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Influence of Selected Parameters on the Product Yields of Broom Weed (*Sida acuta***) Pyrolysis**

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Authors' contributions

This work was carried out in collaboration among all authors. Authors read and approved the final manuscript.

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ABSTRACT

Biomass feedstocks have been utilized for the commercial production of a wide range of fuels and chemicals through the pyrolysis process. Broom weed is abundant but it has not been widely used in Nigeria as a means of useful fuel and chemical thereby constituting an environmental menace. This study aimed to determine the effects of selected process parameters on the product yields during the pyrolysis of broom weed in a fixed bed reactor. Broom weed was obtained at the premises of the National Centre for Agricultural Mechanization, Ilorin, Kwara State, Nigeria. 0.1kg of a dried sample of broom weed was loaded into a steel retort, and the retort interior was rendered airtight. The retort was then placed into the furnace chamber and was pyrolysed at 300°C between 10 - 30 minutes at 5 minutes intervals. This was repeated for temperatures of 350, 400, 450, and 500 \degree C, and in each case, the quantities of char, bio-oil, and gas produced were determined. The average minimum values of the product yielded char, bio-oil, and gas for broom weed were 62.43 wt% at 500 $^{\circ}$ C, 4.34 wt% at 300 $^{\circ}$ C and 12.62 wt% at 300 $^{\circ}$ C, respectively, while the maximum

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values were 83.37 wt% at 300 $^{\circ}$ C, 8.80 wt% at 450 $^{\circ}$ C and 30.96 wt% at 500 $^{\circ}$ C, respectively. This study showed that pyrolysis of broom weed can serve as a renewable source of energy rather than constituting an environmental menace.

Keywords: Pyrolysis; fixed bed reactor; broom weed; char; bio-oil; gas.

1. INTRODUCTION

"Incessant increases in petroleum product prices and depletion of the crude oil deposits had brought non-conventional and renewable energy sources to greater attention" [1]. "The dependency on renewable energy sources like solar, wind, geothermal, hydrothermal, and biomass has received extensive recognition in recent years and is considered to be the most suitable and sustainable source of energy and the possible solution to human concerns" [2]. "Bio-fuel produced from biomass residues is an alternative to petroleum products and has received great attention due to the depletion and environmental problems associated with the usage of fossil fuels. Presently, it provides about 14% of the worldwide energy demand" [3]. "Apart from its abundant availability, the Combustion of biomass produces lesser oxides of sulphur, nitrogen, and carbon which makes it more suitable for energy generation than conventional fossil fuel thus keeping the environment and the public's health safe" [1,2,4]. Nigeria as one of the oil-producing countries, is faced with energy crises and a shortage of petroleum products coupled with the periodic escalation in prices of fuel; therefore, Nigeria as a country must search for an alternative source of energy. Therefore, Nigeria needs to begin and encourage research on biofuel production from available and economically feasible feedstock to back up its dependency on fossil fuels. Broom weed (*Sida acuta*) is an excellent example of such feedstock because it has comparatively few competing food uses, which encouraged research into its potential for biofuel generation.

"Biochar is a stable solid containing carbon and can last in the soil for thousands of years. The carbon in biochar resists degradation and can hold carbon in soils for hundreds to thousands of years, it is produced through gasification or pyrolysis processes that heat biomass in the absence of oxygen" [5]. According to [6] "biochar has attracted significantly growing attention due to its effectiveness in terms of both cost and environmental safety in removing trace metals from soil and water. Its metal sorption capacity depends on its properties, which are in turn governed by pyrolysis temperature and type of biomass". The method of transforming biomass into a useful wellspring of energy can be accomplished by utilizing various techniques that can be grouped into two fundamental classes namely; thermochemical processes and biochemical/biological processes as in fermentation and aerobic digestion [7]. The thermochemical conversion techniques incorporate pyrolysis, gasification, and combustion and these constitute one of the promising routes among the sustainable energy alternatives for the future since almost all biomass can be utilized as feedstock even waste unlike fermentation and aerobic digestion which are precise in their biomass feedstock requirement. Pyrolysis has pulled in more enthusiasm for producing liquid fuel products out of the conversion process due to its potential benefits in storage, transport, and usefulness in applications such as combustion engines, boilers, and turbines, among others [8]. "The product yields (char, bio-oil, and gas) obtained during pyrolysis generally depend upon various operating parameters like residence time, heating rate, and temperature" [9]. Similarly, "biomass composition, particle size, and density also play a vital role in determining the grade and quantity of pyrolysis products" [10]. High heating rates and shorter vapour residence time in fast pyrolysis produce greater pyrolytic-oil yield than in slow pyrolysis which employs low heating rates and longer vapour residence time. Research has been conducted on the effect of process parameters on the pyrolysis characteristics of biomass feedstock [11,12,13]. Others have studied the effect of different types of biomass materials on pyrolysis products [3,14,15]. Therefore, in this study, the influence of pyrolysis temperature and time on the product yields during the pyrolysis of broom weed was investigated.

