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Characteristics of Bio-oil from the Fast Pyrolysis of Elephant Grass (*Pennisetum purpureum Schumach*) in a Fluidized Bed Reactor

Joao F. Sousa¹, Marcio B. D. Bezerra^{1*}, Marlon B. B. Almeida², Gustavo T. Moure², Juan M. Mesa-Perez³ and Elina B. Caramao⁴

¹DEQ – Universidade Federal do Rio Grande do Norte, Brazil.
 ²Centro de Pesquisas e Desenvolvimento (CENPES), Brazil.
 ³UNICAMP – Universidade de Campinas – São Paulo, Brazil.
 ⁴DQI – Universidade Federal do Rio Grande do Sul, Brazil.

Authors' contributions

This work was carried out in collaboration between all authors. Authors JFS and MBDB designed the study and wrote the manuscript. Authors MBDB and MBBA carried out the experiments, collected and analyzed products. Authors GTM and JMMP analyzed the biomass, helped with the experiments and maintenance of apparatuses. Author EBC was responsible for the chromatographic analyses. All authors read and approved the final manuscript.

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ABSTRACT

Bio-oil was obtained from the fast pyrolysis of elephant grass in a semi-pilot scale pyrolysis plant equipped with a 40 kg/h fluidized bed reactor. The temperature varied between 480 and 520°C. The biomass (moisture of 10%) was fed with a rate between 20 and 35 kg/h. Approximately 4.5 kg of sand was used as fluidizing agent. The pyrolytic oil was analyzed for the contents of carbon, hydrogen and nitrogen and sulfur. Water content, acidity, pH, viscosity at 40°C, density and higher heating value were also determined. Chromatographic analysis showed that it was composed mostly of acetic acid, syringol, vinyl-phenol, guaiacol and hexanoic acid. Main results are used to compare the influence of different configurations of a gas-washing column and vapor condensation system on the properties of the bio-oil.

*Corresponding author: E-mail: marciobarbalho@live.com;

Keywords: Elephant grass; bio-oil; char; pyrolysis; fluidized bed reactor.

1. INTRODUCTION

The interest in alternative energy sources has increased considerably in the last decades. Due to problems caused to the environment by the burning of fossil fuels and the concern with global energy security, renewable sources of energy guickly became an interesting solution and were then inserted in the global energy matrix. The demand for alternative sources of energy was initially judged as inadequate and non-viable. However, recent developments have shown that they are driving the technological development of many fuels and energy conversion processes [1-5]. Biomasses, in general, have played an important role on this scenario and became one of the most important sources of renewable energy.

Pyrolysis is one of the alternative processes that can convert solid biomass into valuable products that can be easily stored and transported such as bio-oil and char. Synthesis gas is a by-product of this reaction. Various types of pyrolysis processes have been studied in recent years, the most promising technologies are the fluidized bed and ablative reactors; which are usually associated with high productivity, low energy consumption, easy and fast temperature control. These are key factors to produce high quality bio-oils with potential to be used in different applications such as fuel for burners, internal combustion engines and turbines [6]. Once upgraded and treated, bio-oil can also be a source for valuable chemicals, [2,4].

Elephant grass, just like many other energy crop, is abundant in Brazil, especially in the northeast region. It can be harvested 4 times a year, and local suppliers can produce it at a rate of 1000 kg/month. As the pyrolysis of elephant grass has been investigated in very small scales (in the 10^{-6} g to 1 g range) only, there is a need to design experiments and study this subject using a scale in which the bio-oil can be fully analyzed and further processed for economically viable bio-fuels.

In this work, fast pyrolysis of elephant grass is carried out in a fluidized bed reactor. The pyrolytic vapors are washed with water to remove acids and then condensed (mostly) via centrifugation. Three experiments were designed to compare the influence of different configurations of the gas-washing column system on the properties of the bio-oil.

2. MATERIALS AND METHODS

It is presented below some characteristics of the biomass and the sand used as fluidizing medium, a brief description of the experimental device, the characterization of the elephant grass and the products obtained from its pyrolysis.

