

STUDY ON THE PREPARATION OF BIOCHARS FROM SOME PLANT MATERIALS

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Abstract

This research concerns with the study on the preparation of biochar from some plant materials. Baw-za-gaing (*Leucaena leucocephala* (Lam.) De Wit), Kathit-su (*Zanthoxylum budrunga* Wall), Htiyo-wa (*Thyrsostachys siamensis* (Kurz ex Munro) Gamble), Pan-mezali (*Peltophorum pterocarpum* (DC.) Back. ex K.), and rice husk (*Oryza sativa* L.) were collected for preparation of biochars. Different amounts of biochar were 18.75 % from Baw-za-gaing, 10.0% from Kathit-su, 19.04% from Htiyo-wa, 10.0 % from Pan-mezali and 10.0 % from rice husk. In the biochar preparation process, the effectiveness of TLUD (Top-Lit UpDraft) furnace was studied by using various chimney height (1'-3'). The highest yield % of biochar from different plant materials was acquired with the chimney height in the range of 1' 6" - 2' 6". Among the five different samples, Htiyo-wa gave the highest yield % of biochar (19.04 %). Furthermore, ash content (1.2 % from baw-za-gaing, 0.13 % from htiyo-wa, 0.2 %, from kathit-su, 0.18 % from pan -mezali, and 0.62 % from rice husk), moisture content (2.65 % from baw-za-gaing, 1.75 % from htiyo-wa, 2.32 % from kathit-su, 0.18 % from pan-mezali, and 3.251 % from rice husk), and bulk density (84.78 g/100 mL from baw-za-gaing, 79.12 g/100 mL from kathit-su, 82.21 g/100 mL from pan-mezali, 80.12 g/100 mL from rice husk, and 75.12 g/100 mL from htiyo-wa) were also determined in the laboratory. Similarly, the volatile matter contents of biochars were determined 2.12%, 2.00%, 2.10 %, 2.50%, and 2.15%, respectively, from Pan-mezali, Baw-za-gaing, Htiyo-wa, Kathit-su, and Rice- husk .The fixed carbon contents were 95.775 % from Baw-zagaing, 98.72 % from Htiyo-wa, 97.23 % from Kathit-su, 97.718 % from Pan-mezali, and 96.015 % from Rice Husk. Nitrogen content (3.1% from baw-za-gaing, 2.5 % from kathit-su, 3.0 % from pan-mezali, 2.1 % from rice husk, and 2.4 % from htiyo-wa), potassium content (0.1814 % from baw-za-gaing, 0.8459 % from kathit-su, 0.8457 % from pan-mezali, 0.8471 % from rice husk, and 1.802 % from htiyo-wa) and phosphorous contents (0.032% from baw-za-gaing, 0.023 % from kathit-su, 0.026 % from pan-mezali, 0.018 % from rice husk, and 0.027 % from htiyo-wa) were observed in biochar, which can be used as soil amendment for plant growth.

Keywords: Biochar, TLUD furnace, ash contents, bulk density, fixed carbon

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Introduction

Biochar is the carbon-rich product when biomass (such as wood, manure or crop residues) is heated in a closed container with little or no available air. It can be used to improve agriculture and the environment in several ways, and its stability in soil and superior nutrient-retention properties make it an ideal soil amendment to increase crop yields (Lehmann and Joseph, 2009). Biochar is a form of charcoal produced by super-heating biomass. It is found naturally in soils around the world as a result of vegetation fires. Biochar has also been created and used by humans in traditional agricultural practices in the Amazon Basin of South America for more than 2,500 years. Addition of BC to agriculture soils has been projected as a means to improve soil fertility and mitigate climate change (Rondon *et al.*, 2007 ; Wardle *et al.*, 2008 ; Amutio *et al.*, 2013). Dark, charcoal-rich soil (known as *Terra preta* or black earth) supported productive farms in areas that previously had poor, and in some places toxic, soils. *Terra preta* was discovered in the 1950's by Dutch soil scientist Wim Sombroek in the Amazon rainforest. Terra preta still covers 10 % of the Amazon Basin. Similar sites have been found in Ecuador, Peru, Benin and Liberia in West Africa. Today biochar can be produced in an environmentally friendly way - and it needs to be; Pyrolysis, burning with limited oxygen in a closed system, allow material to be burned at high temperatures and all the emissions captured.

