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The Effect of Pyrolysis Methods and Particle Size on Biochar Characteristics of Surian (*Toona ciliata*) as Ameliorant

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Abstract. Waste to biochar as a solution in organic farming applications and reduce CO_2 emissions on agricultural land. The study determined the effect of pyrolysis methods and particle size on biochar characteristics of Surian (*Toona ciliata)* wood waste as ameliorants. This research was carried out in two experimental steps. First, the effect of pyrolysis methods in biochar production, namely A = Kon-Tiki method; B = Drum method, and C = Soil-Pit method, and secondly the effect of particle size (Fig. 2) as follows: A = 2.80 – 4.74 mm; B = 2.00 – 2.80 mm, C = 1.00 – 2.00 mm, D = 0.50 – 1.00 mm, E = ≤ 0.50 mm, in each experiment using used a completely randomized design (CRD) with three replications. The first study showed that Kon-Tiki methods give a significant effect on the chemical properties of Surian wood waste biochar. The second study showed that particle size gives a significant effect on the chemical properties of Surian wood waste biochar, where, ≤ 0.5 mm is the best particle size for Surian wood waste biochar. It is recommended in the production of Surian wood waste biochar using the Kon-Tiki method and application at a particle size of ≤ 0.5 mm.

INTRODUCTION

The utilization of organic or wood waste has been widely carried out with the concept of pyrolysis to produce charcoal from waste to biochar (WTB) as a solution in organic farming applications and reduce CO_2 emissions on agricultural land. Pyrolysis is the term given to the thermal depolymerization of organic matter in the absence of oxygen [1].

Biochar is a material that is rich with carbon produced by biomass or organic waste pyrolysis at the range of temperature from 400 to 700^{0}C (slow, conventional, fast), forming predominantly recalcitrant and stable organic C structures in the soil [2]. Biochar may have heterogeneous properties depending on the characteristics and composition of the raw material and conditions of pyrolysis [3]. Organic waste carbonization process with various types of pyrolysis methods that will produce biochar in various sizes (Fig.1).

The properties of biochar produced from biomass via pyrolysis are highly dependent on many factors, including pyrolysis temperature, heating rate, type and composition of the feedstock, particle size, and reactor conditions [4]. The difference in the pyrolysis methods will affect the temperature produced during the pyrolysis process. This pyrolysis temperature will affect the physicochemical properties and structure of biochar such as elemental components, pore structure, surface area, and functional groups [5]. The particle size of biochar also plays an important role in determining not only the most suitable application for specific biochar products but also the best soil application method [6]. Small biochar particles interact more easily with soil particles to form aggregates than large ones [7]. In addition, a larger specific surface area per unit mass increases water retention (8) and water available to plants [9]. Biochar with a small size has more micro-pores than biochar with a large size. So the biochar

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with a small size can hold water more strongly and has more available water content than the large one. However, small particles can reduce the flow of saturated water in the soil by clogging the pore spaces [8].

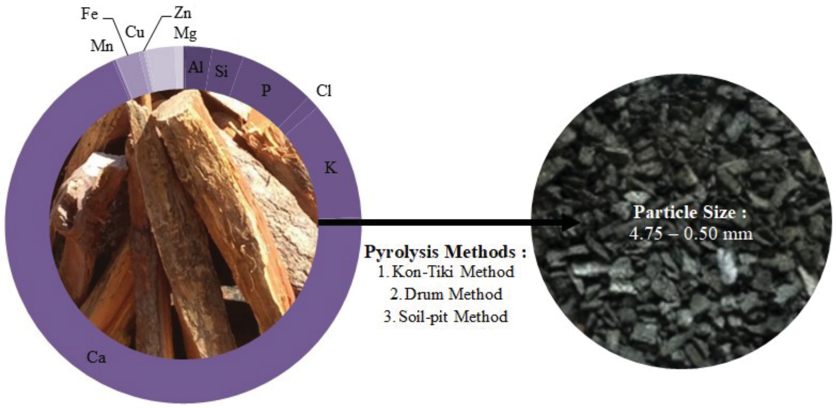

FIGURE 1. Wood waste to biochar (WTB) from Surian (*Toona ciliata*) as an ameliorant.

