SEM for visualization the surface morphology characteristic of the charcoal cocoa shell

## 3. Result and Dissucion

### 3.1. Identification of compound chemical

Analysis of the lignin content of the cocoa shell from Barru Regency was 44.98%, cellulose was 60.03%, hemicellulose 15.54%, while the lignin content analysis of cocoa shell from Sidrap Regency was 43.17%, cellulose was 43.26%, hemicelluloid is 3.13%, and other is 31.3%. At a pyrolysis temperature of 514 °C, the acetic acid content was 8.94% and the phenol content was 1.23% for the liquid smoke of the fruit skins of Barru Regency, while the acetic acid content was 4.37% and the phenol content was 9.34% for liquid smoke of the fruit skins of Sidrap Regency.

**Table 1.** Ratio acetic acid and phenol cacao vinegar shell District Barru and Sidrap

No	Type Sample	Acid Acetic Ratio (%)	Phenol Ratio (%)
1.	Cacao Vinegar Shell District Barru	8,94	1,23
2.	Cacao Vinegar Shell District Sidrap	4,37	9,34

Lignin content depends on the different types of materials separation processes raw material also performed to determine the acetic acid compounds that have the potential as a natural preservative. Identification of the phenolic compounds, acids, esters, ketones, alcohols, furans and so on, then the separation process is carried out to determine the furfural compounds, phenol and toluene potential as a renewable bioenergy. Results of this study are supported by [7].

Analysis GC MS for liquid smoke for Cacao waste Distric Barru at temperature 314° C (Table 2) as 2-Octylamine, n-Butane, Acetic acid, Pyridine, 2-Methylpyridine, 1,3-Butadiene, 2-methyl-, 2(3H)-Furanone, dihydro- (CAS) Butyrolactone, Carbamic acid, phenyl ester, Phenol, 2-methoxy, n-Pentanal, 2(3H)-Furanone, dihydro- (CAS) Butyrolactone, 7-Oxabicyclo[4.1.0]heptane (CAS) Cyclohexene oxide, Pyrrolidine, 1-nitro-, Phenol, 2,6-dimethoxy- HEXA-2,4-DIYNE-1,6-DIOL, NERYL ETONE, 1,6-ANHYDRO-BETA-D-GLUCOPYRANOSE, (LEVOGLUCOSAN), 3-Methyl-anti-tricyclo[4.3.1.1]undec-3-en-10-one, Hi-oleic safflower oil (CAS) Safflower oil, 4-N-BUTYL-1,3-CYCLOPENTANEDIO, 1,5-Heptadiene, 3,6-dimethyl-3-(methyl-d)- (CAS) Androst-1-en-3-one, 17-hydroxy-1-methyl-, (5.alpha.,17.beta.). From the separation of chemical compounds from the liquid smoke of cocoa pod skin in Barru Regency, it turns out that the highest content of acetic acid is 42.61% and phenol by 5.56% compared to other chemical compounds.

**Table 2.** Analysis GC MS for liquid smoke for Cacao shell Distric Barru at 314° C

Peak#	Retention Time	Area	Concentration (%)	Chemical Composition
1	6.617	18163319	1.07	Acetic acid (CAS) Ethylic acid
2	7.658	45216014	2.66	Acetic acid (CAS) Ethylic acid
3	8.017	17956873	1.06	Acetic acid (CAS) Ethylic acid
4	8.206	37695435	2.22	Acetic acid (CAS) Ethylic acid
5	12.225	16238303	0.96	2(3H)-Furanone, dihydro- (CAS) Butyrolactone
6	12.793	15141929	0.89	3-CYCLOPROPENOIC ACID, 1-BUTYL, METHYL ESTER
7	13.064	94920528	5.59	(E)-Hex-2-en-4ynal
8	13.605	24175493	1.42	4-Heptenal, (Z)- (CAS) cis-4-Hepten-1-al
9	14.002	42598268	2.51	Phenol, 2-methoxy- (CAS) Guaiacol
10	14.506	175003465	10.30	1-Hexyn-3-ol (CAS) 1-Hexyne-3-ol
11	14.977	112564724	6.62	Lauric acid, 2,3-bis(trimethylsiloxy)propyl ester (CAS) 1-DODECANOYL, BIS
12	15.789	88410895	5.20	2-BUTYN-1-OL, 4-METHOXY-
13	16.081	64942268	3.82	2-Propanone, methyl-2-propynylhydrazone (CAS)

