

## Chemistry

# Sustainable petrochemical plataform from Elephant Grass

## Plataforma petroquímica sustentável de Capim Elefante

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## ABSTRACT

The objective of this research is to demonstrate the potential of elephant grass (*Pennisetum purpureum* Schum) as biomass for obtaining other value-added byproducts, such as biofuels, lignocellulosic ethanol, adsorbents, among others. Through the pyrolysis process, biochar and bio-oil with average yields of 24.45% and 4.92% were produced, respectively. The main components found in the bio-oil were carboxylic acids (64.47%), followed by esters (19.74%). The carbohydrate adsorption percentage observed in the biochar was lower than that found in commercial activated carbon, which is desirable in the food industry. Through scanning electron microscopy (SEM), it was possible to observe that the biochar consists of non-uniform particles with numerous pores, suggesting its potential use as an adsorbent material. The (BET and LANGMUIR) surface area values did not yield significant results; however, there was an increase in pore size after the pyrolysis process. The glucose content found in this study was 70.33%, and the reducing sugar content was 14.24 g.L<sup>-1</sup>. Based on the theoretical yield calculations for 2G ethanol, it is possible to infer a potential production of 0.88 tons of 2G ethanol per hectare of elephant grass planted per year. The calculated theoretical yield for levulinic acid (LA) was 35.16%, and for formic acid (FA), it was 14.07%. It is worth noting that the use of such waste for the production of biochar and bio-oil represents a promising and cost-effective alternative, as elephant grass is currently highlighted among the most exploited crops for energy purposes.

**Keywords:** Pyrolysis; Bio-oil; Biochar; Ethanol; Elephant grass

## RESUMO

O objetivo desta pesquisa é demonstrar o potencial do capim-elefante (*Pennisetum purpureum* Schum) como biomassa para a obtenção de outros subprodutos de valor agregado, como biocombustíveis, etanol lignocelulósico, adsorventes, entre outros. Através do processo de pirólise, foram produzidos biochar e bio-óleo com rendimentos médios de 24,45% e 4,92%, respectivamente. Os principais componentes encontrados no bio-óleo foram os ácidos carboxílicos (64,47%), seguidos

dos ésteres (19,74%). A porcentagem de adsorção de carboidratos observada no biochar foi menor do que a encontrada no carvão ativado comercial, o que é desejável na indústria alimentícia. Por meio de microscopia eletrônica de varredura (MEV), foi possível observar que o biochar consiste em partículas não uniformes com numerosos poros, sugerindo seu potencial uso como material adsorvente. Os valores de área superficial (BET e LANGMUIR) não produziram resultados significativos; no entanto, houve um aumento no tamanho dos poros após o processo de pirólise. O teor de glicose encontrado neste estudo foi de 70,33% e o teor de açúcares redutores foi de 14,24 g.L<sup>-1</sup>. Com base nos cálculos teóricos de rendimento para o etanol 2G, é possível inferir uma produção potencial de 0,88 toneladas de etanol 2G por hectare de capim-elefante plantado por ano. O rendimento teórico calculado para o ácido levulínico (AL) foi de 35,16% e para o ácido fórmico (FA) foi de 14,07%. Vale ressaltar que o uso de tais resíduos para a produção de biochar e bio-óleo representa uma alternativa promissora e econômica, já que o capim-elefante é atualmente destacado entre as culturas mais exploradas para fins energéticos.

**Palavras-chave:** Pirólise; Bio-óleo; Biochar; Etanol; Capim-elefante

## 1 INTRODUCTION

Elephant grass is a perennial plant native to Africa, used as forage for livestock, and its scientific name is *Pennisetum purpureum Schum.* In appearance, the grass is similar to sugarcane but has narrower and shorter leaves compared to sugarcane, which can grow up to 6 meters tall. It is disease-resistant, adaptable, easy to propagate, and has significant potential as a precursor for the production of biochar (Adesemuyi et al., 2020; Ferreira et al., 2019).

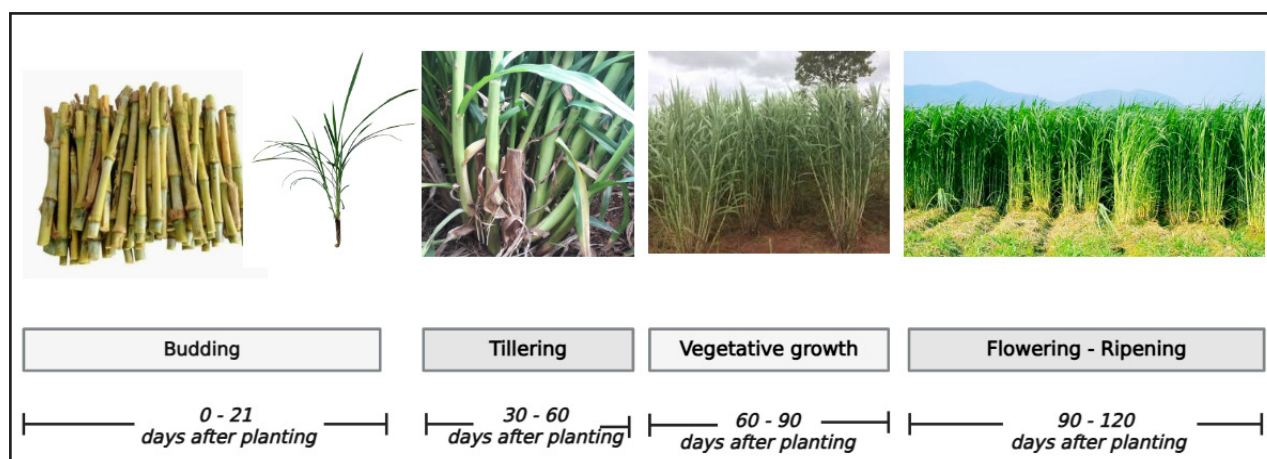
Elephant grass, as biomass, has garnered significant research interest as an alternative source of energy, as well as a producer of biochar and bio-oil. In this context, it has proven to be a valuable energy crop due to its high productivity, short production cycle, low requirement for soil nutrients, easy adaptation to different climatic conditions, and energy properties owing to its low ash content and high carbon and hydrogen content (Silveira Junior et al., 2022).

