Characteristics of Biochar from Jengkok Tobacco : The Effect of Quenching in Pyrolysis Process

by Ayu Chandra Kartika Fitri

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ABSTRACT: *The cigarette industry produces wastes from tobacco leaves and clove flowers which is called Jengkok. The wastes contain metal Arsenic (As), a toxic metalloid chemical element that is dangerous for the environment. The organic compounds in the wastes can be decomposed using pyrolysis, a chemical process at elevated temperatures in the absence of oxygen. The purpose of this study is to examine the chemical compositions and characteristics of biochar produced from quenching technology treatment in the pyrolysis process using a Rotary Kiln machine. The variables used were the temperatures ranging from 400, 450, 500, 550, 600°C, the duration from 30, 35, to 40 minutes, and water quenched temperature at 25°C. The Biochar water content and temperature produced were then observed. The results analysis included properties test, ultimate analysis, namely elemental testing using the SEM-EDX method and proximate analysis. This research found that water quenched treatment produces Biochar of Jengkok from tobacco is bases (pH. 9-10), and low bulk density. The specific characteristic is C-organic, N, and O has a large enough value, while the optimal processing time is 40 minutes at a temperature of 550 0C, a time of. Therefore, biochar from jengkok is very good for farming with acid soil conditions.*

Keywords: *biochar, jengkok, pyrolysis, quenching, SEM-EDX*

1. INTRODUCTION

The cigarette industry produces wastes from tobacco leaves and clove flowers, which is called Jengkok. The wastes contain metal Arsenic (As), a toxic metalloid chemical element that is dangerous for the environment. The use of Jengkok directly is considered inefficient and needs to be converted into biochar[1]. Biochar is a relatively stable carbon compound, much more stable than organic compounds that are not made up[2]. To reduce the harmful effects of these wastes and not endanger the natural environment, air, water, soil, and the social environment, then the right processing technology, pyrolysis, is needed. According to Paris O. et al. (2005), pyrolysis is a complex event in which organic compounds in biomass (such as carbon) are decomposed by heating using a little oxygen to produce solids (char) and liquid smoke by-products[3]. Likewise, Swithenbank et al. (2005) and Blasi (2008) stated that pyrolysis is a process of thermal degradation without oxygen in a vacuum or vacuum to produce solids (char), liquid (pyrolitic liquid), and gas (permanent gas) at temperatures between 400^0C–800^0C.[4,5]

Several studies have been conducted and shown that biochar yield is highly dependent on pyrolysis conditions such as temperature and heating time [6,7] and strongly influenced by the chemical, physical and biological properties of biomass [8–10]. Meanwhile, Demirbas, 2004 states that biochar characteristics are very dependent on raw materials and how they are made.[11] Oguntunde et al., 2004, emphasized that the pyrolysis process's raw materials and operational conditions included temperature and time, can affect the stability and nutrient content of biochar.[12] Furthermore, the higher pyrolysis temperature will increase the aromatic structure of biochar [13], so that biochar will have a high resistance to decomposition and demineralization[14], and amorphous structure[15]. Carbon in biochar is in aromatic compounds in which six oxygen atoms are bound in a ring without oxygen or hydrogen [14,15]. The atoms are covalently bonded to form flat hexagonal lattices, resembling carbon atoms' arrangement in graphite. Noting the previous studies and following the recommendation of Iskandar T. (2012), Aldobouni (2015), Fadhil, AB (2008), the process of curved pyrolysis need to be carried out at a temperature range of 500^0C with operational conditions using a closed system and taking place with the use of a little oxygen from the atmosphere.[16–18]

So the amount of CO_2 formed is a way smaller compared to those resulted from open burning. Moreover, at the end of the process, a Quenching process was carried out through a sudden cooling process by spraying 25 ^{0}C water into the resulting biochar. The Water Quenching treatment is intended to improve biochar characteristics so that it can have a wider surface and higher volume, micropores, density, macropores, and better absorb water. Therefore, converting jengkok into biochar can be considered as tool and optimally improve agricultural businesses.

2. EXPERIMENTAL

This research was conducted at PT. Gudang Garam Tbk Kediri by using a case study method. The characteristics of the biochar products were observed, analyzed, and descriptively reported. The primary data is obtained from the results of the production process. The pyrolysis processes were performed at different temperatures of 400^{0}C, 450^{0}C, 500^{0}C, 550^{0}C, 600^{0}C, and various processing time from 30 minutes, 35 minutes, to 40 minutes. Compounds testing was conducted at the Laboratory of PT Gudang Garam Tbk. Gempol and SEM-EDX analysis was performed at The State University of Malang Central Laboratory.