Surian (*Toona ciliata*) are known as Toon and Red cedar from the Meliaceae family. This wood is used for the Versaille timber like building houses and ships, and also for furniture. This processing will result in waste. This wood has holocellulose of 65.10-74.50% and lignin of 16.83-28.50% [10]. The tree and wood feedstocks usually contain huge lignin as compared with crop feedstocks (less lignin) and lignin is more stable thermally than hemicellulose and cellulose. Therefore, high lignin-content feedstocks are more able to form solid biochar [11]. So that we can use this wood waste as an ameliorant by converting it into biochar. This research studies the effect of different methods of production of biochar and particle size of biochar to be applied as a soil ameliorant. So, it is necessary to characterize the effect of the pyrolysis method and particle size on Surian (*Toona ciliata*) wood waste biochar.

MATERIALS AND METHODS

This research was carried out at the Laboratory of the Department of Soil Science, Faculty of Agriculture, Andalas University, and the Chemical Laboratory of Soil Research Institute, Bogor from April - August 2021.

Experimental Design

FIGURE 2. Morphology of biochar particle size from Surian (*Toona ciliata*) wood waste

This research was carried out in two experimental steps. First, the effect of pyrolysis methods in biochar production, namely A = Kon-Tiki method; B = Drum method, and C = Soil-Pit method, and secondly the effect of particle size (Fig. 2) as follows: A = 2.80 – 4.74 mm; B = 2.00 – 2.80 mm, C = 1.00 – 2.00 mm, D = 0.50 – 1.00 mm, E = ≤0.50 mm, in each experiment using used a completely randomized design (CRD) with three replications

Biochar Production

The preparation process for biochar production uses SW_w and is dried for one week in the Greenhouse of the Faculty of Agriculture, Andalas University until it reaches a moisture of 12.28%. Furthermore, the production process is carried out based on the method that has been carried out with three repetitions. The production process of SW_W biochar used as much as 10 kg with the specifications of the method is as follows: (a) the Kon-Tiki method is made of conical steel which has a top diameter of 100 cm, a height of 90 cm, a wall slope of 63.50^0, and a capacity of 827 liters; (b) the drum method is made from modified waste oil drums with a diameter of 58 cm, a height of 86 cm, and a capacity of 200 liters and (c) the Soil-Pit method is a conical hole dug in the ground which has a top diameter of 150 cm, a height of 90 cm, a wall slope of 63.50^0, and a capacity of 827 liters. The results of the production of biochar from each method are watered to stop the combustion process and then dried in a 40^0C oven for 2*24 hours with the aim of homogeneous biochar water content and separation of SW_W biochar particle size using Electromagnetic Sieve Shaker EMS-8 on a 4.75 – 0.50 mm sieve. The next step is to analyze the characteristics of biochar in the laboratory [12, 13, 14].

Characteristics Analysis of Biochar and Statistical Analysis

Biochar of SW_w with each method and each particle size was characterized based on a guidebook, namely Biochar: a guide to analytical methods e.g pH, EC, LP, proximate, CEC, and C In/Organic [15; 16]. The statistical analysis has carried the software SPSS 16, Statistix 8®, and Microsoft Exel 2016 to pyrolysis method and particle size on characteristics of biochar. It submitted to an analysis of variance [ANOVA] and If the F test> F table, then the treatment results show a significant effect at the 5% level [*] and a very significant effect at the 1% level [**] of Duncan's Test.

RESULTS AND DISCUSSION

Production of SW_W biochar within three methods of pyrolysis is shown in table 1. The pyrolysis methods do not give an effect on the production of SWw biochar.

TABLE 1. Production of biochar from SW_W

Pyrolysis Methods	Moisture	Dry Weight	Duration of Firing	Temperature	Moisture	Dry Weight	Yield Ratio
	Feedstock			Biochar			
	%	Kg	Minute	^{0}C	%	Kg	%
Kon-Tiki	12.28	10	28.3	525	45.58	3.57	35.67
Drum			31.7	522	43.70	3.23	32.33
Soil-Pit			31.7	523	56.12	3.12	31.20
CV (%)	-	-	18.61	4.86	32.18	11.15	
Duncan's Test	-	-	ns	ns	ns	ns	

CV = Coefficient of variation ** = Significant at the 0.01 level; * = Significant at the 0.05 level and ns = non-significant; n = 9.

Based on Table 1, it means the production of SW_w biochar isn't affected by the pyrolysis method. So that we can use any methods to produce the SW_w biochar. But Kontiki methods show the highest biochar yield with the faster duration of firing and the higher temperature. It may be caused by the specification and the concept of the Kontiki that makes the combustion spread well because of a more uniform temperature distribution. Kon-tiki has the highest biochar yield ratio, this is influenced by temperature and duration of combustion during production. The highest temperature gives a faster duration of firing. The lowest temperature gives a longer duration of firing. Also, high lignin tends to improve thermal resistance. It makes feedstocks have more lignin quantity may enhance biochar production [11].