2. MATERIALS AND METHODS

2.1 Feedstock Procurement and Preparation

Broom weed used for the pyrolysis experiments in this study was obtained from National Centre for Agricultural Mechanization, Ilorin, Kwara State, South-Western Nigeria. The residues were cleaned to remove foreign particles such as stones, leaves, debris, and other unwanted components in order not to contaminate the pyrolysis product and as well sun-dried for 21 days. The weight of the sample (W_1) was measured using an Ohaus top loading digital weighing scale of sensitivity \pm 0.001 g (Model: PA4102, range: 0-4100 g, Ohaus company, Manufactured in Switzerland) and then ovendried at a temperature of 105° C until constant weight (W_2) was obtained following official methods of the [16].

2.2 Determination of Proximate and Ultimate Analysis

2.2.1 Determination of moisture content

The moisture content of the broom weed was determined by gravimetric method according to [17]. 2 g each of the samples was weighed and placed in a crucible. The crucible and the measured feedstock were oven-dried at 103 \degree C to constant weight for 24 hours. The percentage moisture content of the samples was determined using Equation 1:

$$
\% MC = \frac{(W_i - W_f)}{W_i} \; X \; 100 \tag{1}
$$

Where:

MC = Moisture content (%) W_i = Initial weight of the sample (g) W_f = Final weight of sample (g)

2.2.2 Determination of ash content

The ash content of the broom weed was determined gravimetrically according to [16] by weighing 2 g of the sample and oven-dried it in an electric oven for 24 hours. The dried samples were powdered and 2 g of the samples were accurately weighed and placed in a crucible of known weight. These were ignited in a muffle furnace for 8 hours at 550°C. The crucible containing the ash was then removed, cooled in a desiccator, and weighed. The percentage ash content of the siam and broom weeds was determined using equation 2.

$$
\%AC = \frac{w_a}{w_s} \times 100\tag{2}
$$

Where:

AC = Ash content (%) W_s = Weight of the sample (g) W_a = Weight of ash (g)

2.2.3 Determination of the volatile matter

The volatile matter was determined according to [16] by pulverizing 2 g of each of the samples in porcelain and placing it in an oven-dried electric oven until a constant weight is obtained. The samples were heated in a furnace at 550 °C for 10 mins. It was cooled in a desiccator and weighed. The percentage of volatile matter was calculated using Equation 3.

$$
\%VM = \frac{(W_x - W_y)}{W_s} \times 100
$$
 (3)

Where:

 $VM = Vol$ atile Matter (%) W_c = Weight of the sample (g) W_x = Weight of the dry sample (g) W_v = Weight of residue (g)

2.2.4 Determination of fixed carbon content

The fixed carbon was determined in accordance with [18] by representing the content in a sample that has not combined with any other element (in a free state) and was calculated by computing the difference between 100 and the sum of moisture content (MC) volatile matter (VM) and ash content (AC) of the feedstock samples using equation 4:

$$
\%FCC = 100 - (MC + AC + VC) \tag{4}
$$

2.2.5 Determination of heating values

The higher and lower heating values of the siam and broom weeds were determined using a bomb calorimeter in accordance [19]. An ignition mechanism started the stoichiometric combustion of two moles of hydrogen and one mole of oxygen at 25 °C in a steel container, and the processes were allowed to complete. Water vapor was created during burning when hydrogen and oxygen react. After cooling the vessel and its contents to 25 °C, the higher heating value was determined as the amount of heat released between the initial and final temperature. The [lower heating value](https://en.wikipedia.org/wiki/Lower_heating_value) (LHV) was determined when cooling was stopped at 150 °C and the reaction heat is only partially recovered.