2.1 Biomass and Fluidizing Medium

The raw biomass was provided in the form of dry stem (8-12% moisture) by Avicultura Santa Mônica Ltda - located in Parnamirim/RN/Brazil. The samples were chipped and then processed through a Marconi grinder to produce a granular material with diameter distribution between 0.8 and 1.2 mm. The elephant grass was also analyzed for C, H and N contents at CENPES/RJ/Brazil using a LECO CHN-1000 Elemental analyzer. The method of analysis was based on the ASTM D 5291. The amount of oxygen was determined by difference. The thermogravimetric analysis was carried out using a Shimadzu thermobalance model 51H. Nitrogen was used as carrier gas at a flow rate of 20 ml/min. The mass of the samples were 9.98, 10.02 and 10.03 mg and the heating rates were 5, 10 and 20°C/min.

Sand with particle diameter between 0.42 and 1.2 mm was provided by *BRASILMINAS Indústria e Comércio Ltda* – São Paulo-Brazil, and used in the pyrolysis reactor to help transfer heat to the biomass during the reaction.

2.2 Pyrolysis Plant

The processing plant promotes the fast degradation of biomass under low quantities of oxygen. The schematic flowsheet and layout of the plant are shown in Fig. 1.

The carbon steel pyrolysis reactor (06) consists of a 100 cm x 20 cm pipe and can process up to 40 kg/h of biomass. The 80 cm gas-washing column (11) has an inner perforated tube that allows acid extract (initially water as the solvent) to make direct contact with pyrolytic vapors thus washing them to remove acid compounds, which are recovered as acid extract (12). The remaining components are then condensed via

Sousa et al.; ACSJ, 14(2): 1-10, 2016; Article no.ACSJ.25843

centrifugation (10). The condensation system (10) decreases the residence time of the vapors during the reaction. All valves (V1, V2, V3 and several others) are controlled manually. Rotors and all other devices are activated via a control panel. The monitoring system records pressure and temperature data from the entire plant. All liquid product is collected in stainless steel reservoirs.

2.3 Pyrolysis Process

2.3.1 Operating conditions

The biomass flow varies between 20 and 35 kg/h. The mass of sand is 4.5 kg. The ideal operation temperature is 500°C; time of reaction usually varies between 45 and 90 minutes.

Table 1 shows the operating conditions for the experiments carried out during the elephant grass degradation via fast pyrolysis.

2.3.2 Procedure

Inert particles is placed in the reactor and then fluidized by the flux of air. The process starts by blowing air (01), which passes through a flow meter (02), a heat exchanger (03) and then enters the reactor (06). The reactor is heated electrically by heating elements that make the fluidized bed of inert particles reach the desired reaction temperature. K-type thermocouples and pressure sensors read data from the inside of the reactor and send them to a computer-aided data acquisition system so that the operator can make a decision as to stabilize both temperature and pressure. The variables that can change the thermodynamic conditions (P and T) inside the reactor are the flow rate of air (set by valve V1), the velocity at which the centrifuge (10) operates (set by rotor R2), the flow rate of gases (set by valve V3) and the biomass flow rate (set by rotor R1).



Fig. 1. Pyrolysis plant layout

	Table	1.	Oper	ating	conditions
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	Exp A	Exp B	Exp C
Temperature (°C)	480-520	480-520	480-540
Biomass flowrate (kg/h)	20–22.5	20-22.5	30–35
Reaction time (min)	82	55	85

2.3.3 Biomass feeding and reaction

After the fluidized bed is formed and the temperature is set, the biomass is fed (05) into the reactor, heat is transferred from the inert particles to the biomass, which is rapidly transformed into pyrolytic vapors and fine char particles.

2.3.4 Bio-oil production and phase interaction

Char acts as a vapor cracking catalyst so rapid separation from the pyrolytic vapors is important. Cyclones (08) were used in this work for char removal; however, some fines always pass through the cyclones and collect in the condensed bio-oil where they accelerate aging of the bio-oil [5].

The gaseous products consist of aerosols, true vapors and non-condensable gases (NCG). They all pass through the gas-washing column (11) where water (provided by the tank identified by 12 in Fig. 1) removes the light acid and phenolic compounds so secondary reactions are minimized. Those that are heavy are condensed at the top of the column by centrifugation (10) and then recovered in small stainless steel (09) to be analvzed collectors bv chromatography. Very light molecules such as CO, CO₂, H₂ and hydrocarbons from methane to propane that could not be condensed by any of the apparatuses described above were sampled using a customized air mattress, the rest of it

was sent to a burner and then the combustion product gases were released to the atmosphere. The burner operated at 680°C and was fed with air and liquefied petroleum gas (LPG) in excess.

