The Effect of Solvent for Extraction for Removing Nicotine on the Development of Charcoal Briquette from Waste of Tobacco Stem

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Abstract: Waste from stems can be utilized as alternative energy source by turning into a charcoal briquette. The nicotine content in the tobacco stem can be removed by several extraction processes using many types of solvent such as n-hexane, ethanol, kerosene and steam. The goal of this research was to develop charcoal briquette from waste of tobacco stem. The variables studied were type of solvent for extraction; temperature, time and pressure of pyrolysis, and pressure of briquetting. The indicators of the briquette quality were calorific value and gas emission resulting from the burning of the charcoal briquette. Our results showed charcoal briquette had a maximum calorific value at a pyrolysis temperature of 300°C and 90 min while the load for pressuring the briquette was 4 ton. The calorific maximum value was 5438.9 cal/g or 22.8 MJ/kg. The gas emissions consisted of 0.03% carbon monoxide, 2.7% carbon dioxide, 18% oxygen the rest of the gas were nitrogen.

1. Introduction

Indonesia has a predominantly rural economy based around agriculture and plantations. One important cash crop comes from tobacco plantations that supply the numerous cigarette industries in the country. In 1994 there were about 160,000 ha under tobacco plantation and the annual production was around 95,000 tons of tobacco leaves [1]. This tobacco production was used to produce 180 trillion cigarettes. There several negative points in the production of tobacco leave. One of them is the waste of stems of the tobacco plant. According to Haygreen and Bowyer [2] 25% of the total weight of tobacco is the stems, therefore, there will be around 24,000 tons of waste per year or around 70 tons per day. Currently this waste from the cigarette industries cannot be utilized well and only a small part of them are usually sold to farmers to be converted to be organic fertilizer. Some of them was used to produce pesticide by taking the nicotine out of the stem (nicotine content is around 2%).

In the tobacco stems, there is a carbon element bonded in the cellulose and it can be utilized as an energy source of solid fuel (see in Table 1) [2]. This waste can be directly burned as a solid fuel, however, this will create problem such storage and smoke from the combustion process. The goal of this research was to convert tobacco stem into charcoal briquette with initial treatment to reduce nicotine content using extraction with several types of solvents. The variables studied were temperature and time of pyrolysis, pressure of briquetting and CO and CO_2 emission.

Table 1. Chemical composition of the tobaco leave (% weight).

Composition	Leave	Stem	Total
Sugar (as dextrose)	0.25	0.06	0.21
Pectin (as calcium pectate)	8.25	15.31	9.77
Cellulose	12.09	18.18	13.28
Citric acid	7.27	2.41	6.31
Maleic acid	3.62	1.46	3.20
Oxalic acid	2.80	1.55	2.55
Protein	9.08	4.75	8.23
Amide and Amino acid	10.53	1.27	8.71
Nicotine	3.43	0.82	2.92
NO ₃	1.74	9.07	3.19
Total resin	6.14	1.44	5.20
Ash	17.83	28.96	20.01

The indicators of the briquette quality were calorific value and gas emission resulting from the burning of the charcoal briquette.

2. Experimental

2.1 Chemicals used in the process

Chemicals used in this research and obtained in the laboratory were: distilled water, benzoic acid, cuprum chloride, salt, red methyl indicator, calcium hydroxide, sodium carbonate, oxygen, and pyrogalol. The waste of tobacco leaves was from PD Tarumartani, CO Ltd., Yogyakarta (a local cigarette industry).

2.2 Extraction process

In the preparation stage, several extraction processes were done to remove the nicotine content in the waste of tobacco stems. The solvents used were ethanol, normal hexane and steam. Extraction was done by putting 1 kg of sample into an extractor which was connected into the solvent tank which contained 5 L solvent. The extraction was done at temperatures close to the boiling point of the solvent.