Materials and Methods

Collection and Preparation of Samples

The selected samples such as Htiyo-wa, Kathit-su, Pan-mezali, Rice-husk and Baw-za-gaing were collected from an area of 20-Quarter, Shwepyithar Township, Yangon Region. The samples were especially cultivated. The collected samples were cut into many pieces of nearly equal size, which is 1.5 cm in length and 0.4 cm in width. And then, the samples were kept in air to dry at room temperature for a few weeks. Furthermore, the samples were dried in an oven at 99 °C for 4 h because of its moisture content.

Determination of Some Parameters of Plant Materials

Determination of bulk density of samples

A clean dry 100 mL graduated cylinder was weighed. It was then filled with the dry sample to the 100 mL mark and reweighed. The graduated cylinder was placed in a tapping box and the cylinder was gently tapped until there was no more reduction in volume. The minimum volume was recorded and the bulk density was calculated (Antal and Gronli,2003). The results are illustrated in Table 1.

Determination of moisture content (Oven Drying Method)

Into a flat-bottom metallic dish, finely divided asbestos were spreaded in a thin layer. It was firstly dried at 110 °C for 1 h, the dish was covered, cooled and weighed. Sample (20 g) was uniformly spread over the asbestos layer. It was weighed as quickly as possible to avoid loss of moisture. The cover was removed and dried in a hot air oven at atmospheric pressure. A temperature of 100 °C was maintained in the case of plant tissue. The duration of heating will vary with the type of tissues; 16 – 18 h is sufficient for most tissues. After drying, the lid was replaced, the sample was cooled in a desiccator, and it was reweighed. The sample was reheated, if necessary, until the consecutive weighings do not vary by more than 3 – 5 mg. Tissues which contain volatile organic constituents or high percentage of sugars cannot be brought to a constant weight. In such cases, a compromise procedure must be adopted. A standard technique should be employed. Drying at 55 °C for four days is generally suitable. The sample after determination of moisture content–could be used for ashing and estimation of minerals (Buzarbarua, 2000). The results are shown in Table 2

Determination of ash contents of plant materials

Accurately weighed about 10 g of some plant materials such as Htiyo-war, Kathit-su, Pan-mezali, Rice-husk, and Baw-za-gaing were added to the five tared porcelain crucibles and the organic matter was dried and burnt off without flaming and finally heated in a muffle furnace at 823 K (550 °C). Heating was continued until the resultant ashes were white in color and free from particles of unburnt carbon and fused 12 mps. Then, the crucibles

containing the residues were cooled to room temperature in a desiccator and weighed. Heating, cooling and weighing were repeated until a constant weight was obtained. The ash contents were then calculated (Buzarbarua, 2000). The results are shown in Table 3.

Preparation of Biochars from Some Plant Materials

Accurately 600 g wood chips of sample (Pan-mezali) was put into top-lit up draft (TLUD) Furnace. Three nails, which were triangle in position, were placed under a TLUD can. One-third of sample was mixed with fuel such as absolute ethyl alcohol (25 mL) and it was put into a TLUD-can as a top-layer. Then, they were started to burn with a candle flame. As burning continues, the crown was set up at the top of TLUD – can and then chimney, two feet height, was kept over the crown. After complete burning, a blue colored smoke comes out, it was stopped to prevent-ventilation, because air enters from bottom to top during burning which rise in temperature 252 °C by using two feet chimney height for 26 min and it was allowed for cooling. And then, the weight of biochar was determined. Furthermore, biochars were prepared from 600 g wood chips of Pan-mezali by varying the chimney height (Antal and Gronli, 2003).

Similarly, biochars were prepared from baw-za-gaing, kathit-su, htiyo-wa, and rice-husk by using above the same procedure.

Determination of Plant Nutrients in Biochars

Determination of nitrogen contents in biochars

0.1 g of finely ground biochar was transferred to a Kjeldahl flask. 1 mL of salicylic acid sulphuric acid mixture was added and thoroughly mixed. After 20 min, approximately 0.3 g sodium thiosulphate was added and gently heated until fumes are evolved. The mixture was cooled and 0.06 g of catalyst (a mixture of CuSO_4 and K_2SO_4 with the ratio of 1:2) and 0.75 mL nitrogen free H_2SO_4 were added. The mixture was heated on a digestion rack (electric) over a small flame for about an hour until the solution became apple green in colour. The digested sample was cooled and diluted with about 10 – 15 mL of distilled water to dissolve the sample.