The temperature and the rotation of the centrifuge are the key variables for an efficient condensation of pyrolytic vapors. Other combinations of variables also may affect the condensation temperature, which should not exceed 80°C.

To investigate the influence of the washing column on the bio-oil properties and quality, three different configurations (as shown in Fig. 2) were tested:

- Exp A The recuperation of the acid extract (AE) was deactivated; the washing column had a single pass for the pyrolytic vapors (V) to pass through. The column acted as a heat exchanger. The cooling fluid (CF) was water.
- Exp B Recuperation of the acid extract was activated so vapors and water would be in continuous and direct contact. The single-pass column was replaced by an eight-pass heat exchanger.
- Exp C Recuperation of acid extract was deactivated. The heat exchanger was the same as in Exp B.



Fig. 2. Gas-washing column configurations

AE recirculated continuously in Exp B and was measured by the rise (in cm) in the liquid level of the 200-liter tank (12). For Exp A and Exp C, the acid extract was collected at the bottom of the gas-washing column (11).

2.3.5 Mass balance

The actual amount of biomass processed was determined gravimetrically by subtracting the remaining inside the reservoir (05) from the initial amount fed. It was also possible to estimate the amount processed integrating the flow over time. The amount of gases produced was estimated by weighing the sample collected (the amount of air was known and then subtracted) after a given time period using the customized air mattress and then extrapolated to the reaction time. The yield of each phase was then determined from the equation below:

 $Biomass \rightarrow Bio-oil + Acid Extract + Char + Gases$

2.3.6 Product characterization

The bio-oil was characterized using the techniques and procedures below. The char was characterized via proximate analysis.

The chromatographic analyses were performed using a Thermo GC/qMS. The column used was an HP5ms (30 m x 0.25 mm x 0.25 μ m), the temperature of the injector and detector was 280°C, the injector split ratio was set to 50:1, and the flow rate was 1 ml/min of the Helium carrier gas.

The elemental composition was analyzed using a Flash EA 1112 Series CHNS-O analyzer.

The water content in the bio-oil was determined by Karl-Fischer titration.

The Viscosity was measured using a Brookfield viscometer model DV-I PRIME at 40°C with a sample volume of 16 ml.

Solids content was determined by vacuum filtration as suggested by Oasmaa and Peacocke (2001). Approximately 1-2 g of sample was washed with ethanol using Whatman No 2 paper filters. The residue was then air dried for 15 min and then dried in an oven at 105°C for 30 min.

The acidity was determined using a Mettler Toledo G20 Titrator. The methodology was based on ASTM D644-04 and consisted in adding the sample to a mixture of toluene, 2isopropanol and water and then titrating with KOH.

The pH was obtained using a Sartorius pHmeter model PB-11.

The HHV was determined using a Parr 1341 bomb calorimeter.

Proximate analysis for the char was performed in accordance with the ASTM D1762-84 method [7].

3. RESULTS AND DISCUSSION

The main products obtained from thermochemical pyrolysis of elephant grass were bio-oil, acid extract and char, as can be seen in Fig. 3.

The bio-oil is dark brown, viscous, acid and is composed of a very complex mixture of oxygenated hydrocarbons with an appreciable proportion of water from both the original moisture and reaction product Acid extract is composed mostlv [5]. of acetic acid [8], water (>60wt %) and small amounts of phenols. The char consists essentially of carbon; the oxygen content is usually low [5].

Each one of these products and the biomass were analyzed for composition and physicochemical properties.



Fig. 3. Elephant grass pyrolysis products. (a) Bio-oil; (b) Acid extract; (c) Char

3.1 Biomass Elemental Analysis

Table 2 shows that the content of carbon is high and almost the same as those obtained from elephant grass [4] and sugarcane straw [3]. The chemical composition and physical characteristics of the biomass can significantly affect the pyrolysis product distribution [8], as well as the products' compositions and properties.

High O/C molar ratios indicate that oxygen must be removed [5] and are used as reference to assess the effect of the pyrolysis on the elephant grass.