The characteristic of pyrolysis methods and particle size of biochar from SW_w are shown in Table 2. and Table 3, respectively. From the table, we know that pyrolysis gives a very significant effect on biochar properties except for LP and Ash. And for the particle size, it gives a very significant effect on SW_w biochar. In general, it shows that Kontiki and size of ≤0.50 mm have a higher amount of the biochar characteristic. The highest pH on the pyrolysis effect was found in Drum Methods. This pH alkaline is caused by the carbonization process, acidic functional groups are removed and alkali metals and alkaline earth elements from salts are increased [17]. These salts include (i) readily soluble salts, (ii) carbonates, (iii) sparingly soluble metal oxides and hydroxides, and (iv) silicates, the latter especially when feedstocks contain soil particles [18, 19]. Biochars produced under high temperatures (>400°C) may have higher pH values than the low temperature (<400°C) biochars from the same raw materials [20, 21].

The increase in EC from the effect of pyrolysis methods and particle size, in general biochar EC values ranging from 0.04 dS m^{-1} [22] to 54.2 dS m^{-1} [23]. Similar to pH, the EC of biochar samples is also dependent on the feedstock and the pyrolysis temperature. Biochars produced at higher pyrolysis temperatures generally have higher EC values [24, 25, 26]. This effect has been attributed to the increasing concentration of residues or ash caused by the loss of volatile material during pyrolysis [26]. Differences in the EC of biochars produced using different feedstocks have been attributed to differences in their ash contents [25]. The ratio of biochar to water in the suspension affects the EC value, with EC values decreasing with increasing dilution. In samples with high soluble salt contents, the equilibration time also affects EC values, with longer equilibration times associated with higher EC values. It also looks the same against liming properties on biochars and allows their use as liming agents in acidic soils [21, 27, 28].

The highest moisture was found in Drum methods with 29.87%. Biochar produced at lower temperatures contained more moisture than at higher temperatures. At the initial stage, the slight mass loss was due to sorbed water volatilization. The minimum amount of water was not reabsorbed under the ambient situation as reported by reduced water loss with increasing the biochar generation temperature [11]. And for the highest of volatile matter found in the Kontiki method with 51.79%. Lower temperature pyrolysis reduces the volatile matter losses and strengthens the secondary char creating a pyrolysis mechanism [11]. The volatile matter fraction of biochar decreases primarily in response to the highest heating temperature during pyrolysis [29, 30, 31, 32, 33] and secondarily to time at that temperature [21] [33]. The heating rate also plays a role. Fixed carbon is, strictly speaking, not pure carbon but simply a dry mass that is not volatile matter nor ash, and therefore dominated by fused aromatic carbon structures. Concerning low ash feedstocks, fixed carbon content increases in response to a pyrolysis temperature. For high ash feedstocks, however, the fixed carbon content may decrease with temperature [30].

TABLE 2. Characteristics of pyrolysis methods of biochar from SWW

Pyrolysis Methods	pH H_2O	EC	LP	Proximate Analysis				CEC	C Inorganic	C Organic
				Moisture	Volatile Matter	Ash	Fixed Carbon			
	Unit	dS m^{-1}	% $CaCO_{3eq}$	%				mmol kg^{-1}	g kg^{-1}	
Kon-Tiki	9.43 a	0.53 a	6.38	29.20 a	51.79 a	14.73	37.06 a	523.55 a	0.06 b	9.25 b
Drum	9.51 a	0.47 b	5.58	29.87 a	47.89 b	22.08	25.81 b	195.47 c	0.13 a	11.67 a
Soil-Pit	8.70 b	0.28 c	6.40	21.87 b	32.06 c	20.39	11.67 c	317.60 b	0.07 b	6.90 c
CV (%)	1.54	3.65	7.73	2.53	2.36	17.80	12.35	10.98	13.76	5.39
Duncan's Test	**	**	ns	**	**	ns	**	**	**	**

EC= Electrical conductivity; LP = Liming potential; CEC = Cation exchange capacity; CV = Coefficient of variation; ** = Significant at the 0.01 level; * = Significant at the 0.05 level and ns = non-significant; n = 9.