The digest was transferred to the flask of the distillation unit through the side tube. The digestion flask was repeatedly washed with 2 – 3 mL of distilled water so that no digest is left in the flask. Excess of 40 % NaOH was added to the flask and the distillation process was continued. A conical flask containing 5 mL of 2 % boric acid solution was placed below the condenser. The distillation process was continued until 20 mL of distillate was collected in the receiving flask.

Two drops of Conway's indicator (mixed indicator of methyl red and bromocresol green) was added to the conical flask containing boric acid and it was titrated against 0.01 N HCl until a faint pink colour is obtained. Blank determination (without sample) using all the reagents as in the case of sample was also made (Buzarbarua, 2000) .

Determination of phosphorus content in biochars (Colorimetric Method)

2 mL of digested sample extract was transferred into 25 mL volumetric flask. A few drops of 2,4-dinitrophenol indicator was added and the contents was neutralized with 4 N ammonia. Any excess of ammonia was neutralized with 2 N H₂SO₄ and the volume to about two third of the flask was made with water. Sulphomolybdate solution (1 mL) was dispensed into it. The neck of the volumetric flask was washed with distilled water, and 0.5 mL of freshly prepared stannous chloride solution was added. The contents were thoroughly mixed and the volume was made to 25 mL. Then, within 4 to 20 mins the absorbance was recorded at 660 nm using a spectrophotometer. Following the above procedure prepare a standard curve containing 0.2 – 1.0 ppm phosphorous. The amount of phosphorous in the sample was found out from the standard cure and the results were expressed as mg /100 g dry weight of the sample after taking into account the dilution factors.

Determination of potassium content in biochars

The sample mineral extract to be analysed was aspirated into the instrument and the observation was recorded after compensation for the blanks. Readings of the standard solutions were periodically taken in between the samples to ensure proper functioning and reproducibility of the instrument response.

Determination of Volatile Matter Contents in Biochars

Accurately 5 g of the sample was weighed into a tared dish which had been previously dried and cooled in a desiccator. It was dried in an oven at 105 °C (AOCS, recommendation 101 °C ± 1°C), and it was removed from the oven. And then, it was cooled in a desiccator and weighed. The procedure was repeated until the loss in weight does not exceed 0.05 % per 30 min drying period.

Results and Discussion

Some Parameters of Plant Materials

Bulk density

Bulk density is an important property of biomass that directly affects the costs of distribution and storage. The bulk density as the physical characteristic depends on material composition, shape and dimensions of particles, orientation of particles, their density and size distribution, moisture content, pressure, contamination degree, rate and kind of deposit formation. The bulk densities of some plant materials such as Pan-mezali, Baw-zagaing, Kathit-su, Htiyo-wa, and Rice husk were determined. The results are shown in Table 1 and Figure 1.

Table 1 Bulk Density of Plant Samples for Preparation of Biochars

Parameter	Baw-za-gaing	Htiyo-wa	Kathit-su	Panmezali	Rice husk
Density (g cm ⁻³)	84.78	80.12	79.12	82.21	75.12
Moisture content (%)	2.65	1.75	2.32	1.89	3.25
Ash content (%)	1.2	0.13	0.20	0.18	0.62

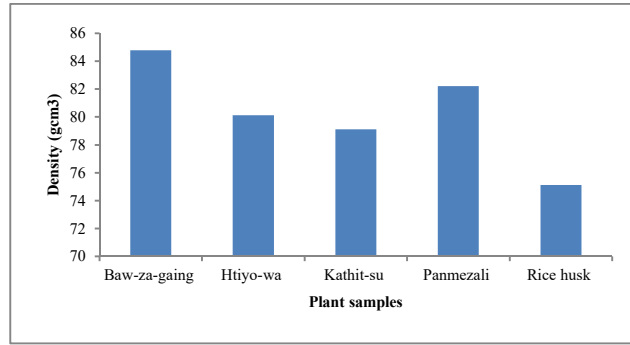


Figure 1 Bar graph showing bulk densities of plant samples for biochar preparation

Moisture content

Moisture content was determined by drying in an oven. This method consists in measuring the weight loss by plant materials due to the evaporation of waters. Drying methods are generally used as they give accurate results. The moisture content of rice-husk is the highest and the yield percent is the least as well as the moisture content of htiyo-wa is the least and the yield percent is the highest (Table 1 and Figures 2 and 3).