2.3 Pyrolysis and briquette development

Charcoal briquettes from tobacco stem can be developed with and without preliminary treatment to remove the nicotine content using extraction processes. Solvents used for the extraction were ethanol, kerosene, n-hexane and steam. After extraction, the tobacco stem was dried, ground and then screened through at mesh between 30 and 60. The powder then was put into an oven at temperatures between 90–105°C, for about one hour to remove the remaining water. After that the powder was put into a retort to be pirolyzed at temperature between 250–500°C. The pyrolysis time varied from 30 to 90 minutes. The products of pyrolysis were gas, liquid and solid. The solid was then removed from the reactor and pressurized up to 4 tons to form the charcoal briquette. Figure 1 shows the schematic diagram of pyrolysis process.



Figure 1. Equipment for pyrolysis experiment.

2.4 Analysis

Nicotine content

The content of nicotine in the waste of tobacco stem was assessed by planar chromatography and was done in the Gadjah Mada University Research Centre. Standards ($0.5 \ \mu g/\mu l$ in acetonitrile) and dichloromethane sample solutions were applied ($1.5-3 \ \mu l$ from 20 μl) into C18-TLC plates as well as C30-TLC plates. Chromatograms were developed to a distance of 7.5 cm in a horizontal chamber using acetonitrile-water (88+12,v/v) with the addition of sodium 1-octanesulfonate (50 mg/100 ml) as mobile phase.

Calorific value

The calorific value was measured using a Bomb Calorimeter and using the ASTM D 2015 procedure [7]. The procedure is as follows: first of all the bomb should be transferred to calorimeter making sure that the bomb is gas tight and connected to the firing circuit. Then place the cover, stirrer and thermometers in position. Start the stirrer and keep it operating throughout the procedure. After that adjust the jacket temperature and stir for at least 5 min before starting to read the temperatures. Lightly tap the thermometers before each reading to avoid errors due to the sticking of the mercury to the glass. The bucket temperature should be read at 1 min intervals until same bucket temperature as the initial calorimeter temperature and the wetted thermometer length or the scale reading (°C).

After the charge was fired, the cooling water flow rate to the jacket was manually manipulated to adjust its temperature to match that of the calorimeter during the period of rise. Controlling the jacket temperature is necessary to minimize heat loss from the calorimeter to the surrounding air.

The combined pieces of unburned firing wire were removed and measured and subtracted from the original length and then recorded as "wire consumed".

3. Results and Discussion

3.1 Extraction of nicotine content

The effect of solvents on extraction from tobacco stems are shown in Table 2. It is clear that ethanol is the best solvent and therefore for the whole experiment we used ethanol as the solvent for the extraction processes.

3.2 Pyrolysis process

In the pyrolysis process, there were three products, gas, liquid and solid. The solid product was then presurized to charcoal briquettes. From the preliminary research, it was found that increasing temperature and pyrolysis time decreased the solid product. This is caused by different mechanism of cracking material during the pyrolysis. The thermal decomposition of hydrocarbons involves a series of primary and secondary reactions leading to a complex mixture of products. Studies show that the distribution of pyrolysis products varies considerably with the pyrolysis conditions and the type of reactor used. A study by Tsai and Albright [6] reported that at least seven surface reactions occur during most industrial pyrolysis processes.

In the pyrolysis experiments, the condensate formed is in the range of 25 to 45 percent of the original weight of tobacco stems while the gas formed is in the range of 10 to 20 percent while the rest is the solid product. This study demonstrated that more than 36 components mainly alcohols such as methanol, ethanol and propanol were produced during the pyrolysis process. The experimental results also show that the condensate and gas formed are affected by the temperature in the reactor and the heating rate. Further studies show that the reaction rate of the decomposition process depends on temperature.

According to Nurmala [4], there are four steps in the carbonization processes of wood or biomass:

1. Firstly by heating the wood, the water in it is vaporized and cellulose decomposition occurs up to a temperature of 260°C. In this process, the distillate formed consists of acids and methanol.