3.2 Biomass Thermogravimetric Analysis

The thermogravimetric analysis allowed to verify the evaporation of water starting at the room temperature and ending at 220°C when the initial mass was reduced by 11%, as can be seen in Fig. 4. From 220 to 800°C, the weight loss is due to the decomposition of cellulose, hemicellulose and lignin (4.17, 12.82 and 17.98% respectively, [11,12]).

3.3 Biomass Pyrolysis

3.3.1 Product yields

Table 3 presents the yields of each phase:

The highest acid extract yield was obtained without the heat exchanger, which means the direct contact was effective. The low bio-oil yield in Exp B can be justified by the low efficiency of the vapor condensation system. The highest biooil yield was obtained in Exp C. The least sophisticated configuration favored the production of gases, as the condensation was not effective enough.

The use of a heat exchanger inside the column has the advantage of the partial condensation of pyrolytic vapors (acid extract specifically) without the need to use large amounts of water as it was the case for Exp B.

The heaviest and most viscous pyrolytic vapor molecules usually condense before they reach the condensation system. This is specifically the case for semi-pilot plants and usually affects the mass balance, as part of the product cannot be collected.

Table 2. Elemental analysis of	different biomasses
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	This work	Elephant grass [9]	Sugarcane straw [10]
C (wt %)	41.85	41.10	41.60
H (wt %)	6.77	5.55	5.80
N (wt %)	0.72	1.78	0.45
O (wt %)	48.64	43.51	37.43
O/C (molar)	0.87	0.79	0.67
H/C (molar)	1.94	1.62	1.67



Fig. 4. Thermogravimetric analysis at three different heating rates

Sousa et al.; ACSJ	, 14(2): 1-10,	, 2016; Article no.ACSJ.25843	
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	Table 3.	Pyroly	ysis	products	yields
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	Ехр А	Ехр В	Exp C
Bio-oil (%)	11.0	3.0	28.2
Acid extract (%)	31.1	11.5	20.0
Char (%)	25.2	23.8	20.0
Gases (%)	31.1	25.8	27.3
Closure (%)	98.4	64.1	95.5

3.3.2 Bio-oil characterization

As reported by many authors [5-8,13-15], the presence of oxygen in many components of the bio-oil is the primary reason for the lower quality in comparison to hydrocarbon fuels. Oxygen is the cause of high viscosity, acidity, fast ageing (caused by polymerization reactions).

In this work, by comparing Tables 1 and 4, it can be observed that the pyrolysis reaction was successful in decreasing the oxygen concentration in all experiments. It was initially 48.64% (solid biomass) and now is 39.10 (Exp A), 34.49 (Exp B), and 38.81 (Exp C), which represent a decrease by 19.61%, 29.09%, and 20.21%, respectively. The sulfur content is known to be low [5,9].

As a consequence of oxygen removal, part of the hydrogen and small amounts of carbon were also removed. The H/C ratio found for the biomass was 1.64 and the closest the resulting bio-oil could get was 1.61 in Exp C. Although the configuration used in Exp B was the most successful in removing oxygen via direct contact with water, and the overall properties clearly indicates the oil is of better quality if compared to those obtained in Exp A and Exp C, it is still more

desirable to have high yields with the highest H/C ratio possible, which was not the case in Exp B.

It was also observed that the pH of the bio-oils were very low, mainly due to the presence of compounds such as acetic acid, as demonstrated elsewhere [16,17]. Acetic acid, phenols and alcohols also affect the acidity. For the three experiments under investigation, it was not possible to state clearly how these two properties were affected by the different configurations, as seen in Table 4.

The water content was 17.29% in Exp A and 10.14% in Exp C, which indicates that contact area for heat transfer was more efficient in removing water from the vapors than direct contact with water itself. Part of this water is originated from the biomass itself and from dehydration reactions. Other researchers [18,19] have found that the water content usually varies between 15 and 30% using similar apparatuses.

The bio-oil density, 1.2 g/cm^3 , is the same as those found in the literature. As discussed in [20], such values are common due to the presence of water in the liquid. The same authors report density values between 1.2 and 1.3 g/cm³ and water content from 10 to 15%.