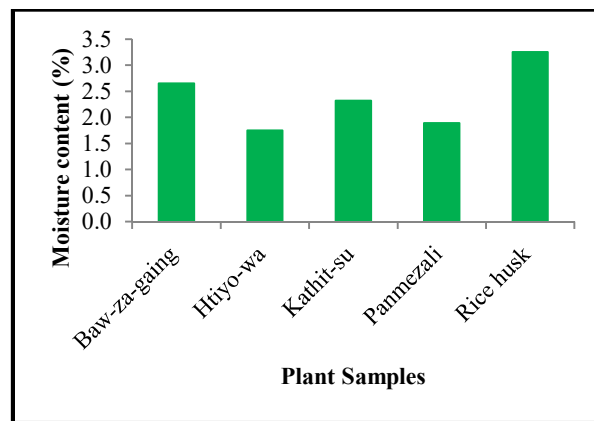


Figure 2 Bar graph showing moisture contents for plant samples used to prepare biochars

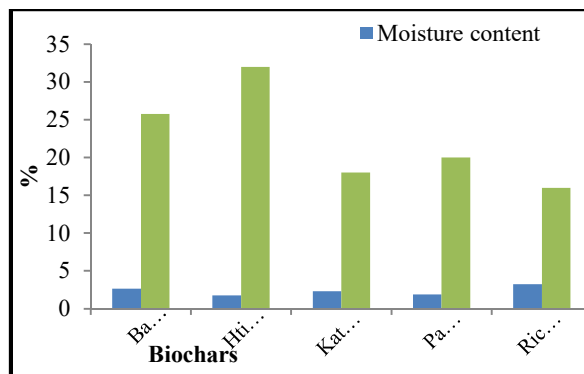


Figure 3 Bar graph for comparison of moisture content and yield percent of biochars

Ash content

Ash content of plant materials represents inorganic residues remaining after destruction of organic matter. It may not necessarily be exactly equivalent to the mineral matter as some changes may occur due to volatilization or some interaction between constituents. High ash content and / or a low alkalinity of the ash may in some cases be suggestive of the presence of adulterants. The acid insoluble ash is a measure of sand and other silicious matter present. Difficulty of effecting complete combustion in some sample, and the possible loss by volatilization on ignition may be overcome by moistening the substance to be ignited or the carbonaceous residue therefrom with concentrated sulphuric acid. In the determination of ash content in the collected plant materials, it was observed in Figure 4 that the ash content in Baw-zagaing was the highest and that of Htiyo-wa was the lowest. The results are shown in Table 1 and Figure 4.

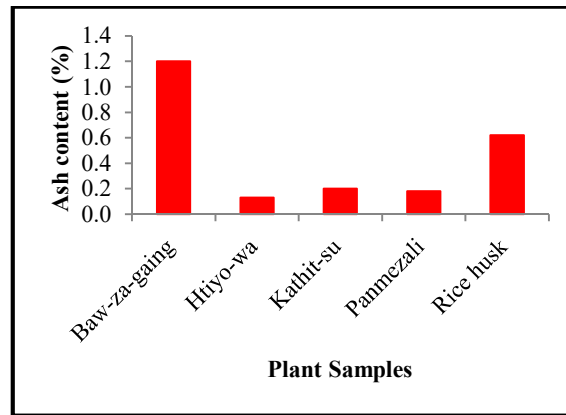


Figure 4 Bar graph showing ash contents of plant samples

Biochar from pan-mezali

Many methods can be used to produce biochar. Even with the same equipment process parameters can vary. It is generally known that the yield of products from pyrolysis varies heavily with temperature. The lower the temperature the more char is created per unit biomass. But TLUD method gave the yield percent between 10 and 19.04. Biochar was prepared from pan-mezali by using two feet chimney height and the rise of temperature was recorded. Furthermore, biochar was prepared by changing different chimney heights (1 feet – 3 feet) and the rise in temperature was recorded. In the preparation of biochar by changing the chimney height, the yield percent gave the highest at two feet-six inches. The results are shown in Table 2. With one feet chimney height rise in temperature to 248 °C within 29 mins, with one and half feet chimney height rise in temperature to 250 °C within 30 mins, with two and half feet chimney height rise in temperature to 275 °C within 28 min and with three feet chimney height rise in temperature to 272 °C within 22 min. were observed (Antal and Gronli, 2003).