2.1 Materials

The material used was Jengkok Tobacco, with a moisture of 10% -15% and a weight of 500 kg per batch. The tool used was a pyrolysis machine equipped with Water Quenching.

2.2. Research procedure

The pyrolysis process began with the collection of raw materials for tobacco squeezing from cigarette production waste. The water content must not exceed 10% of the weight of the material. The carbonization process was then performed using a pyrolysis machine at different predetermined temperatures and processing durations.

A Rotary kiln was used to help the process with a Natural Gas heater placed in several zones to distribute the combustion process evenly. At the end of the process, a cooling water unit in the form of a spray water chamber was added, which functioned as water quenched. The variables used were the temperatures ranging from 400, 450, 500, 550, 600^{0}C, the duration from 30, 35, to 40 minutes, and water quenched temperature at 25^{0}C . The biochar water content and temperature produced were then observed. The results analysis included properties test, ultimate analysis, namely elemental testing using the SEM-EDX (Scanning Electron Microscopy-EDX) method and proximate analysis.

3. RESULTS AND DISCUSSION

Characteristics of Jengkok biochar

Initial observations were made with Bulk Density and pH tests to determine the Jengkok Biochar characteristics from the pyrolysis process, then Ultimate and Proximate analysis.

Table. 1. Bulk Density and pH Test Results on Jengkok Biochar

Temperature (^{0}C)	Time (minute)	with Quenching		without Quenching	
		Bulk Density (kg/m^3)	pH	Bulk Density (kg/m^3)	pH
400	30	0.44	8.93	0.52	9.84
	35	0.47	8.82	0.74	9.77
	40	0.49	8.29	0.47	9.79
450	30	0.43	9.47	0.42	9.84
	35	0.37	9.48	0.45	9.72
	40	0.41	9.72	0.46	9.61
500	30	0.41	9.11	0.69	9.57
	35	0.37	9.89	0.50	9.41
	40	0.42	9.98	0.60	8.93
550	30	0.37	9.38	0.51	9.51
	35	0.41	9.84	0.51	9.90
	40	0.34	10.23	0.59	9.83
600	30	0.42	10.31	0.52	8.90
	35	0.45	10.33	0.45	9.67
	40	0.37	10.41	0.50	9.82

In Table 1, it can be seen that the bulk density test results obtained are between 0.34 - 0.43 kg / m3. This value describes the presence of a solid layer in biochar, which contains organic and mineral materials. It is very influential in the formation of pores, water-binding power, and drainage properties. The small bulk density value will be better because the pores are increasing. It is perfect for microbes' growth, such as rhizobia and mycorrhizal, which are nitrogen-fixing microbes that are very useful for providing plant nutrition.

Meanwhile, the pH value of jengkok biochar tends to increase at higher temperatures. It occurs because of the water quenched treatment resulting in the washing of mineral ash in biochar. The nature of the raw material can also influence the pH value and the temperature of the pyrolysis process. Furthermore, the laboratory test results will be analyzed the correlation factor between bulk Density, pH to Quenching Temperature, Temperature, and Time.

Table 2. Correlation factors between Temperature, Time and Quenching Temperature on pH and Bulk Density

Variable		pH	Bulk Density
Temperature	Correlation Coefficient	0,392	-0,0134
	Sig. (2-tailed)	**0,032***	0,481
Time	Correlation Coefficient	0,234	-0,026
	Sig. (2-tailed)	0,214	0,891
Temperature Quenching	Correlation Coefficient	0,066	**-0,757**
	Sig. (2-tailed)	0,731	**0.000**

* significant at the 5% significance level

Analysis of the correlation factor between temperature and pH and Bulk Density obtained a significance value (sig) for temperature with a pH of 0.032. This value is less than 0.05, so it can be concluded that there is a significant relationship between temperature and pH. The correlation coefficient between temperature and pH is 0.392, which indicates that an increase will follow an increase in temperature in pH. The significance value between temperature and Bulk Density is 0.481. This value is more significant than 0.05, so the result of the correlation factor analysis concludes that there is no significant relationship between temperature and bulk density.