Based on table 2., the highest CEC was found in the Kontiki Method with 523.55 mmol/kg and the lowest in Drum Method. Increasing CEC with an increase in temperature was associated with the augmentation in mineral nutrients, pH, and EC. The charge density of biochar and plenty of exchangeable ions in the aqueous phase mainly control the CEC. Thus, biochar's negatively charged sites augmented with the increase in biochar pH and allowed it to grip cationic bases via an electrostatic mechanism and raise their exchangeability with alternative ions in the soil. In addition, the quantity of nutrients is augmented in the aqueous phase with the rise in biochar EC and accordingly exchange capability of the biochar [11]. In biochars that are not compounded with added clay minerals, most negative charges are of the variable (pH-dependent) type. They develop because of the dissociation of protons from oxygenated functional groups at biochar surfaces. Since dissociation increases with increasing pH, biochar negative charge increases with increasing pH and, as a result, the CEC of biochar increases with increasing pH [34, 35]. Also, the decrease in CEC is influenced by the ash content. When the ash content is higher it causes the CEC lower due to the presence of the ash that clogs the biochar pore and reduces its surface area. We can see the drum method has the highest ash content (22.08%) so the CEC is the lowest.

Pyrolisis methods influence the amount of $C_{Inorganic}$ and $C_{Organic}$ on the SWw biochar. We can see that the highest amount of $C_{Inorganic}$ and $C_{Organic}$ on drum methods. This might indicate that duration and temperature affect the production biochar process. Inorganic carbon (C_{In}) is a common constituent of the ash fraction in biochar. It is mainly present in calcite ($CaCO_3$) and/or dolomite [$CaMg(CO_3)_2$] forms although other phases, such as kalicinite ($KHCO_3$), have also been detected in some biochars [36]. These salts can contribute both to the liming properties and the nutrient value of biochar [37].

The chemical properties of biochar particle size from SW_w as shown in Table 3. The highest pH and EC were found in the size of ≤0.50 mm with 9.50 units and 0.49 dS/m. The smaller particle use will spread more OH^- to increase the pH and more soluble ions in the water that make increasing EC values. If there is more OH^- there will be less H^+ which makes the pH higher. The EC speaks to the total quantity of water solvent ions that exist in biochar samples. When it is smaller in size, more ions are dissolved. It gives a higher EC value. It has disadvantageous effects on crop development (supplements instability and diminished water uptake) when existing in bigger concentration [11]. The biochar in this experiment showed comparatively minor EC (< 4.0 dS m^{-1}. Usually, ≥ 4,0 ds m^{-1} EC represents saline soil. Therefore, the small EC containing biochar could be applied in soil and they ought to not have a notable negative role on salinity. The highest LP was found in the size of ≤0.50 mm with 7.07%$CaCO_{3eq}$. The smallest particle that is used in this analysis will make more $CaCO_3$ equivalent. The role of biochar in acting as a liming agent for short-term effect was due to the presence of ash content and the long-term effect was due to its oxygen-containing functional groups. At the time of high temperature-induced biochar production, the basic cations (Ca^{2+} and Mg^{2+}) tend to convert into their oxides, hydroxides, and carbonates and thus accelerated the $CaCO_{3eq}$ of biochar [11].

TABLE 3. Chemical Properties of biochar particle size from SW_W

Particle Size (mm)	pH H_2O	EC	LP	Proximate Analysis				CEC	$C_{Inorganic}$	$C_{Organic}$
				Moisture	Volatile Matter	Ash	Fixed Carbon			
	Unit	dS m^{-1}	% $CaCO_{3eq}$	%				mmol kg^{-1}	g/kg	
4.75 - 2.80	8.98 c	0.37 d	5.21 d	33.33 a	52.40 a	10.59 b	41.81 a	244.44 d	0.05 c	2.94 e
2.80 - 2.00	9.09 c	0.40 cd	5.53 cd	29.17 b	47.03 b	15.33 b	31.69 b	308.36 c	0.08 b	7.43 d
2.00 - 1.00	9.17 bc	0.42 bc	6.17 bc	27.89 c	42.90 c	21.04 a	21.87 c	343.33 bc	0.09 b	9.83 c
1.00 - 0.50	9.32 ab	0.45 b	6.63 ab	24.61 d	40.42 c	22.86 a	17.56 c	377.78 b	0.10 b	12.24 b
≤0.50	9.50 a	0.49 a	7.07 a	19.89 e	36.84 d	25.53 a	11.31 d	453.78 a	0.12 a	13.93 a
CV (%)	1.33	4.32	6.92	2.01	3.39	14.42	12.01	9.64	13.76	5.39
Duncan's Test	**	**	**	**	**	**	**	**	**	**

EC= Electrical conductivity; LP = Liming potential; CEC = Cation exchange capacity; CV = Coefficient of variation; ** = Significant at the 0.01 level; * = Significant at the 0.05 level and ns = non-significant; n = 15.