Table 2 Biochar from Pan-mezali Wood at Different Chimney Heights

No.	Chimney height	Sample wt (g)	Time taken (min)	Temperature (°C)	Product wt (g)	Yield (%)
I	1' 0"	600	29	248	60	10.00
II	1' 6"	600	30	250	60	10.00
III	2' 0"	600	26	252	60	10.00
IV	2' 6"	600	28	275	100	16.70
V	3' 0"	600	22	272	60	10.00

Biochar from baw-za-gaing

Biochar was prepared from baw-zagaing by using two feet chimney height and the rise in temperature was recorded. Furthermore, biochar was prepared at different chimney heights and the rise in temperatures was recorded. In the preparation of biochar by changing the chimney height, the yield percent was the highest at two feet and one feet-six inches heights. The results are shown in Table 3.

Table 3 Biochar from Baw-za-gaing Wood at Different Chimney Heights

Sample No.	Chimney height	Sample wt (g)	Time taken (min)	Temperature (°C)	Product wt (g)	Yield (%)
I	1' 0"	400	18	280	50	12.50
II	1' 6"	400	18	289	75	18.75
III	2' 0"	400	18	300	75	18.75
IV	2' 6"	400	20	320	50	12.50
V	3' 0"	400	17	300	50	12.50

Biochar from kathit-su

Biochar was prepared from kathit-su by using two feet chimney height and the rise in temperature was recorded. Furthermore, biochar was prepared by changing different chimney heights and the rise in temperature was also recorded. In the preparation of biochar by changing the chimney height, the yield percent of the char was highest at one feet-six inches. The results are shown in Table 4.

Table 4 Biochar from Kathit-su Wood at Different Chimney Heights

Sample No.	Chimney height	Sample wt (g)	Time taken (min)	Temperature (°C)	Product wt (g)	Yield (%)
I	1' 0"	300	15	130	30	10.00
II	1' 6"	300	16	128	50	16.69
III	2' 0"	300	13	152	30	10.00
IV	2' 6"	300	17	238	30	10.00
V	3' 0"	300	13	200	30	10.00

Biochar from htiyo-wa

Biochar was prepared from htiyo-wa by using two feet chimney height and the rise in temperature was recorded. Furthermore, biochar was prepared by changing different chimney heights and the rise in temperature was recorded. In the preparation of biochar by changing the chimney height, the yield percent was the highest at two feet and two feet-six inches. The results are shown in Table 5.

Table 5 Biochar from Htiyo-wa Stem at Different Chimney Heights

Sample No.	Chimney height	Sample wt (g)	Time taken (min)	Temperature (°C)	Product wt (g)	Yield (%)
I	1' 0"	1050	50	280	180	17.14
II	1' 6"	1050	53	279	180	17.14
III	2' 0"	1050	56	296	200	19.04
IV	2' 6"	1050	55	280	200	19.04
V	3' 0"	1050	53	295	180	17.14

Biochar from rice-husk

Biochar was prepared from rice-husk by using two feet chimney height and the rise in temperature was recorded. Furthermore, biochar was prepared by changing different chimney heights and the rise in temperature was recorded but, it was observed that it did not give the complete combustion as the above samples.

Plant Nutrients in Biochars

Nitrogen content in biochars

Nitrogen content was estimated by the Kjeldahl method which is based on the determination of the amount of reduced nitrogen (NH₂ and NH) present in the sample (char). The various nitrogen compounds are converted into ammonium sulphate by boiling with concentrated H₂SO₄. The ammonium sulphate formed is decomposed with an alkali (NaOH), and the ammonium liberated is absorbed in excess of neutral boric acid solution and then titrated with standard acid. The results are shown in Table 6.

Table 6 Nitrogen Contents in Biochars

Parameter	Baw-za-gaing	Htiyo-wa	Kathit-su	Panmezali	Rice husk
Nitrogen content (%)	3.1	2.4	2.5	3.0	2.1