Elephant grass has been satisfactorily studied as biomass for the production of biochar and bio-oil through the pyrolysis process (Adeniyi et al., 2022). Biochar represents an emerging technology that has been recognized for its potential role in carbon sequestration, resulting in reduced greenhouse gas emissions, waste

management, renewable energy, soil improvement, increased crop productivity, and environmental remediation. The application of biochar to the soil influences physical and chemical properties such as pH, porosity, soil density, and water retention capacity (Manna et al., 2015; Lewoyehu, 2021).

Adsorption using activated carbon is employed for the purification of water, air, chemicals, and natural products, as well as for the removal of pollutants from both gas and liquid phases. It finds numerous applications in medical, industrial, and pharmaceutical processes. The use of activated carbon is primarily attributed to its relatively low cost compared to other adsorbents, widespread availability, high performance in adsorption processes, surface reactivity, and versatility in modifying its physical and chemical properties to synthesize adsorbents with very specific characteristics (Manna et al., 2015; Lewoyehu, 2021). Furthermore, Brazil has the potential to produce 1.2 Gt (Gt = 10<sup>9</sup> metric tons) of biochar and 2 Gt of bio-oils annually from elephant grass (Figure 1).

Figure 1 – Elephant grass life cycle



Source: Private collection, Santos (07, 2023)

Elephant grass is a fast-growing plant (80 to 100 days after planting and 60 days for regrowth). It is a perennial species with vegetative propagation, rapid leaf expansion in the first months after planting or cutting, and a high photosynthetic efficiency. It has

a high potential for dry matter production (approximately 40 t ha<sup>-1</sup>year<sup>-1</sup>) with efficient water use (Peterlini et al., 2013). One of the main challenges for the rapid expansion of elephant grass cultivation is related to its method of propagation, which is done through cuttings. This increases the cost of transporting and planting the forage, makes long-term storage impossible, and also hinders the widespread distribution of improved cultivars (Strezov; Evans; Hayman, 2008). Elephant grass regrowth can remain viable for more than 5 years, depending on agronomic management and the soil and climate conditions of each region (Pereira et al., 2021).

The BRS Capiaçú cultivar stands out due to its high production potential (50 t/ha/year of dry matter) and can be used for energy biomass production. Among the favorable attributes for this use are its high calorific value and the quality of the biomass, including the appropriate lignin and ash content. These characteristics make this cultivar an excellent source of raw material for bioenergy production (Samson, et al., 2005).

In this study, the BRS Capiaçú elephant grass will be analyzed, due to the advantages presented above, by the pyrolysis process and its properties, as well as the potential of biomass to obtain value-added by-products, such as biofuels, lignocellulosic ethanol, adsorbents, among others.

## 2 MATERIALS AND METHODS

### 2.1 Raw material

Samples of Elephant Grass (*Pennisetum purpureum*) BRS Capiaçú were collected from the experimental unit at EMBRAPA Pesca e Aquicultura in Tocantins (Figure 1), approximately 180 days after planting. After collection, the material was separated into leaves and stems, the leaves being selected due to their greater potential for ethanol production (Muniz, A. R.C.; Berdet, G.; da Silva, L., 2015). The leaves were dried in an oven at 85 °C for 72 hours, then processed in a knife mill (Wiley type TE-648), sieved

to 0.354 mm (45 mesh) (Silva, D. A., *et al.*, 2018) in the Laboratory of Chemistry at the Federal University of Tocantins, and stored in containers for subsequent analysis. All tests were conducted in duplicate.

Figure 2 – Collection site of elephant grass



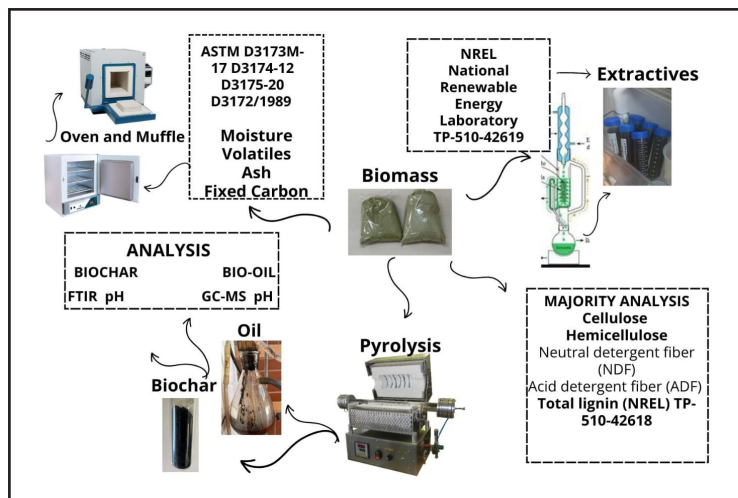
Source: Google Maps 10°08'47.7"S 48°18'46.1"W

## 2.2 Characterization of biomass

### 2.2.1 Chemical analysis

The samples were characterized according to the standardized procedures of the American Society for Testing and Materials (ASTM) (Figure 3). The moisture content was determined following ASTM D3173M-17 (American Society For Testing And Materials, 2017); the ash content was determined in accordance with ASTM D3174-12 (American Society For Testing And Materials, 2018); the volatile matter content (Mv) was determined according to ASTM D3175-20 (American Society For Testing And Materials, 2020). The fixed carbon content is an indirect measure and was determined by the difference between the ash content and the volatile matter.

Figure 3 – Flowchart of the methodology performed



Source: Private collection, Pimentel (9, 2023)

## 2.2.2 Determination of polysaccharides: cellulose and hemicellulose

For the determination of ADF approximately 0.5g of the dried and defatted sample was weighed and placed in a TNT fabric bag, then digested with 1.25% H<sub>2</sub>SO<sub>4</sub> using the MA-444/CI fiber digester at 90°C. For NDF approximately 0.5g of the dried and defatted sample was weighed and placed in a TNT fabric bag, and then digested with 1.25% NaOH using the MA-444/CI fiber digester at 90°C. After digestion, the samples were dried in an oven at 105°C until a constant weight was achieved. The moisture-free content of hemicellulose was estimated by the difference between NDF and ADF (A.O.A.C, 1995).