The influence of particle size on ash content on SWw biochar of 25.53%. Preferential loss of organic over inorganic species during pyrolysis results in increasing ash content with increasing extent of pyrolysis. Ash content is largely determined by feedstock origin [30]. Additionally, the ash from waste streambiochars is likely to be enriched in certain elements that may create a nutrient imbalance e.g., Na [22]. The highest particle size on biochar will be an impact on increasing moisture content, volatile matter, and fixed carbon. Volatile matter decreases as the heating rate increases from 10°C to 50 °C min^{-1} [31, 32] but is comparatively higher for fast pyrolysis chars produced at near-instantaneous heating rates. The volatile matter has been used as a proxy for the readily mineralisable fraction of biochar. Analysis of 53 biochars from a wide range of feedstocks and production conditions demonstrated that volatile content per unit ash-free mass correlated reasonably well with H/C organic ratios [30]. The increase in FC with increasing size explains the catalytic effect of certain inorganic species on pyrolysis, which impedes the condensation of aromatic carbon. In conjunction with an understanding of feedstock properties, notably ash, the fixed carbon, volatile matter ratio can describe pyrolysis conditions where biochar is produced by unmonitored or uncontrolled technologies. Fixed carbon content may be used as an indicator of the carbon sequestration potential of biochar [30].

The highest of amount CEC and cation base on the size of 0.50 mm with 453.78 mmol/kg, the indirect effect of particle size on the majority of variable-charge hydroxyl groups at biochar surfaces belongs to organic acid functional moieties that can be broadly categorized into carboxylic, lactonic, and phenolic groups. Given that the distribution, concentration, and dissociation constants of such groups will differ from biochar to biochar, it can be expected that CEC–pH dependencies will be specific for particular biochar. By and large, most carboxylic acid groups have pKa values that fall between 2 and 4, such that they are largely dissociated at pH values above 5 [38]. Particle size relates to the surface area of a particle where the smaller the size of a particle gives a larger surface area of the particle. With the larger surface area, it means that there are more negative charges that it has, thus giving a higher CEC value.

Particle size also influences the amount of C inorganic and C organic. Whereas the highest amount of C inorganic and C organic on the size of ≤0.50 mm with 0.12g/kg and 13.93 g/kg, respectively. Carbonates in biochar can be derived from either: (a) CO_2 evolved from thermally decomposing C_{Org} during slow pyrolysis and trapped in the alkaline charred material; (b) carbonate-containing diluents mixed with the original biomass; and/or (c) carbonate present in the original biomass [39]. At temperatures greater than 600 ^oC carbonates decompose and a concomitant enrichment in sparingly soluble metal oxides occurs [40]. The total elemental analysis allows the determination of total C in biochar. When inorganic C is present, total C cannot be used as a proxy for organic C (C_{org}) and needs to be corrected accordingly [20]. Not doing so would lead to erroneous assessments of the condensed aromatic C fraction from the molar H/C ratio [41] and that of biochar C_{Org} storage value. Unless C_{Org} is determined directly by the amount of C_{In} in biochar is needed to determine C_{Org} from total C. The presence of carbonates in biochar, as well as that of other soluble salts, may also influence the determination of other properties, such as CEC, thus the knowledge of potential artifacts and how these can be overcome needs to be considered [42] [43]. Moreover, the dissolution of carbonate in biochars on incubation creates a pulse of CO_2 that causes an overestimation of short-term mineralized biochar-C [44, 45, 46].

CONCLUSIONS

The results of the first study showed that Kon-Tiki methods give a significant effect on chemical properties (EC of dS/m; VM of %FC of %and CEC of mmol/kg) of SW_W biochar. The second study showed that particle size gives a significant effect on the chemical properties of SW_W biochar, where, ≤ 0.5 mm is the best particle size (pH of 9.50 unit; EC of 0.49 dS/m; LP of 7.07 $\%CaCO_3$; ash of 25.53%; CEC of 453.78 mmol/kg; $C_{inorganic}$ of 0.12 g/kg; $C_{organic}$ of 13.93 g/kg) for SW_W biochar.

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