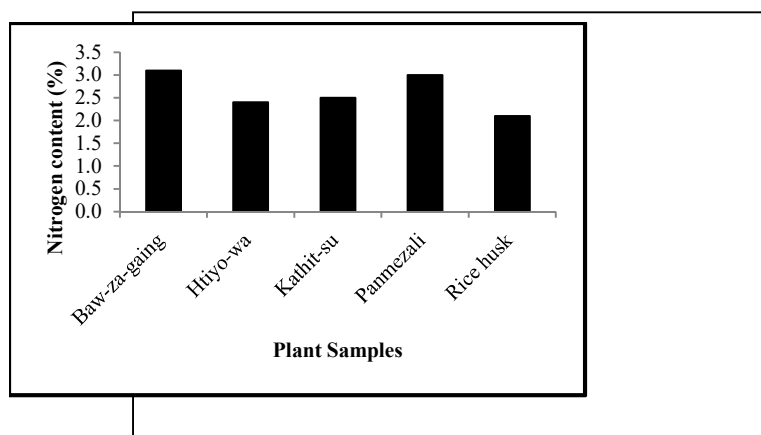


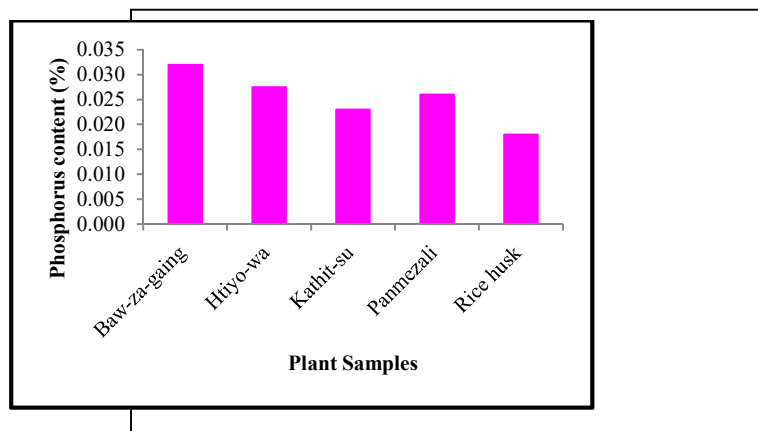
Figure 5 Bar graph showing nitrogen contents of biochars

Phosphorous content in biochars

For the determination of total phosphorous, the sample in which organic matter has been destroyed by tri-acid mixture is used. The phosphate containing solution is treated with sulphomolybdic acid to produce phosphomolybdic acid. This is then reduced by stannous chloride giving a blue coloured complex whose colour intensity is proportional to the amount of phosphate in the preparation. The results are shown in Table 7.

Table 7 Phosphorous Contents in Biochars

Parameter	Baw-za-gaing	Htiyo-wa	Kathit-su	Pannezali	Rice husk
Nitrogen content (%)	3.1	2.4	2.5	3.0	2.1

**Figure 6** Bar graph showing phosphorous content of biochars

Potassium content in biochars

The atomic absorption spectrophotometry was used for the determination of potassium content. The plant tissue must first be properly processed before its introduction into the atomic absorption spectrophotometer (AAS). Dry ashing can effectively be used for determination of potassium in plant tissue. The results are shown in Table 8.

Table 8 Potassium Contents in Biochars

Parameter	Baw-za-gaing	Htiyo-wa	Kathit-su	Pannezali	Rice husk
Potassium contents (%)	0.1814	1.807	0.8459	0.8457	0.8471