The analysis of the correlation factor between time and pH and bulk density gave a significance value of 0.214 and 0.891, respectively. The significance value is more significant than 0.05, so the conclusion obtained is that there is no significant relationship between time and pH and time with Bulk Density. This conclusion can also be defined that changes in time have no impact on pH and bulk density changes.

The correlation analysis results for the Quenching Temperature factor with pH and Bulk Density were -0.731 and 0.000, respectively. These results provide information that quenching temperature has a significant relationship with bulk density because the resulting significance value is less than 0.05. The correlation coefficient between Quenching Temperature and bulk density is -0.757. This value provides information that a decrease will follow an increase in quenching temperature in the bulk density value.

Furthermore, the element content test (Ultimate Analysis) using Scanning Electron Microscopy is equipped with reading results (SEM-EDX). This tool is used to analyze surfaces or layers that are about 20 μm thick from the surface. The image obtained shows the difference in chemical elements where the light color indicates the presence of a chemical element with a higher atomic number. In this study, examination and analysis of jengkok biochar were carried out at 3 point positions with the magnification of 20 μm, 50 μm, 100 μm. The reading result is 15 elements / chemical elements, but the displayed results are limited to five elements while the other ten elements have small values.

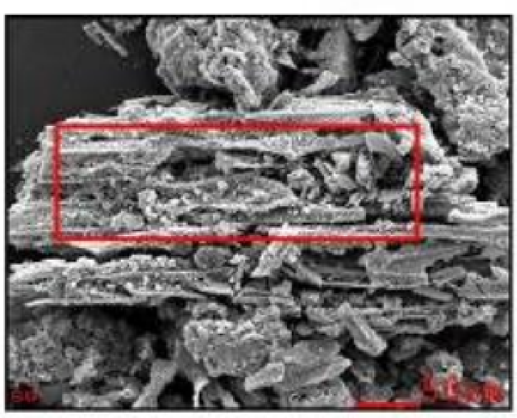

Figure 1.

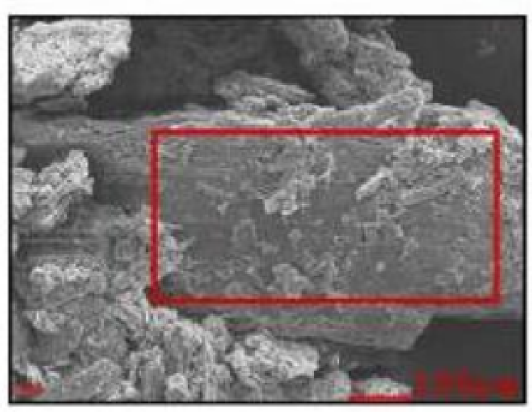

Figure 2.

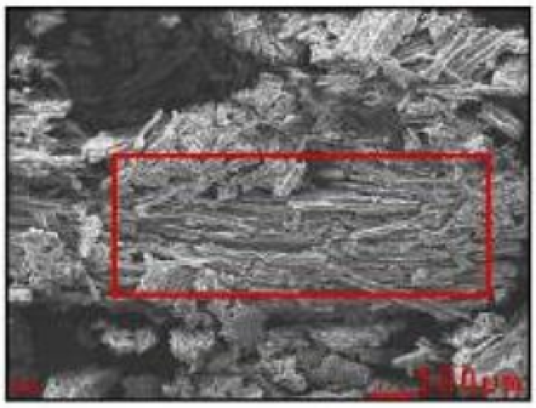

Figure 3.

In the three images, it can be seen that the difference in shape and level of pores of the jengkok biochar on the influence of temperature, processing time, and water quenched. This result could be due to the pyrolysis devolatilization process[19]. Visually, the morphological shape in Figure 1 has irregular pores, and in Figures 2 and 3, we can see that the surface is getting smoother. The addition of water quenched treatment to biochar can cause cracks in the biochar so that the biochar pores will open more so that they are more porous and neatly arranged (see Figure 3). The addition of water quenched treatment to biochar can remove impurities that cover biochar, such as Polycyclic Aromatic Hydrocarbon (PAH) compounds, and can be used to avoid condensation of PAH compounds [20]. The results of the reading from the figure are presented in table 3 below:

Table. 3. Results of the Ultimate analysis of jengkok biochar using the SEM-EDX method