## 2.2.3 Determination of lignin content

2.0 g of previously dried biomass in an oven at 60°C for 24 hours were weighed. The samples were transferred to 100 mL beakers, and 10 mL of 72% v/v H<sub>2</sub>SO<sub>4</sub> was added. The mixture was placed in a water bath at 50°C for 7 minutes and continuously stirred with a glass rod. After this step, the samples were transferred to 250 mL Erlenmeyer flasks, and 40 mL of distilled water was added. The flasks were autoclaved for 15 minutes at 121°C. After cooling, the samples were filtered. The solid fraction

was placed in pre-dried and weighed porcelain crucibles, and the liquid fraction was stored. For lignin determination, the solid fraction was dried in an oven at 60°C for 24 hours and then weighed using an analytical balance. Subsequently, the sample was incinerated at 550°C. After cooling, the crucibles were weighed again. The liquid fraction was used to determine the concentration of glucose and total reducing sugars using enzymatic methods, specifically the glucose oxidase assay (enzymatic kit) and the DNS method (3,5-dinitrosalicylic acid) (NREL, 2011).

#### 2.2.4 Determination of total reducing sugars

A total of 150 µL of the liquid fraction sample, previously separated, were taken and placed in test tubes. Then, 2850 µL of DNS (3,5-dinitrosalicylic acid) solution were added to the tubes, and they were thoroughly mixed. The tubes were placed in a boiling water bath for 5 minutes and then removed and cooled in an ice bath. The readings were taken using a spectrophotometer (METASH UV/VIS 5100) at a wavelength of 540 nm. For the standard curve, dilutions of a 1.0 g/L glucose standard solution with distilled water were prepared in test tubes. After mixing the tubes for homogenization, a 1.0 mL aliquot was taken, and the DNS test was performed (Miller, G. L., 1959).

#### 2.2.5 Enzymatic method of glucose oxidase

For the determination of glucose, 10 µL of the sample were added to 2 mL Eppendorf tubes, and then 1 mL of GOD PAP reagent (Labtest enzymatic kit) was added to the tubes, which were mixed. The tubes were placed in a water bath at 37°C for 15 minutes, and then 1 mL of distilled water was added. Readings were taken using a spectrophotometer (METASH UV/VIS 5100) at a wavelength of 505 nm (Bergmeyer, H.U., 1985).

#### 2.2.6 Determination of the bioproducts

The maximum yields of levulinic acid (LA) and formic acid (FA) were estimated from the fermentable sugars obtained during the hydrolysis process using Equations

(1) and (2), as described by Rambo et al. (2015).

$$LA (\%) = 0,5 \times \text{hexoses } \% \quad (1)$$

$$FA (\%) = 0,2 \times \text{hexoses } \% \quad (2)$$

#### 2.2.7 Gas chromatography (GC-MS) of extractives

The chromatographic analyses were performed on a gas chromatograph, model 7890B, coupled to a mass spectrometer (GC-MS), model 5977B MSD, from Agilent Technologies. The capillary column used was the HP-5MS stationary phase, measuring 30 m x 250  $\mu\text{m}$  x 0.25  $\mu\text{m}$ , with helium as the carrier gas. The sample was manually and directly introduced into the equipment by injecting 1  $\mu\text{L}$  of the sample in split mode (split sample 1:50). The oil sample was diluted in hexane at a 1/100 (V/V) ratio. The system temperatures varied as described: injector, 155°C; oven temperature programming starting at 45°C for three minutes with a subsequent increase to 150°C, remaining for five minutes at a rate of 20°C/minute, ending at 250°C with a running time of 48 minutes; ionization source at 230°C and the quadrupole analyzer at 150°C.

#### 2.2.8 Calculation of the theoretical yield of second generation ethanol (2G)

To assess the theoretical potential for 2G ethanol production using elephant grass biomass, simulating the pretreatment, hydrolysis, and fermentation steps of the materials, calculations were performed based on the models proposed by Santos et al. (2012) and Almeida (2019).

#### 2.2.9 Pyrolysis

For the pyrolysis procedure, the ground samples were introduced into a fixed-bed quartz tubular reactor, with an external diameter of 10 cm and a length of 100 cm. A total of 80 g of biomass at a temperature of 500 °C, with a residence time of 30 minutes and a heating rate of 20 °C/min were used in batch mode. To promote the cooling of the condenser, a condensation system was attached to the end of the

reactor. This system consisted of a Friedrich-type condenser, a vacuum flask, two tubes, and a 20-liter water tank. Water vapor was used as the carrier gas, and the passage of steam allowed for the separation of biogas from other liquid products (bio-oil and acidic extract), which were retained in the vacuum flask. For the separation of bio-oil and the acidic extract, 20 mL of dichloromethane (Merck, Darmstadt, Germany) were used (Pedroza, 2017). After the pyrolysis process, the liquid residue (bio-oil) and the solid residue (biochar) were stored for subsequent analysis.

#### 2.2.10 Determination of bio-oil by GC-MS

The analysis of the bio-oils produced organically and aqueously was performed separately using a GC-MS instrument (model GCMS QP2010 Plus) equipped with a Rtx-5MS WCOT capillary column (30 m × 0.25 mm × 0.25 µm). Chromatographic separation was achieved following the temperature profile as follows: 60°C for 1 minute, followed by an increase of 7°C per minute until reaching 100°C, and then an increase of 4°C per minute until 320°C. The temperature was held at 320°C for 10 minutes. Helium was used as the carrier gas, with a flow rate of 1.90 mL per minute. Samples were injected into the system, and the ionization source was operated at an energy level of 70 eV.

#### 2.2.11 FTIR biochar and bio-oil

The spectra were obtained using a single-beam spectrometer from Agilent Technologies (model Cary 630 FTIR) in diffuse reflectance mode. The scanning range was from 500 to 4000 cm<sup>-1</sup> with increments of 0.5 nm, and each spectrum was obtained by averaging 32 scans. For each sample, three analyses were performed in triplicate, and the resulting average spectrum was used.

#### 2.2.12 Determination of pH in biochar and bio-oil

The pH measurement of the bio-oil was conducted using 10 mL with a digital pH meter (TECNAL, model 3MP) in a thermostatic bath maintained at a temperature

of 20 °C. For the pH of the biochar, 1g of the sample was mixed with distilled water in a ratio of 1:20 (w/w) to form a homogeneous suspension, and after 1.5 hours, the pH was determined.