14	16.492	34608980	2.04	Pentane, 2-bromo- (CAS) 2-Bromopentane
15	16.821	94553480	5.56	Phenol, 2,6-dimethoxy- (CAS) 2,6-methoxyphenol
16	17.166	111291238	6.55	HEXA-2,4-DIYNE-1,6-DIOL
17	17.542	22372917	1.32	2(3H)-Benzofuranone, hexahydro-3-methylene- (CAS) 6-HYDROXYCYCLOH
18	17.613	25634489	1.51	2,5-Dimethoxybenzyl alcohol
19	17.870	94774550	5.58	
20	18.067	80071755	4.71	Acetic acid, pentyl ester (CAS) n-Amyl acetate
21	18.527	58010948	3.41	
22	19.093	203539120	11.98	1,2,3,4-Cyclopentanetetrol, (1.alpha.,2.beta.,3.beta.,4.alpha.)- (CAS) 1,2,3,4-
23	19.417	74189200	4.37	2,5-Hexanedione
24	19.992	13452952	0.79	2-Hexen-1-ol, 2-ethyl- (CAS) 2-Ethyl-2-xen-1-ol
25	20.167	18928815	1.11	Ethanone, 1-(4-hydroxy-3,5-methoxyphenyl)- (CAS) Acetosyringone
26	20.333	28105086	1.65	1-Azabicyclo[2.2.2]oct-2-ene-3-carboxylic acid, methyl ester (CAS) METHYL .
27	20.675	24531907	1.44	1,3-Cyclopentanedione, 4-butyl- (CAS) 4-N-BUTYL-1,3-CYCLOPENTANEDIO
28	21.017	12549992	0.74	
29	21.403	31255266	1.84	2-OCTENE, 1-BROMO-1,1,3-TRIFLUORO-
30	22.102	18594900	1.09	1-Cyano-2-methylallyl alcohol
		1699493109	100.00	

Table 3. Analysis GC MS for liquid smoke for Cacao shell Distric Sidrap at temperature 314° C

Peak#	Retention Time	Area	Concentration (%)	Chemical Composition
1	3.869	490990	0.11	Ammonium bicarbonate )
2	6.499	47548787	10.21	Acetic acid (CAS) Ethylic acid
3	6.692	14587013	3.13	Acetic acid (CAS) Ethylic acid
4	7.615	198078746	42.51	Acetic acid (CAS) Ethylic acid
5	8.908	9201128	1.97	3(2H)-Pyridazinone, 4,5-dihydro-6-methyl- (CAS) 2,3,4,5-TETRAHYDRO-6-M
6	10.408	4975307	1.07	2-Cyclopenten-1-one (CAS) Cyclopentenone
7	11.208	2158140	0.46	2,4-PENTADIENOIC ACID
8	11.486	4903776	1.05	4-VINYL-4,5-DIHYDRO-3H-PYRAZOLE

9	11.708	2702270	0.58	Ethanone, 1-(2-furanyl)- (CAS) 2-Acetylfuran
10	12.146	14194847	3.05	Cyclopropane (CAS) Trimethylene
11	12.508	5054585	1.08	2-Oxetanone, 4,4-dimethyl- (CAS) .BETA.,.BETA.-DIMETHYLPROPIOLACTO
12	12.845	8569833	1.84	2,4-Hexadienal (CAS) Sorbaldehyde
13	13.349	29211974	6.27	(E)-Hex-2-en-4ynal
14	14.108	21143156	4.54	Phenol, 2-methoxy- (CAS) Guaiacol
15	14.719	40116342	8.61	Butanal, 3-methyl- (CAS) 3-Methylbutanal
16	15.486	12923567	2.77	9-Oxabicyclo[6.1.0]nonan-4-one (CAS) 4-Oxo-9-oxabicyclo[6.1.0]nonane
17	15.914	6773916	1.45	Pentylamine, N-isopentyl-N-nitroso- (CAS) PENTYLISOPENTYLNITROSAMI
18	16.142	6968544	1.50	2,5-Dimethoxytoluene
19	16.708	4140556	0.89	Ethanone, 1-(2-ethyl-5-ethynyl-4-methyl-1,3,2-dioxaborolan-4-yl)- (CAS) 1,3,2
20	16.977	10382591	2.23	Phenol, 2,6-dimethoxy- (CAS) 2,6-Dimethoxyphenol
21	18.355	3175467	0.68	5.alpha.-Androstan-16-one, cyclic ethylene mercaptole (CAS)
22	19.069	9241873	1.98	Ethane, 1,1,1-triethoxy- (CAS) Triethyl orthoacetate
23	21.239	2230451	0.48	
24	21.637	2461221	0.53	2-Cyclopenten-1-one, 4-hydroxy-3-methyl-2-(2-propenyl)- (CAS) Allethrolone
25	22.274	4674730	1.00	Propanoyl chloride, 2-methyl- (CAS) Isobutyryl chloride
		465909810	100.00	