The limit of 150 °C is based on [acid gas](https://en.wikipedia.org/wiki/Acid_gas) dewpoint. Higher heating value (HHV) is calculated with the product of water being in liquid form while lower heating value (LHV) is calculated with the product of water being in vapor form.

$$
HHV = 4.18 \times (78 \times C + 241.3 \times \left(\frac{Hx0}{8}\right) +
$$

22.1 \times S) (5)

$$
LHV = 4.18 \times (94.19 \times C - 0.550 - 52.14 \times H \tag{6}
$$

Where:

HHV ─ Higher Heating value LHV ─Lower Heating value C ─Percentage of carbon content H ─Percentage of hydrogen content O ─Percentage of oxygen content S-Percentage of sulphur content

2.2.6 Determination of the carbon content

The percentage of carbon (%C) was determined in accordance with [18]. The carbon is converted to carbon dioxide by combustion in a stream of oxygen. In the thermal conductivity test method, the carbon dioxide is absorbed on a suitable grade of zeolite, released by heating the zeolite and swept by helium or oxygen into a chromatographic column. Upon elution, the amount of carbon dioxide is measured in a thermistor-type conductivity cell. The percentage of carbon was determined using equation 7.

$$
\%C = \frac{(A-B)\times C}{D} \tag{7}
$$

Where:

- $A = Diqital volume$ volumetric reading for sample
- $B =$ Digital volumetric reading for blank
- C = Mass compensator setting
- D = Sample setting

2.2.7 Determination of nitrogen content

The percentage of Nitrogen (%N) was determined using the Kjeldahl method [19,20]. 0.5-1.0 g of dried samples was taken in a digestion flask and 20 ml of concentrated H_2SO_4 and about 0.2g selenium catalyst was added. The flask was swirled to mix the contents thoroughly and then placed on the heater to start digestion till the mixture become clear. The digest was cooled before being transferred to a

100 ml volumetric flask, where it was given the correct volume by adding distilled water. The Markam Still Distillation Apparatus was used to distill the digest. Ten milliliters of digest were introduced into the distillation tube then ten milliliters of 40% NaOH were added gradually in the same manner. Distillation was continued for at least 10 minutes and $NH₃$ produced was collected as NH4OH in a conical flask containing 5ml of 2% boric acid solution with a few drops of modified mixed indicator. The distillate was then titrated against standard 0.002M HCI solution till the appearance of pink color. A blank was also run through all steps as above. The percent crude protein content of the sample was calculated by using Equation 8.

$$
\%N = \frac{(S-B) \times N \times 0.014 \times V1 \times D}{Ws \times V2} \times 100
$$
 (8)

Where:

S = Sample titration reading $B =$ Blank titration reading $N =$ Normality of HCI $D =$ Dilution of the sample after digestion V_1 = Volume of the digested sample after it was made up V_2 = Volume taken for distillation 0.014 = Milli equivalent weight of Nitrogen. $Ws = Weight of sample (g)$

2.2.8 Determination of sulphur content

The percentage sulphur (%S) was determined in accordance with [17]. 1.0 g of the dried samples, and approximately 1.5 g of accelerator to a crucible. The sample was combusted in a stream of oxygen that converts the sulfur in the sample to sulfur dioxide. The sulfur is measured using infrared absorption spectrometry. The percentage of sulphur was calculated using equation 9.

$$
\%S = \frac{(A-B) \times Y}{Z} \tag{9}
$$

Where:

A = DVM (Digital Volt Meter) reading for samples

 $B = DVM$ reading for blank

 $Y =$ Mass compensator setting,

 $Z =$ Sample mass (q)

2.2.9 Determination of hydrogen content

The percentage of hydrogen (%H) was determined in accordance with [14]. 0.5g of the test sample is burnt in a stream of oxygen at a $temperature$ of 1350 $^{\circ}$ C. The products of incomplete combustion are further burnt over copper oxide to ensure complete conversion of all carbon and hydrogen to carbon dioxide and water respectively. The percentage of hydrogen is determined by using Equation 10

$$
\%H = \frac{W_W \times 0.119 \times 100}{W_S} \tag{10}
$$

Where:

 W_w = Weight of water (g) W_s = Weight of sample (g)

2.2.10 Determination of oxygen content

The percentage of oxygen (%O) was determined in accordance with [14] by using Equation 11

$$
\%O = 100 - (C + H + N + S + \%ASH) \quad (11)
$$

2.3 Determination Of Effect On The Selected Parameter

Pyrolysis experiments were carried out to determine the effect of selected parameters on the product yields of broom weed. 0.1kg of dried broom weed was fed into the retort. The retort was placed into the furnace and pyrolysed within the temperature range of 300 to 500 $^{\circ}$ C, at 50 $^{\circ}$ C intervals with a retention time of 10 to 30 minutes at 5 minutes intervals. The retort was connected through a pipe to the condensate receiver which was placed in an ice-cooling unit for the quick recovery of the condensable products (bio-oil), and from the condensate receiver, the uncondensed gases moved through a lagged pipe into the gas collection unit. The char in the retort and the bio-oil in the condensate receiver were collected and weighed using Ohaus's top-loading digital weighing balance. The weight of the gas was evaluated by subtraction. The percentage of product yields was determined by equation 12.