The viscosity was measured after 05 days of storage for Exp A, which contributed for obtaining values as high as 249.0 cp. The values obtained in Exp B (78.4 cP) and Exp C (90.5 cP) are within the range reported in the literature [5,12]. The viscosity of a fuel plays an important role in the design and operation of the fuel injection system as well as on the quality of atomization

Та	ble	4.	Bio-oil	charac	terization

	Exp A	Exp B	Exp C
C (wt %)	53.10	57.37	52.74
H (wt %)	6.45	6.94	7.08
N (wt %)	1.49	1.20	1.37
S (wt %)	<0.3	<0.2	<0.3
O* (wt %)	39.10	34.49	38.81
O/C (molar)	0.55	0.45	0.55
H/C (molar)	1.46	1.45	1.61
$H_2O - KF$ (% m/m) TFCC	17.29	11.35	10.14
Acidity TFCC (mgKOH/g)	50.22	45.9	48.7
pH	3.8	4.1	4.0
Viscosity at 40°C (cP)	249.0	78.4	90.5
Density (g/cm ³)	1.21	1.20	1.20
Higher Heating Value (MJ/kg)	22.65	24.34	23.01
Solids content	2.55	1.32	3.18

*Determined by difference

0.98

0.54

0.12

0.45

0.36

	Ехр А	Exp B	Exp C
Acetic acid (%)	2.00	1.88	1.92
Syringol (%)	1.11	1.9	1.75
Vinyl-phenol (%)	0.98	0.60	0.65
Hexanoic acid (%)	0.85	0.71	0.89
Vinvl-quaiacol (%)	0.78	0.65	0.72

Table 5. Bio-oil chemical components

Table 6. Char proximate analysis

	Ехр А	Ехр В	Exp C	Elephant grass [9]	Sugar cane straw [10]		
Humidity (%)	4.79	5.21	4.99	-	1.2		
Volatile (%)	24.57	26.14	26.25	27.4	21.9		
Ash (%)	19.87	17.39	18.12	26.8	42.9		
Fixed carbon* (%)	50.77	51.26	50.64	45.8	35.1**		

*Determined by difference, **A different methodology was used

and its combustion [21]. It is important that the viscosity of bio-oil be then evaluated immediately after the experiment ends. Due to secondary reactions performed by oxygenate compounds, the viscosity is expected to change rapidly.

Methoxy-eugenol (%)

Trans-iso-eugenol (%)

Vanillin (%)

Solids in bio-oils are composed of char fines entrained out of the cyclone and the secondary char formed by secondary reactions of pyrolysis vapors such as re-polymerization and recondensation [5]. It can be seen in Table 4 that the recirculation of water (Exp B) helped capture a significant amount of char fines.

The Higher Heating Values varied between 22.65 and 24.34 MJ/kg, which is expected for bio-oil from lignocellulosic biomasses [5,10]. The elephant grass had an HHV of 17.29 MJ/kg, which indicates the pyrolysis was effective in transforming the biomass into a valuable liquid fuel [22].

Table 5 above presents the main components found in the bio-oils. The acetic acid is most abundant and suggests the bio-oil from Exp A was the most acid.

Compounds such as acetol, toluene, cresols, benzene, xylenes, naphthalene, levoglucosan and several others were also detected but in very small amounts and were not present in all samples. The literature on fast pyrolysis of elephant grass on bench scale is very scarce so it was not possible to build data to contrast and compare.

3.3.3 Char proximate analysis

The results obtained are within the range of values reported in the literature [9,10]. The high amounts of volatiles can be associated with the presence of bio-oil impregnated in the char. The results are shown in Table 6 above.

4. CONCLUSIONS

0.66

0.63

0.52

Elephant grass was pyrolyzed in a fluidized bed at 500°C coupled to a gas-washing column and a centrifuge condensation system. The products were bio-oil, acid extract, char and gases. Three experiment were designed to investigate the influence of different gas-washing column configurations on guality of the bio-oil. Results showed that the recirculation of acid extract improves the quality of the oil but at the same time decreases its yield. The simplest configuration with a single pass heat exchanger also produced interesting results. The use of a multi-pass heat exchanger proved to be the most effective because the resulting bio-oil was almost as good quality but with a much higher yield. Overall, fast pyrolysis was efficient in converting elephant grass into a liquid that is storable, transportable and has the potential to supply a number of valuable chemicals. In this manner, pyrolysis also provides an opportunity for the processing of agricultural residues, wood wastes and municipal solid waste into clean energy.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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Sousa et al.; ACSJ, 14(2): 1-10, 2016; Article no.ACSJ.25843

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