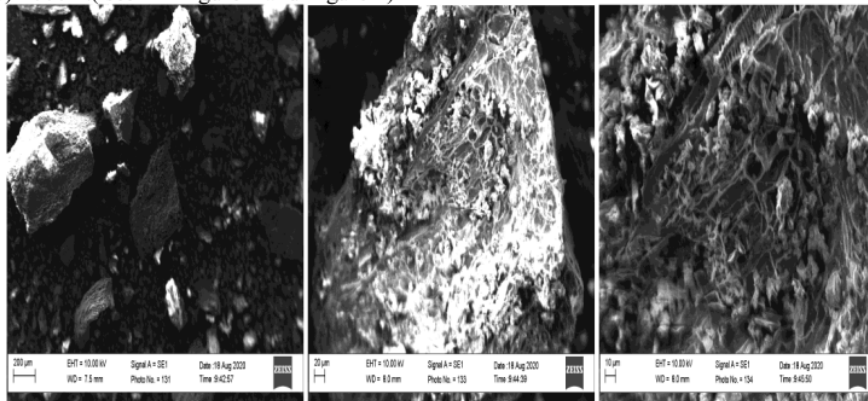
Analysis GC MS for liquid smoke for Cacao waste Distric Sidrap at temperature 314 C.(Table 3) as Ammonium bicarbonate, Acetic acid. , 2,3,4,5-TETRAHYDRO-6-M, 2- Cyclopentenone, 2,4-PENTADIENOIC ACID 4-VINYL-4,5-DIHYDRO-3H-PYRAZOLE, Ethanone, 1-(2-furanyl)- (CAS) 2-Acetylfuran Cyclopropane, 2-Oxetanone, 4,4-dimethyl- (CAS) .BETA.,.BETA.-DIMETHYLPROPIOLACTO, Sorbaldehyde, (E)-Hex-2-en-4ynal, Phenol, 2-methoxy- 3-Methylbutanal 9-Oxabicyclo [6.1.0]nonan-4-one Pentylamine, N-isopentyl-N-nitroso- (CAS), PENTYLISOPENTYLNITROSAMI 2,5-Dimethoxytoluene Ethanone, 1-(2-ethyl-5-ethynyl-4-methyl-1,3,2-dioxaborolan-4-yl)- (CAS) 1,3,2, Phenol, 2,6-dimethoxy- (CAS) 2,6-Dimethoxyphenol 5.alpha.-Androstan-16-one, cyclic ethylene mercaptole (CAS), Ethane, Triethyl orthoacetate, 2-Cyclopenten-1-one, 4-hydroxy-3-methyl-2-(2-propenyl)-, Propanoyl chloride, 2-methyl. From the separation of chemical compounds from the liquid smoke of the cocoa pods in Sidrap Regency, it turned out that the highest content of acetic acid was 42.51% and phenol by 5.54% compared to other chemical compounds.

Bioactive chemical compounds produced from pyrolysis of banana peel waste produce liquid smoke and banana waste tar are acids (68.6%), alcohol (62.5%), ketones (27.6%), phenols (25.7%) and furan (21.8%). [8]. The research other was alcohol, phenol, and furan compounds are products that are used in the production of biofuels and chemicals that have added value in the pyrolysis process. Bioactive

chemical compounds derived from liquid smoke from pine wood waste are acetic acid, butyrolactone, methyl-2-furfural, phenols and their derivatives. [9]. While bioactive chemical compounds derived from liquid smoke from cocoa waste are n-Amyl acetate, Resorcin, Levoglucosan functions as bio fuel. This is supported by other studies, that pyrolysis of corn cobs with a hot carrier at a temperature of 430-620 ° C, gives a maximum bio-oil yield of 14.24% at a temperature of 510 ° C. [10]. From these two liquid smoke resulted from pyrolysis of raw materials, that gave the highest yield of liquid smoke was liquid smoke of pine wood sawdust by 49.60% and teak wood sawdust 43.78%. [11].