#### 2.2.13 Adsorption of carbohydrates

Fifty milliliters of a 50% carbohydrate solution were added to 0.5g of biochar, and the mixture was kept in a water bath for 15 minutes. The mixture was centrifuged at 10,000 rpm and then filtered with 80 g/m<sup>2</sup> filter paper. The filtered solutions were diluted to 250 mg/L. A 0.6 mL solution of 0.5% phenol and 3.0 mL of concentrated sulfuric acid were added to 0.6 mL of the diluted 250 mg/L solution. To determine the carbohydrate content (CC), the analysis was performed at a wavelength of 490 nm (Essig et al., 1988).

#### 2.2.14 X-ray Diffraction (XRD)

The analysis was conducted using a Shimadzu XRD-700 X-ray diffractometer. Scanning was performed in the angular range of 5° to 50°, and the radiation source used was K $\alpha$ . The crystallinity index (CI) was calculated from the ratio of the crystalline peak height (I<sub>c</sub>) to the amorphous peak height (SEGAL et al., 1959).

#### 2.2.15 Scanning Electron Microscopy (SEM)

The morphology and physical structure of the elephant grass samples were subjected to SEM using a Shimadzu SSX-550 instrument from Kyoto, Japan. The samples were dried at 60°C until a constant weight was reached and then coated with an Au/Pd film. All images were generated at magnifications of 500× and 5000× and observed at 5 kV.

#### 2.2.16 Determination of specific surface area, volume, and average pore size (BET)

The elephant grass biochar samples (0.5 g) were subjected to Surface Area System and Porosimetry analysis using a Micromeritics ASAP 2010 instrument to

determine the N<sub>2</sub>-BET specific surface area and pore size distribution. The diameter range used as a standard was from 0.35 to 300 nm for pores and from 0.01 to 3,000 m<sup>2</sup>/g in the surface area range. The treatment temperature ranged from 30 to 350 °C. For comparison purposes, commercial activated carbon from the Synth brand was also used.

#### 2.2.17 Calorific Value

Knowing the contents of Carbon (C), Nitrogen (N), Hydrogen (H), Oxygen (O) and Ash (A), the upper calorific value (PCS) was estimated, according to NREL/TP-433-7965, calculated according to Equation (3).

$$\text{PCS (MJ.Kg}^{-1}\text{)} = 0.3491 \times \text{C} + 1.1783 \times \text{H} - 0.1034 \times \text{O} - 0.0151 \times \text{N} - 0.0211 \times \text{A} \quad (3)$$

### 3 RESULTS AND DISCUSSION

The results obtained for the physicochemical characterization of elephant grass biomass on a dry basis are presented in Table 1. For comparative purposes with other lignocellulosic biomasses, Table 1 presents the physicochemical compositions studied by other researchers for similar biomass, such as *Brachiaria* grass, elephant grass (from two different studies), and *Miscanthus* (a type of grass found in Europe). Knowledge of biomass composition is crucial in optimizing strategies for bioenergy, biofuels, and biochemicals production (Brosse et al., 2012).

It is observed that when compared to the extractive content found in *Brachiaria brizantha* grass, elephant grass has an advantage because the presence of high extractive contents is advantageous for energy production since these components have high calorific value. For a composite sample of elephant grass, an extractive content of 23.85% was obtained, which is close to the value found in this experiment (Peterlini et al., 2013). Extractives serve as metabolic intermediates, energy reserves, or are part of protective mechanisms; they are responsible for the color, odor, and

resistance to wilting of plants (Ortega-Santiago et al., 2016).

The lignin content of elephant grass has been shown to be higher compared to the results of research conducted with *Miscanthus* grass and *Brachiaria brizantha* grass. Lignin is not fermented to ethanol, and its presence is detrimental to fermentation. Therefore, in the viable process of ethanol production from lignocellulose, it should be extracted and can be used to obtain other products, such as pyrolysis (Ortega-Santiago et al., 2016).

Regarding the hemicellulose content, the elephant grass samples also demonstrated higher levels than the other biomass, consistent with this experiment. The same did not occur for the cellulose content, where the elephant grass samples in this study and the others mentioned obtained the lowest values. Hemicelluloses are sources of carbohydrates in lignocellulosic biomass, and the characterization of sugars in each species is necessary to identify the appropriate enzymes that increase the efficiency of the fermentation process and ethanol yield (Ortega-Santiago et al., 2016).

The moisture content of elephant grass biomass, at 8.53%, is considered low and relevant. This is because moisture has an inverse relationship with calorific value, meaning that higher moisture content leads to lower energy generation capacity, and vice versa. The ash content of 3.06% indicates that the biomass contains a moderate amount of non-combustible inorganic components. Literature mentions that values above 7% compromise the combustion process (Vale et al., 2011) because ash represents the part of the biomass that remains after burning. For biomass intended for energy generation, it is expected that the ash content is low; this way, there is greater energy utilization and less waste generation.

The fixed carbon percentage was lower than that found by Ferreira et al. (2019) in their research on elephant grass biomass. On the other hand, the fixed carbon percentage in *Brachiaria brizantha* grass was higher. The value of 6.9% indicates the amount of carbon present in the biomass that can be converted into energy during

combustion. Higher amounts of fixed carbon lead to higher bioethanol yields (Ortega-Santiago et al., 2016).

The volatile matter content (95.31%) directly affects the ease of burning biomass because a higher volatile content results in greater reactivity and, consequently, easier ignition. At the beginning of combustion, CO, CO<sup>2</sup>, H<sub>2</sub>, and other gases derived from volatile compounds are released through an exothermic process that facilitates combustion (Marasca et al., 2022). The amount of volatile matter found in elephant grass for this study showed a higher percentage compared to what other authors have reported, with the lowest percentage being in *Brachiaria brizantha* grass.

Table 1 – Results and Comparison Among Biomasses

Types of Biomass Components	Elephant Grass <sup>a</sup>	Elephant Grass <sup>b</sup>	<i>Miscanthus</i> <sup>c</sup>	<i>Brachiaria</i> <sup>d</sup>	Elephant Grass <sup>e</sup>
Extractives	26.6 ± 2.94	-	-	10.2	
Lignin	20.6 ± 0.75	27.15	14.09	17.4	8.4
Hemicellulose	24.54 ± 0.70	38.35	23.97	29.0	27.8
Celullose	25.26 ± 0.70	34.50	54.02	42.1	37.0
Moisture	13.21 ± 0.66	11.70	3.58	7.9	-
Ashes	3.06 ± 0.50	8.07	12.7	7.0	-
Volatile Material	95.31 ± 0.95	82.39	84.55	75.9	-
Fixed carbon	6.9 ± 0.86	9.53	-	16.8	
ART (g.L <sup>-1</sup> )	14.24	-	-	-	16.58
Glucose (%)	70.33	-	-	-	67.54

Source: Organized by the authors (2023)

Legend: a- Authors' data (2023); b- Adesemuyi et al., (2020); c- Rajeswari, G.; Murugan, M.; Mohan (2012); d- Samson, et al., (2005); e- Pereira, (2013).