2. At temperatures between 260–310°C; most of cellulose degrades intensively to form pyrolygeneous liquor (alcohol), gas and a small amount of tar which can be used as a preservative agent

3. At temperatures between 310–500 $^{\circ}\mathrm{C},$ lignin dissociates to form more tar

4. More uncondensable gases, especially hydrogen, were formed at temperature between 500–1000°C. In this step, the solid product (charcoal) has a high purity.

Table 2. Extraction of nicotine using various solvent.

	Materials	Nicotine content		
		Initial	After extraction	
		(%)	(%)	
1	Long tobacco stem	0.49		
	Solvent n-Heksan		0.06	
	Solvent ethanol		0.00	
	Solvent Kerosene		0.06	
	Steam		0.16	
2	Short tobacco stem	0.51		
	Solvent n-Heksan		0.11	
	Solvent etanol		0.03	
	Solvent Kerosene		0.06	
	Steam		0.32	

3.3 Calorific value of charcoal briquette

Calorific value is one primary indicator for the value of commercial fuel. As with other charcoal briquettes (mainly from wood); the charcoal briquettes made from waste of tobacco stems was also analyzed for its calorific value. From the calculation, the optimum calorific value obtained was 5,438.9 cal/g.

The effect of temperature and length of pyrolysis to the calorific value of charcoal briquette can be seen in Table 3 and Figure 2 and 3. From the table it can be seen that the calorific value increased when the temperature and length of pyrolysis was increased which is caused by the increase in the amount of carbon obtained. This result agrees with experiment done by Nurmala and Soeparno [4-5].

This result compares well with the calorific value of wood (4491.2 cal/g) or young age coal (1887.3 cal/g). However this value is still lower than the value of charcoal from wood which has a calorific value of 7047.3 cal/g). Table 4 shows the calorific value of some fuels.

Table 3. Calorific value of charcoal briquette from waste of tobacco stem.

Pressure (ton)	Pyrolysis at 250°C			Pyrolysis pada 300°C				
	Length of pyrolysis (minute)			Length of pyrolysis (minute)				
	0	30	60	90	0	30	60	90
4	3616.2	3940.8	4882.4	5199.3	3617.1	4404.8	5372.8	5438.9
6	3612.1	3885.2	4713.5	5027.9	3612.3	3903.8	5217.7	5112.1
8	3597.6	3665.8	4250.4	4605.5	3597.7	3761.3	4860.4	5165.4



Figure 2. Calorific value of charcoal briquette for pyrolysis at 250°C.



Figure 3. Calorific value of charcoal briquette for pyrolysis at 300°C.

Fable 4. Calorifi	c value of several	l types of fuel [3].
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Type of fuel	Calorific value (cal/g)
Dry wood	4491.2
Young coal (lignite)	1887.3
Coal	6999.5
Charcoal	7047.3
Crude oil	10008.2
Fuel oil	10224.6
Natural gas	9722.9

3.4 Gas emission testing

The testing of gas emissions resulting from the burning of charcoal briquettes can be seen in Table 5. It can be seen that the pyrolysis process affects the production of emission gases with lower values of emission achieved at higher pyrolysis temperature. The length of pyrolysis also affected the amount of gas emitted from the burning of briquette. Nevertheless both results show that the briquette can be used as a solid fuel since the amount of gases emitted are small enabling these types of briquette to be used as an alternative fuel.

Table 5. Results of gas emission test from burning the charcoal briquette, % volume.

Time (minute)	Temperature of pyrolysis					
	Gas CO		Gas CO ₂		Gas O ₂	
	250°C	300°C	250°C	300°C	250°C	300°C
0	0.09	0.09	2.0	2.0	18.7	18.7
30	0.08	0.08	2.1	2.2	18.5	18.4
60	0.06	0.04	2.2	2.5	18.6	18.3
90	0.04	0.03	2.4	2.7	19.0	18.0

4. Conclusion

The results of this investigation that the powder waste from tobacco stems can be used as a raw material for making charcoal briquette. The calorific values of the briquette and gas emissions from their burning depend strongly on the pyrolysis process.

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