Temperature (^{0}C)	Time (minute)	with Quenching					without Quenching				
		C	O	N	P	K	C	O	N	P	K
400	30	49.32	27.5	4.24	0.89	5.53	29.60	29.88	15.6	1.52	8.34
	35	46.02	28.08	5.46	1.06	4.9	40.13	32.67	10.55	0.94	5.41
	40	52.07	34.89	3.98	1.2	4.34	36.41	29.6	10.67	1.4	6.49
450	30	52.01	30.09	1.99	0.86	7.28	27.00	33.58	16.09	1.47	7.35
	35	47.77	27.89	3.54	1.06	11.40	47.04	29.28	4.76	0.78	7.26
	40	47.31	30.28	6.83	0.91	4.67	37.77	29.37	14.22	1.14	7.77
500	30	53.69	28.94	2.08	0.59	5.85	50.11	27.75	0	0.94	8.36
	35	54.22	28.71	0	1.21	5.94	33.22	29.86	15.81	0.82	5.32
	40	47.65	35.14	3.0	1.18	5.61	48.37	28.83	0	1.45	6.8
550	30	51.43	27.42	4.81	1.18	8.19	43.01	35.0	0	0.67	4.98
	35	47.07	34.66	7.31	1.46	6.57	40.02	34.74	3.81	0.73	6.34
	40	49.32	30.41	4.25	1.07	9.55	39.20	31.96	4.01	0.8	6.81
600	30	50.51	32.33	3.42	0.88	9.48	55.31	25.6	0.92	1.25	2.99
	35	43.54	31.27	1.74	0.74	9.38	45.53	27.18	4.19	0.78	5.56
	40	45.39	33.27	3.85	0.82	9.75	58.28	25.02	0	0.77	4.03

In table 3, the C-organic content from the pyrolysis process using water quenched has a minimum value of 43.54%, a maximum of 54.22%. Overall this value is quite high compared to biochar from other biomass. The high C-organic value is due to the influence of the pores formed. The organic C content that does not use water quenched treatment has a wide variation, namely 27.00% -58.28% with a lower average. While the percentage content of N is 0.0% -16.09%, P, 0.67% -1.52% and K; 4.03% - 8.36%. Compared with biochar without water quenched treatment, the jengkok biochar is relatively large due to the evaporation process's obstruction in the production process. As macronutrients or primary / major nutrients in the agricultural industry, the amount of content is relatively large. It can have a double function, namely as a repairer and to help plant vegetative growth.

Furthermore, to find out more about the effect of temperature, time, and temperature quenching on the elements in jengkok, the following statistical analysis results have been presented in table 4.

Table 4. Correlation factors between Temperature, Time and Quenching Temperature against C, O, N, P, K

Variable		C	O	N	P	K
Temperature	Correlation Coefficient	-0,192	0,125	-0,494	-0.253	0,173
	Sig. (2-tailed)	0,309	0,509	0,**006**	0177	0,361
Time	Correlation Coefficient	0,052	0,127	0,151	0,135	-0,108
	Sig. (2-tailed)	0,785	0,502	0,425	0,478	0,568
Temperature Quenching	Correlation Coefficient	0,624	0,37	-0,027	0,220	0,204
	Sig. (2-tailed)	**0,000**	**0,044**	0,887	0,243	0,279

* significant at the 5% significance level

Analysis of the correlation factor between temperature and C, O, N, P, K provides information that the N variable (whose significance value is less than 0.05) equal to 0.006. The conclusion is that there is a significant relationship between temperature and N. The correlation between temperature and variable N is -0.494. This correlation value is negative, which provides information that an increase in temperature will impact decreasing the value of N.

Analysis of the correlation factor for time with variables C, O, N, P, K each had a significance value greater than 0.05. This value indicates that there is no significant relationship between time and variables C, O, N, P, K. The correlation factor analysis results between temperature quenching and C, O, N, P, K in table 4 above provide information that the significance value between temperature quenching and variables C and O is 0.000 and 0.044, respectively. This significance value is smaller than 0.05, so the conclusion is that there is a significant relationship between temperature quenching with variables C and O. The correlation coefficient between temperature quenching and variable C is 0.624, and O is 0.37. This value is positive, so it can be concluded that temperature quenching affects the C and O variables' value.

Proximate analysis is also presented in the following table (table 5) to complement the characteristics of biochar from jengkok tobacco results from this study. Proximate analysis is performed to analyze the amount of moisture, ash content, volatile matter, fixed carbon in units of weight percent of material (wt%), and the biomass's calorific value.