The glucose content found in the present study (70.33%) and the reducing sugar content (14.24 g/L) were similar to the levels found by Pereira (2013) in the raw sample. Considering that elephant grass can yield up to 45 tons of dry matter per hectare per year (Fontoura et al., 2015) and sugarcane only yields 12 tons (Timung et al., 2015), it was possible to infer from the calculations of the theoretical yield of 2G ethanol that there is a potential to produce 0.88 tons of 2G ethanol per hectare of elephant grass planted per year, while sugarcane bagasse yields about 0.96 tons (Dantas; Legey;

Mazzone, 2013). The ethanol yield from sugarcane bagasse is higher, possibly due to the conversion of sugars not determined by the DNS method (Santos et al., 2012). It is worth noting that elephant grass is a grass that has the characteristic of adapting to any type of climate and soil, and it can be cultivated in many areas where sugarcane would not adapt, such as in the southernmost regions of Brazil (Martins; Andrade, 2021).

To demonstrate the potential of elephant grass, the yields of levulinic acid (LA) and formic acid (FA) were estimated using Equations (1) and (2), in which the results are presented and compared in Table 2 with values found by Swia Tek et al. (2020). Levulinic acid was selected as a promising chemical platform by the US Department of Energy due to its high reactivity and a wide range of functionalities based on a ketone and a functional carboxylic group (Badgujar et al., 2019). In addition to LA, formic acid has also received attention as a promising hydrogen carrier due to its relatively high hydrogen content (Kumar et al., 2018). The results obtained in this study were superior to those found in other biomass sources (Swia Tek et al., 2020), in relation to LA and FA income, highlighting the promising potential of elephant grass for the production of high-value bioproducts from fermentable sugars obtained through hydrolysis.

Table 2 – Theoretical and Experimental Yields of Organic Acids for Lignocellulosic Biomass

BIOMASS	YIELD (%)		REFERENCES
	LA	FA	
Elephant grass	35.16	14.07	Autores
Solid wood	20.1	10.06	Swia Tek et al., 2020
Spruce wood	21.7	8.5	Swia Tek et al., 2020
Miscanthus	8.2	29.7	Swia Tek et al., 2020

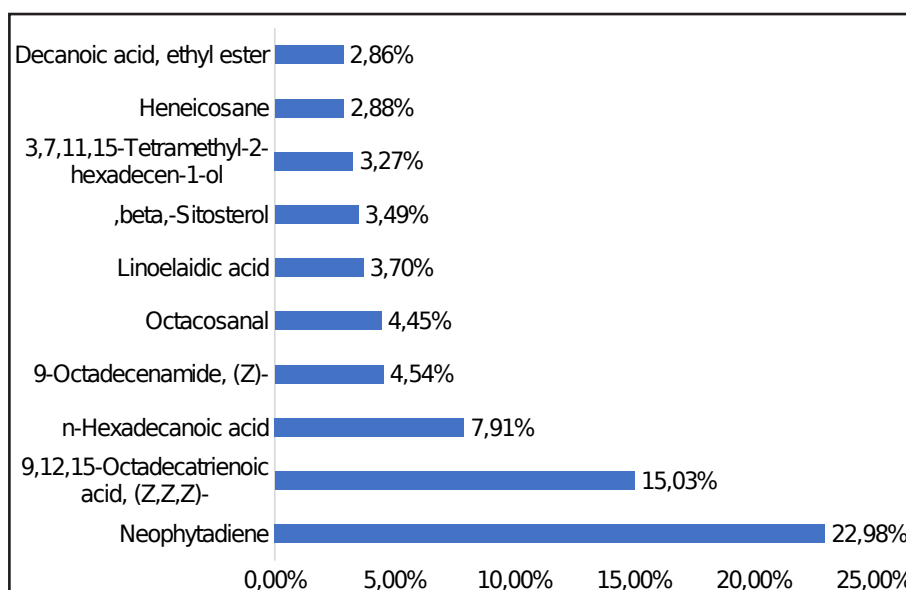
Source: Organized by the authors (2023)

The high extractives content (> 20%) justified the need for the analysis of its component oil (Figure 4), identifying a high content of acids (29.5%), followed by

hydrocarbons (22.98%), alcohols (6.76%), and amides (4.54%). As reported in the literature by Scopel et al. (2020), a total of 83 compounds were identified among the extracts considering leaves and stems of elephant grass via GC-MS. Among the most abundant compounds in the chromatogram of elephant grass extract is n-Hexadecanoic acid, commercially known as palmitic acid. It is known for its antioxidant, anti-inflammatory, hypocholesterolemic, nematocidal, pesticidal, and lubricating properties (Rajeswari et al., 2012). On the other hand, 9,12,15-Octadecatrienoic acid, also known as alpha-linolenic acid (ALA, 18n-3), is associated with the prevention of cardiovascular diseases and is essential for maintaining normal cellular membranes, brain function, and nerve impulse transmission (Martin et al., 2006). The unsaturated hydrocarbon neofitadiene belongs to the group of diterpenes and exhibits antioxidant and antifungal activities (Trevisan; Resende, 2020). In humans and animals, these compounds have pharmacological effects and can be used by the pharmaceutical, food, and cosmetic industries (Azmir et al., 2013).

Regarding the products obtained from pyrolysis, the biochar exhibited an average yield of 24.45% with a standard deviation of  $\pm 1.27\%$ . In contrast, the bio-oil yield reached  $4.92\% \pm 0.30\%$ . However, the low yield of bio-oil can be attributed to the pyrolyzer used, as the process was conducted with the use of only one condenser, in addition to heat loss between the tubular furnace and the reactor. Furthermore, a similar yield was observed for the biochar. Despite the low yield of bio-oil, the objective of its identification was to quantify this material and potentially optimize the process for obtaining the liquid phase, should high-yield, interesting products be detected in the future.