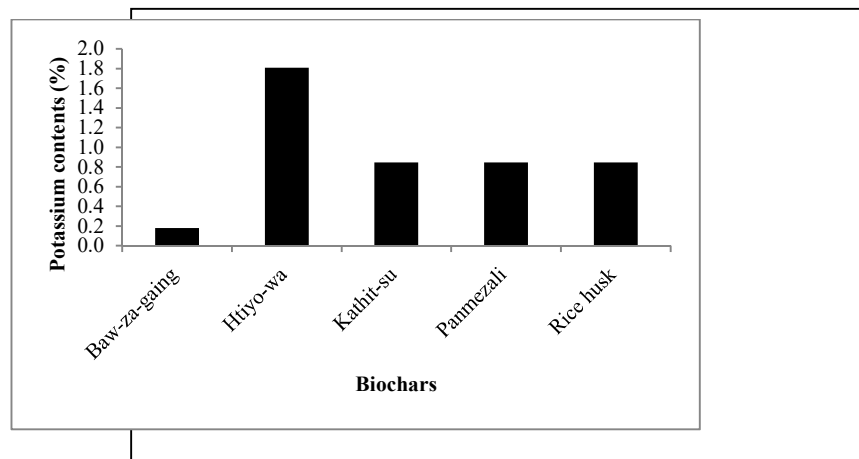


Figure 7 Bar graph showing potassium contents of biochars

Volatile Matter Contents in Biochars

The volatile matter other than water in biochar comprises all those liquid and tarry residues not fully driven-off in the process of carbonisation. If the carbonisation is prolonged and at a high temperature, then the content of volatiles is low. When the carbonisation temperature is low and time in the retort is short, then the volatile matter content increases. The volatile matter in biochar can vary from a the highest of 40% or the lowest to 5% or less. It is measured by heating away from air, a weighed sample of dry biochar at 900°C to constant weight. The weight loss is the volatile matter. Volatile matter is usually specified free of the moisture content, i.e. volatile matter - moisture or (V.M. - moisture). High volatile biochar is easy to ignite but may burn with a

smoky flame. Low volatile biochar is difficult to light and burns very cleanly. A good commercial biochar can have a net volatile matter content - (moisture free) of about 30%. High volatile matter biochar is less friable than ordinary hard burned low volatile biochar and so produces less fines during transport and handling. It is also more hygroscopic and thus has a higher natural moisture content. Volatile matter content in biochars was determined by using muffle furnace at 950 °C. At this temperature some elements in biomass decomposed and changed into volatile gases. The results are shown in Table 9.

Table 9 Volatile Matter Contents of Biochars

Parameter	Baw-za-gaing	Htiyo-wa	Kathit-su	Panmezali	Rice husk
Potassium contents (%)	0.1814	1.807	0.8459	0.8457	0.8471

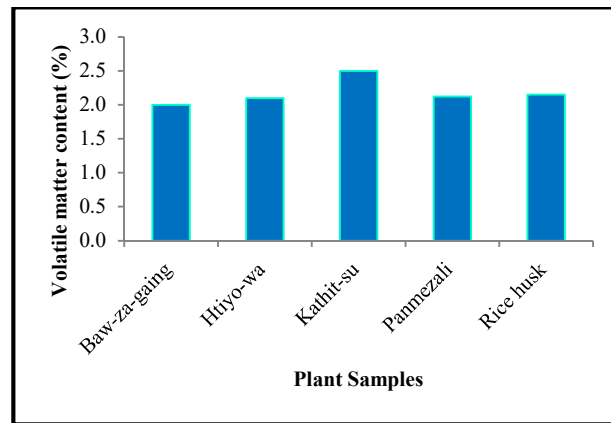


Figure 8 Bar graph showing volatile matter contents in biochars

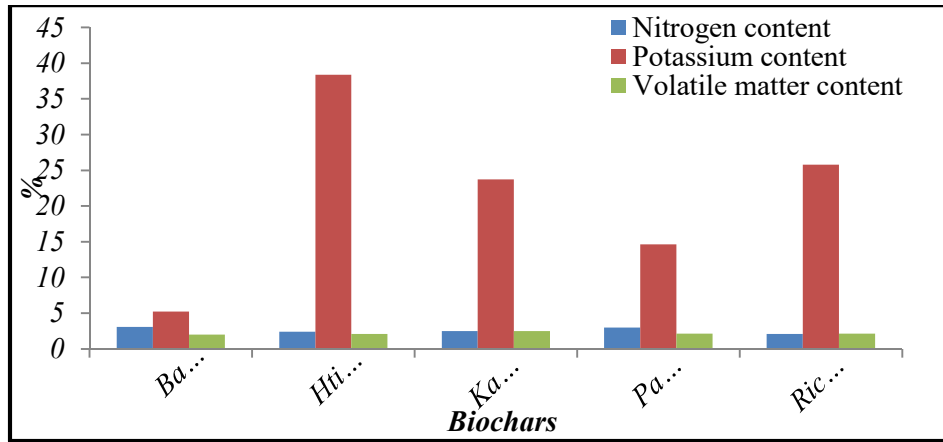


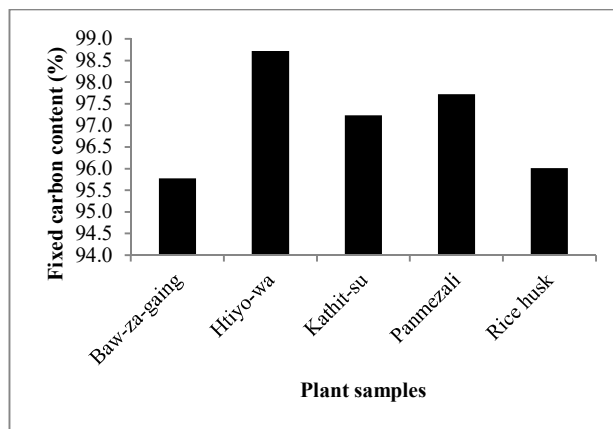
Figure 9 Bar graph for comparison of nitrogen, potassium and volatile matter contents of biochars