### 3.2. Characterization

While SEM analysis for charcoal cacao shell district Barru and Sidrap showed the surface morphology analysis was carried out by Scanning Electron Microscopy (SEM). This is supported by research while SEM analysis for charcoal cacao shell district Barru and Sidrap with magnification (a) 100 x (b) 250x dan (c) 1000 x. ( seen in Figure 1 and Figure 2)

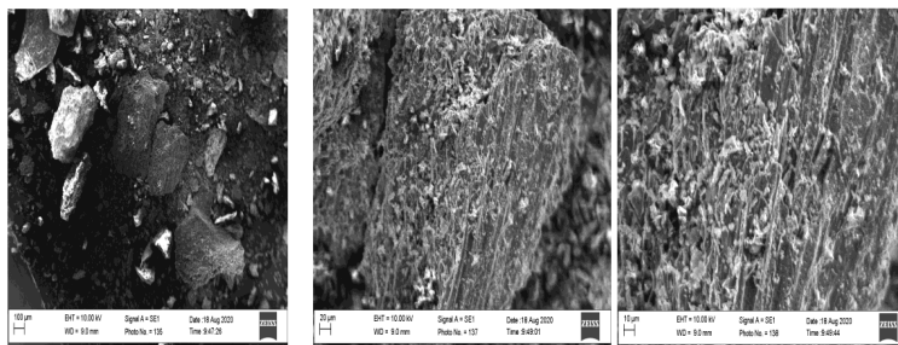


**Figure 1.** While SEM analysis for charcoal cacao shell district Barru with magnification (a) 100 x (b) 250x and (c) 1000 x.

Figure 1 and 2 display the SEM images of charcoal cacao shell district Barru observed under different magnification level. The study on surface morphology was significant in understanding the distribution and fibre arrangement of the charcoal Cacao wastes. The surface morphology of charcoal Cacao wastes. shows major structure of micro pores (Figure 1 (a)) and consist of abundant and long fibres Figure 2 (b) and (c). Slight impurities indicate by the white speckles were present on the surface as seen in Figure 1. SEM images of charcoal cacao shell district inside as shown in Figure 2(b). Besides that, the presence of encrusting substances causes the fibre to have an irregular appearance as shown in Figure 1(c). In addition, Figure 1(c) and 2(c) shows fibrillar arrangements are linear.

Supported by research, Analysis scanning electron microscopy (SEM) was used to visualize the surface morphological of materials. In order to propose the suitability of the studied plant as alternative fibre resources in pulp and paper making, [12]. So [13]. stated that fibre surfaces have waxes and other encrusting substances such as pectin, hemicelluloses, and lignin that form a thick layer to protect the important substances, There are irregular void spaces between the fibrilles and enhance the strength properties of paper produced [14].





**Figure 2.** While SEM analysis for charcoal cacao shell district Sidrap magnification (a) 100 x (b) 250x and (c) 1000 x

#### 4. Conclusions

Based on the objectives and results of the research that has been done a number of conclusions as follows: Leather raw material for cacao shell district Barru analysis showed that the lignin content 44,98%, and so ,60,03% alpha cellulose and hemicelluloses content of 15,54%. and lignin content cacao shell district Sidrap 43,17%, so 43,26% alpha cellulose and hemicelluloses content of 3,13%. Identification of liquid smoke from cacao shell by GC-MS yield potential chemical components including products acetic acid and phenol as preservative natural. From the separation of chemical compounds from the liquid smoke of cocoa pod skin in Barru Regency, it turns out that the highest content of acetic acid is 42.61% and phenol by 5.56% compared to other chemical compounds. From these two liquid smoke resulted from pyrolysis of raw materials, that gave the highest yield of liquid smoke was liquid smoke of pine wood sawdust by 49.60% and teak wood sawdust 43.78%.

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