Figure 4 – Chromatogram of Elephant Grass Extract



Source: Organized by the authors (2023)

The list of the seven most abundant compounds in the chromatogram of the bio-oil (Table 3) demonstrates a high content of carboxylic acids (64.47%), followed by esters (19.74%). Carboxylic acids are organic compounds characterized by the presence of a carboxyl group (COOH). They are found in citrus fruits, vinegar, pharmaceuticals, and preservatives, and they have various industrial and laboratory applications. Additionally, carboxylic acids participate in reactions such as esterification, which is used in the production of flavorings and other compounds. The characteristics of compounds in this functional group vary depending on the size and structure of the carbon chain.

Tabela 3 – Chromatography of Elephant Grass Bio-Oil (GC-MS)

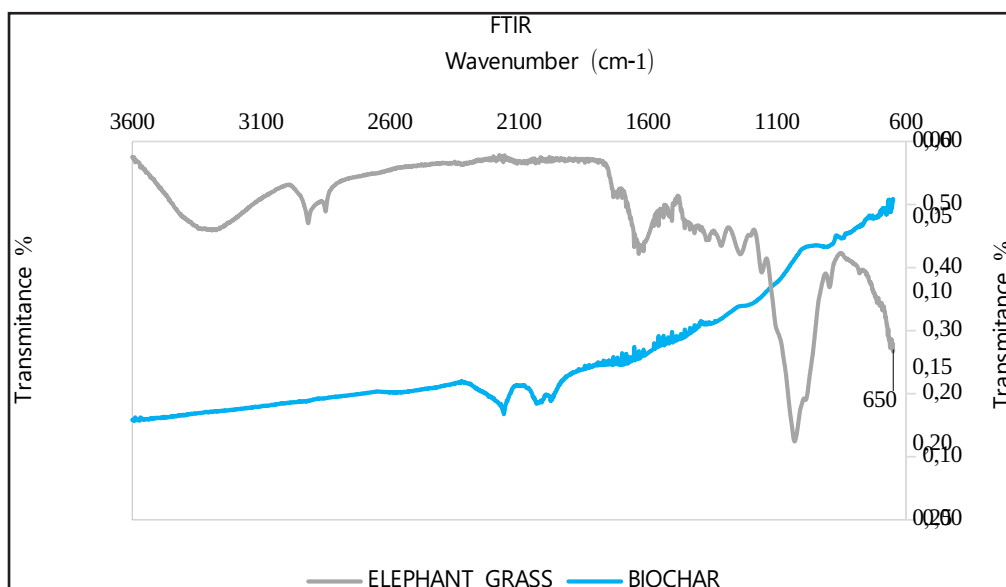
Height%	Name
53.19	1,3-Benzenedicarboxylic acid, bis(2-ethylhexyl) ester
12.43	Di-n-octyl phthalate
8.00	1,2-Benzenedicarboxylic acid, bis(2-methylpropyl)
3.28	Phthalic acid, 4-bromophenyl octyl ester
2.94	Methyl 2-hydroxydodecanoate
2.29	Sulfurous acid, 2-ethylhexyl hexyl ester
2.08	Sulfurous acid, 2-ethylhexyl hexyl ester

Source: Organized by the authors (2023)

After pyrolysis, the biochar and bio-oil displayed the following data, respectively: pH 9.50 and 3.80; and densities of 0.15 and 6.66. Typically, an increase in pyrolysis temperature raises the pH, although the extent of this increase can vary depending on the characteristics of the biomass, including inorganic content and moisture levels. The increase in pH is a result of the formation of oxides and hydroxides of elements like potassium (K), magnesium (Mg), sodium (Na), and calcium (Ca) during combustion (Jendoubi et al., 2011). Due to its alkaline nature, biochar can improve soil quality by helping to correct acidic soils. Therefore, it can be used as a liming agent to increase crop productivity (Marzeddu et al., 2021; Bordoloi et al., 2015).

The functional groups were analyzed, and the corresponding spectra are presented in Figure 5. The bands at  $3294\text{ cm}^{-1}$  and  $2911\text{ cm}^{-1}$  correspond to the stretching vibrations of O-H groups (alcohol, ketone) and C-H groups (alkyl). The small bands at  $1734\text{ cm}^{-1}$  and  $1631\text{ cm}^{-1}$  can be attributed to the acetyl groups or esters of hemicellulose present in the grass and to the aromatic bonds of lignin (Mohammed et al., 2015; Reddy et al., 2018). The absorption regions at  $1631\text{ cm}^{-1}$ ,  $1507\text{ cm}^{-1}$ , and  $1479\text{ cm}^{-1}$  correspond to the stretching of C-H bonds in the methyl, methylene, and methoxy groups, respectively, of lignin (Reddy et al., 2018). The absorption bands at  $1157\text{ cm}^{-1}$  and  $1023\text{ cm}^{-1}$  can be attributed to the stretching of the pyranose ring C-O-C and C-O stretching in lignin, cellulose, and hemicellulose. The observed frequency at  $650\text{ cm}^{-1}$  can be attributed to the stretching C-H bonds in the aromatic rings of lignin and cellulose (Mohammed et al., 2015). The samples exhibited results similar to lignocellulosic materials obtained in studies by Ferreira et al., 2019; Reddy et al., 2018; and Kamarullah et al., 2015.