Percentage product yield (Y) =
$$
\frac{M_p}{M_s} \times 100
$$
 (12)

Where M_p is Mass of the product, and M_s is the Mass of the sample

3. RESULTS AND DISCUSSION

3.1 Proximate and Ultimate Analysis of the Broom Weed

Table 1 shows the results of the physical and chemical properties of broom weed. The

moisture content for broom weed is 6.15%. The level of moisture content in the biomass feedstock has been observed to affect the temperature of the solid due to exothermic evaporation and total heat required to bring the feedstock into pyrolysis temperature. The ash content for broom weed is 9.83%. The amount and chemical composition of ash have effects on the fuel properties of biomass, and therefore important for the selection of the type of conversion technology [3]. Low ash content means low slagging and fouling effects. Biomass with less ash content is reported to have higher heating values as compared with those containing higher ash content. Although ash does not contribute to the overall heating value, certain elements and alkali metals in the ash are catalytic to thermal decomposition, and low ash content also has been reported to maximize biooil yield [2]. The volatile content obtained for broom weed is 22.25%. Combustible volatile content aids the onset of pyrolysis by yielding at higher temperatures as it burns.

Table 1. Results of physical and chemical composition of broom weed

Composition	Broom weed
Moisture content (%)	6.15
Ash content (%)	9.83
Volatile matter (%)	22.25
Fixed carbon content (%)	67.24
Carbon (%)	52.93
Hydrogen (%)	17.29
Oxygen (%)	31.03
Sulphur (%)	1.22
Nitrogen (%)	0.52
Higher Heating Value	20.89
(MJ/Kg)	

Ultimate analysis results showed that broom weed has 52.93% C, 17.29% H, and 31.03% O_2 .
These elements constitute the major These elements constitute compositions of broom weed as this is evident from the results of elemental composition in Table 1. One of the reasons why the heating values of the samples are comparably lower than those of conventional fuels is that the samples have more oxygen content than the conventional fuels. However, these samples when used would be good as chemical feedstock with a high percentage of oxygen content. The percentage of nitrogen in broom weed is 0.52%. The values are in the range of those available for other agricultural residues such as goat weed (0.46%). The percentage of sulphur content broom weed is 1.22%. Generally, the percentage of nitrogen

and sulphur content of the sample is reasonably low which implies that the fuel produced from the sample would be mostly devoid of the toxic nitrogen and sulphur oxides that are present in petroleum-based fuels which are harmful to the environment. The higher heating value of broom weed is 20.89 MJ/kg. The value was compared to other agricultural residues such as goat weed and palm kernel shell [1,2].

3.2 Effect of Temperature on the Product Yields during Pyrolysis of Broom Weed for 10 To 30 Minutes

The result of the product yields during the pyrolysis of broom weed for 10 to 30 minutes at 5 minutes interval is illustrated graphically in Figs. 1-5 and it was deduced that as the temperature increase for 300, 350, 400, and 450 $^{\circ}$ C, the char decreased while the bio-oil and gas increased except when the temperature is at 500 $^{\circ}$ C, the char decreased, the bio-oil decreased and the gas increased. The drop in the bio-oil yield at a pyrolysis temperature range of $450-500$ $^{\circ}$ C was mainly due to the secondary cracking and reaction of the liquid fraction volatiles thereby increasing the gas yield further and reducing the char yield which implies the end of the pyrolysis. According to the reviewed literature, it was observed that the quantity of char produced decreases with an increase in pyrolysis temperature [2,1,4].

Fig. 1. Effect of temperature (⁰ **C) on the product yields (%) during pyrolysis of broom weed for 10 minutes**

Fig. 2. Effect of temperature (⁰ **C) on the product yields (%) during pyrolysis of broom weed for 15 minutes**

Fig. 3. Effect of temperature (⁰ **C) on the product yields (%) during pyrolysis of broom weed for 20 minutes**

Fig. 4. Effect of temperature (⁰ **C) on the product yields (%) during pyrolysis of broom weed for 25 minutes**

Fig. 5. Effect of temperature (⁰ **C) on the product yields (%) during pyrolysis of broom weed for 30 minutes**

4. CONCLUSION

Production of biofuel from the pyrolysis of broom weed has been studied. The results showed that a change in pyrolysis temperatures has significant effects on the pyrolysis product yields. It has also been established that broom weed can serve as an alternative fuel for domestic and industrial application use rather than being considered as waste and environmental menace

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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