Fixed Carbon Content of Biochars

The fixed carbon content of biochars ranges Low between 95.775 % and around 98.72 %. Thus biochars consist mainly of carbon. The carbon content is usually estimated as a "difference", that is to say, all the other constituents are deducted from 100 as percentages and the remainder is assumed to be the per cent of "pure" or "fixed" carbon. The fixed carbon content is the most important constituent in metallurgy since it is the fixed carbon which is responsible for reducing the iron oxides of the iron ore to produce metal. But the industrial user must strike a balance between the friable nature of high fixed carbon biochar and the greater strength of biochar with a lower fixed carbon and higher volatile matter content to obtain optimum blast furnace operation. Figure 10 shows the comparison of fixed carbon content of some plant materials. It was observed that Baw-zagaing had the lowest content and Htiyo-wa had the highest content. The results are shown in Table 10.

Table 10 Fixed Carbon Contents of Biochars

Parameter	Baw-za-gaing	Htiyo-wa	Kathit-su	Panmezali	Rice husk
Fixed carbon content (%)	95.775	98.720	97.230	97.718	96.015

**Figure 10** Bar graph showing fixed carbon contents in biochars

Conclusion

In the preparation of biochars from some plant materials, it could be concluded as follows. The prepared biochars were obtained 10.0 %, 18.75 %, 10.0 %, 19.04 %, and 10.0 % yields from some plant materials: namely Pan-mezali, Baw-za-gaing, Kathit-su, Htiyo-wa and Rice-husk, respectively. In the process of biochar preparation, the effectiveness of chimney height in TLUD furnace was studied by using various chimney heights (1-3). The highest yield % of biochar from different plants materials was acquired in the range of 1' 6''- 2' 6'' chimney height. Among the five different samples, Htiyo-wa gave the highest yield % of biochar (19.04 %). Bulk densities of plant samples such as Pan-mezali 82.21 g/100 cm³,

Baw-zagaing 84.78 g/100 cm³, Htiyo-wa 80.12 g/100 cm³, Kathit-su 79.12 g/100 cm³, and Rice husk 75.12 g/100 cm³ were determined. Moisture contents of plant materials were found out to be, Pan-mezali 1.89 %, Baw-zagaing 2.65%, Htiyo-wa 1.75%, Kathit-su 2.32 %, and Rick husk 3.25 %. Ash contents of plant materials were found to be, Baw-za-gaing 1.20 %, Htiyo-wa 0.13%, Kathit-su 0.20 %, Panmezali 0.18% and Rick husk 0.62 %. The plant nutrients such as nitrogen, phosphorus, and potassium contents in biochars of some plant materials were also determined. The nitrogen contents of biochars from Pan-mezali, Baw-za-gaing, Htiyo-wa, Kathit-su, and Rice-husk were determined to be 3.0%, 3.1%, 2.4%, 2.5%, and 2.1%, respectively. The phosphorus contents of biochars from Pan-mezali, Baw-zagaing, Htiyo-wa, Kathit-su, and Rick-husk were 0.026%, 0.032%, 0.027%, 0.023%, and 0.018%, respectively. The potassium contents in biochars of Pan-mezali, Baw-za-gaing, Htiyo-wa, Kathit-su, and Rice-husk were also determined as, 0.8457 %, 0.1814 %, 1.807 %, 0.8459 %, and 0.8471 %, respectively. Similarly, the volatile matter contents of biochars were found to be 2.12%, 2.00%, 2.10 %, 2.50%, and 2.15%, respectively, from Pan-mezali, Baw-za-gaing, Htiyo-wa, Kathit-su, and Rice-husk. The fixed carbon contents were obtained 95.775 % from Baw-za-gaing, 98.72 % from Htiyo-wa, 97.23 % from Kathit-su, 97.718 % from Pan-mezali, and 96.015 % from Rice Husk, respectively. Further Study is to be made for the biochars for useful and as soil amendment in agriculture.

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