Figure 5 – Infrared Spectrum of Elephant Grass and Biochar



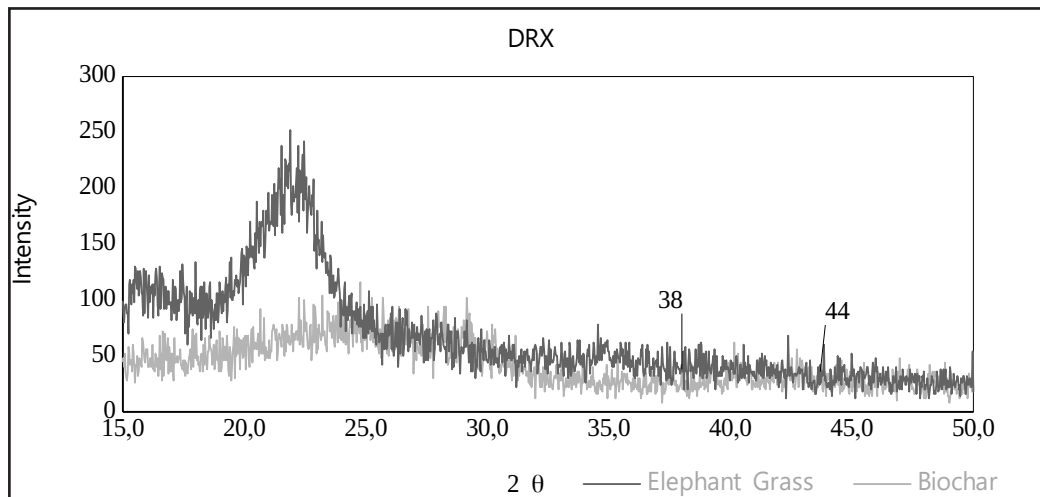
Source: Organized by the authors (2023)

For biochar, the confirmation of the presence of oxygen-containing functional groups can be observed in the C-O stretching of the carboxyl group at around  $985\text{ cm}^{-1}$  (Ferreira et al., 2019). The stretching of the C=C bond occurs in the range of  $739\text{ cm}^{-1}$  (Chen et al., 2012; Antonangelo et al., 2019). Bands in the regions of  $2022\text{ cm}^{-1}$ ,  $2158\text{ cm}^{-1}$ , and  $2322\text{ cm}^{-1}$  represent the stretching of the C=O group (Reza et al., 2020). FTIR analysis suggests that biochar contains carboxyl and carbonyl groups, which, in turn, function as chelating agents, increasing the ion exchange capacity of the biomaterial. This suggests that biochar could be used as a potential adsorbent (Ferreira et al., 2019; Adeniyi et al., 2022).

X-ray diffraction analysis was used to assess the crystallinity of elephant grass and biochar (Figure 6). The diffractogram of elephant grass revealed two broad peaks at  $17.45^\circ$  and  $21.89^\circ$ , corresponding to the characteristic crystalline structures of triclinic ( $I\alpha$ ) and monoclinic ( $I\beta$ ) cellulose, respectively (Ma et al., 2017). However, the biochar was produced at temperatures of  $500^\circ\text{C}$ , and these two peaks gradually merged into an extended peak, indicating a decrease in the crystallinity degree of cellulose in the biochar, possibly due to its degradation (Ma et al., 2019). The peaks at  $38.0^\circ$  and  $44.5^\circ$

correspond to the structures of silicon carbide (SiC) that were initially present in the biomass matrix and were retained in the biochar (Freitas et al., 2009). However, it was observed that the biochar exhibited an overlap of two peaks in the 44° region, corresponding to graphite and SiC (Zhang et al., 2017).

Figure 6 - X-ray diffractograms

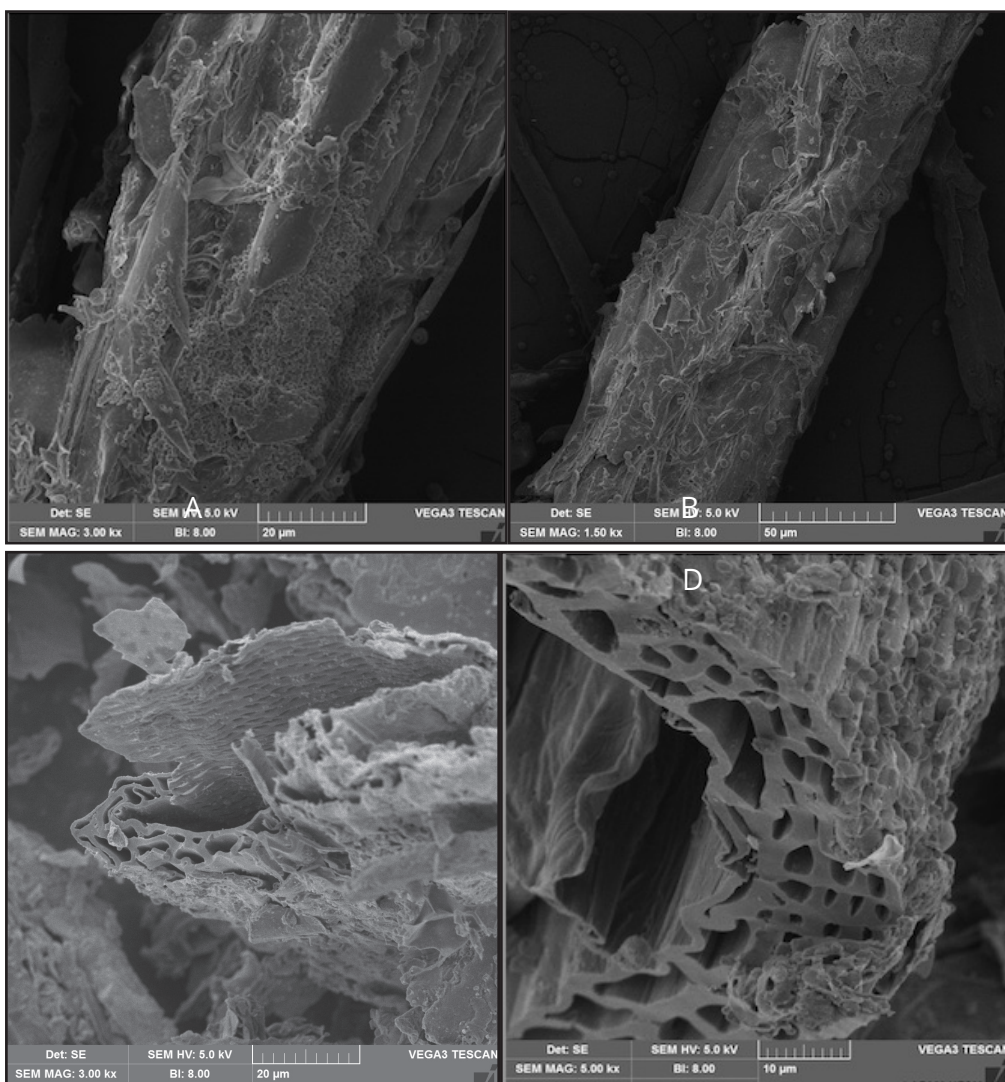


Source: Organized by the authors (2023)