Table 5. Results of proximate analysis of biochar against temperature and time

Temperature (^{0}C)	**Time (minute)**	**With Quenching**					**Without Quenching**				
		Moisture Content	Ash Content	Volatile Matter	Carbon Total	Calor (kcal/kg)	Moisture Content	Ash Content	Volatile Matter	Carbon Total	Calor (kcal/kg)
400	30	60.44	19.20	79.00	48.53	3,420	51.14	28.00	77.00	38.86	3,392
	35	61.24	18.80	80.00	46.98	3,377	62.74	23.60	79.00	34.99	2,949
	40	59.78	17.20	77.00	48.33	3,702	51.30	24.40	79.00	39.83	3,423
450	30	61.34	12.60	88.00	51.04	4,222	55.90	21.20	32.00	37.12	3,582
	35	53.49	15.40	88.00	50.46	4,281	52.70	23.00	82.00	44.08	3,486
	40	60.54	12.80	85.00	51.23	4,324	54.00	19.80	83.00	41.18	3,781
500	30	51.60	11.40	77.00	45.05	3,375	53.80	15.20	89.00	41.57	3,791
	35	54.29	18.60	85.00	47.75	3,939	58.20	23.00	84.00	36.73	3,062
	40	63.37	14.00	90.00	51.04	4,083	53.25	29.40	72.00	31.71	2,444
550	30	59.00	10.80	81.00	47.37	3,174	58.02	20.20	84.00	46.21	3,234
	35	60.70	20.20	84.00	48.14	2,993	52.75	29.00	74.00	41.76	3,118
	40	54.23	21.40	82.00	46.98	3,530	54.65	17.20	82.00	48.33	3,600
600	30	63.57	17.40	87.00	51.62	3,705	59.45	24.48	76.00	44.47	2,703

									10	
35	60.12	18.00	86.00	50.65	3,578	46.37	25.20	67.00	43.69	3,248
40	44.66	23.00	80.00	45.82	3,768	63.22	23.20	75.00	45.82	2,782

The following is a correlation factor analysis to ensure the effect of temperature, time, and quenching temperature on the proximate test results. (see table 6)

Table 6. Correlation factors between Temperature, Time and Quenching Temperature on Moisture Content, Ash Content, Volatile Matter, Total Carbon, and Calor

Variable		Moisture Content	Ash Content	Volatile Matter	Carbon Total	Calor
Temperature	Correlation Coefficient	0,003	0,091	0,026	0,168	-0,223
	Sig. (2-tailed)	0,989	0,631	0,892	0,376	0,236
Time	Correlation Coefficient	-0,066	0,163	-0,026	0,017	0,141
	Sig. (2-tailed)	0,729	0,39	0,892	0,931	0,456
Temperature Quenching	Correlation Coefficient	**0,35**	-0,659	0,444	0,782	0,451
	Sig. (2-tailed)	**0,050**	**0,000**	**0,014**	**0,000**	**0,012**

* significant at the 5% significance level

The temperature analysis results on the proximate test results produced a significant value more generous than the 5% level. By considering this significance value, there is no significant relationship between temperature and Moisture Content, Ash Content, Volatile Matter, Total Carbon, and Calor.

Likewise, the correlation factor between time and the proximate test results also resulted in a significance value more significant than the 5% level. So that the result of the correlation factor analysis is that there is no significant relationship.

In contrast to temperature and time variables, the correlation factor analysis between temperature quenching and Moisture Content, Ash Content, Volatile Matter, Total Carbon, and Calor were 0.050 each; 0,000; 0.014; 0,000; 0.012. This information concludes that there is a significant relationship between temperature quenching and the proximate test results.

4. CONCLUSION

Based on the test results and correlation factor analysis in this study, it shows that changes in the jengkok biochar production process's operational temperature only have a significant effect on the pH and N element values of the ultimate test results. While processing time does not affect the nature and character of the resulting jengkok biochar. The addition of water quenched treatment changed the pH value, bulk density, C, and O values. The correlation coefficient results indicated that an increase in Temperature Quenching would be followed by an increase in pH, the C / O value of the ultimate test results, and a decrease in the bulk density value. Meanwhile, the proximate test results are entirely influenced by temperature quenching. Thus, it is suggested that tobacco jengkok biochar production be good for the agricultural industry with acid soil conditions.

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