The morphology of elephant grass and biochar is depicted in Figure 7. It can be observed that the biochar consists of non-uniform particles with a rough surface, relatively large surface areas, and numerous pores. The surface heterogeneity suggests the potential use of biochar as an adsorbent material (ONIFADE et al., 2020). The biochar (C and D) also exhibits numerous heterogeneous particles, as can be seen in the magnified micrograph. This difference in pore formation plays a significant role in the adsorption process, especially concerning the size of the adsorbate molecule (Mangwandi et al., 2020). Furthermore, interstitial pores, which are significantly pronounced, were also observed within the matrix. These pores can be considered indicators of good adsorption properties as they facilitate the adsorption of metallic species in the interstices (Montero et al., 2018). According to Sharma et al. (2001), the pores present in the analyzed material are possibly a result of the intense removal of volatile compounds during secondary pyrolysis reactions. However, elephant grass

exhibits particles with uniformly compacted filaments and larger surface areas (A and B) compared to biochar. The morphology of elephant grass obtained in this study is similar to what was found in the studies by Reza et al. (2020) on elephant grass pyrolyzed at temperatures ranging from 400–600 °C. The micrographs showed that the biochar retained morphological characteristics of the untreated biomass, indicating a strong influence of the biomass on the properties of the biochar (Al-Wabel et al., 2013).

Figure 7 – Micrographs of elephant grass and biochar



Source: Organized by the authors (2023)

Legend: (A) Micrografia do capim elefante 50 µm; (B) Micrografia do capim elefante 20 µm, (C) Micrografia do biochar 20 µm and (D) Micrografia do biochar 10 µm

Due to the presence of observed pores, adsorption tests were conducted to assess the adsorption capacity of the biochar obtained from elephant grass pyrolysis

compared to commercially used activated carbon. The comparison aims to analyze the biochar's ability to remove impurities and color in the food industry. It is known that activated carbon is a well-established adsorbent used to remove impurities from the environment without interfering with the composition since it has the ability to adsorb substances that may introduce undesirable taste, color, and odor (Venturini Filho, 2014). The percentage of reduction in carbohydrate content (CC) for commercial activated carbon was 15.19%, while for the biochar obtained in this study, it was 13.95% (Table 4). Therefore, the biochar adsorbs a smaller amount of carbohydrates compared to commercial activated carbon, which is a significant result, as the goal in the industrial process is not to adsorb glucose.

Table 4 – Adsorption of carbohydrates

	<b>Ads. (<math>\lambda=490</math> nm)</b>	<b>CC</b>	<b>Reduction (%)</b>
Glucose standard	0.60	-	-
Syrup solution	0.86	16.13	-
Commercial charcoal	0.73	13.68	15.19
Biochar from elephant grass	0.74	13.88	13.95

Source: Organized by the authors (2023)

The values of surface area (BET and LANGMUIR), volume, and size of micropores of the biochar are presented in Table 5. Elephant grass biochar did not show significant values of surface area (BET and Langmuir) when compared to commercial activated carbons, since the minimum recommended surface area is 150 m<sup>2</sup>/g (Nizamuddin *et al.*, 2016). An activation process would be recommended to increase this surface area value.

The pore size increased from 6.35 to 16.30 after pyrolysis, and this increase can be attributed to the breaking of hydrogen bonds or to the temperature and reaction time, which disrupted the fibrous structure of elephant grass and consequently produced an increase in the surface area of the biochar (Chen *et al.*, 2012). According to the recommendations of the International Union of Pure and Applied Chemistry (IUPAC), porous materials are classified based on pore size, considering adsorption properties

(Rouquerol et al., 1994). Based on the results (Table 5), there is a predominance of mesopores (between 2 and 50 nm).

Table 5 - BET, Langmuir surface, and pore size of elephant grass and biochar

	BET Surface Area (m <sup>2</sup> /g)	Langmuir Surface Area (m <sup>2</sup> /g)	Pore volume (cm <sup>3</sup> /g)	Pore size (nm)
Elephant Grass	1.194	1.8480	0.001897	6.353
Biochar from elephant grass	1.497	1.950	0.006100	16.304
Commercial activated carbon Synth	597.33	-	0.22	-

Source: Organized by the authors (2023)

The contents of C, H, N, O and ashes of the biochar of the BRS Capiáçu Elephant grass are presented in Table 6. The contents of C (64.15%) and N (2.03%) obtained were higher than the contents of C (43.4%) and N (0.46%) of Elephant grass found by Marafon *et al.* (2021), however, the H (2.08%) and O (31.74%) contents obtained were significantly lower than the H (6.07%) and O (46.3%) terores. The ash content did not show any difference. Grasses are generally characterized by a higher ash content and their use as fuel generally requires greater boiler maintenance due to the particular characteristics of this type of biomass (Zeng *et al.*, 2016). The calorific value of biochar helps to estimate its potential to be used as a fuel (Li and Chen, 2018). The PCS (Higher Calorific Value) of 21.47% was obtained for the biochar of the BRS Capiáçu Elephant grass, demonstrating the potential of these residues to be used as solid fuels, since the value recorded is similar to that of materials used for the same purpose.

Table 6 – Physical and chemical characteristics of Elephant Grass biochar

Biochar Elephant grass	Elemental composition (%)				Ash (%)	PCS (MJ.Kg <sup>-1</sup> )
	C	H	N	O		
	64.15	2.08	2.03	31.74	3.06	21.47

Source: Organized by the authors (2023)

Legend: C- Carbon; H- Hydrogen; N- Nitrogen; O- Oxygen and PCS- Higher Calorific Value

## 4 CONCLUSIONS

Elephant grass has proven to be an advantageous biomass in obtaining value-added by-products due to its ability to be converted into biochar, bio-oil, ethanol, and organic acids (both above 14%). In the products resulting from pyrolysis, biochar had an average yield of 24.45%, while bio-oil achieved a yield of 4.92%.

Furthermore, grass has practical viability as a source of raw material for generating thermal energy due to its desirable qualitative characteristics, mainly its high calorific value (21.47%).

Therefore, its use in purification processes is promising can be affirmed that it is efficient due the glucose removal (14.0%). Elephant grass biochar has proven to be highly promising as a low-cost raw material for the production of an adsorbent, with the suggestion of an activation process to increase its surface area

These results demonstrate the possibility of integrated utilization of pyrolysis products within a biorefinery concept.

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