



Syzygium cumini residue pyrolysis and chemical dye adsorption using the Plackett–Burman technique

R. N. Lôbo¹ · M. M. Pedroza² · C. S. A. Rezende² · L. R. A. Oliveira³ · E. C. S. Paz² · C. F. P. R. Paschoalato³

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Abstract

Prunings of the *Syzygium cumini* tree were used to produce activated carbon, which was used in a chemical dye adsorption experiment. The characterization analyses of the biomass revealed the following experimental data: moisture 6%, volatile matter 82% and ash content of 0.7%. During the pyrolysis tests of the residue, the following input variables were analyzed: reactor temperature (350–650 °C), carrier vapor flow in the reactor (4–8 mL/min), furnace heating speed (25–45 °C/min), particle size (6–12 mm), heating temperature of the residue with steam (110–130 °C) and residence time of the residue with heated steam (20–40 min). The charcoal obtained presented the following characteristics: (a) pH of 8.5, (b) C/H ratio = 31, (c) pHP CZ = 7.0 and (d) iodine number equal to 1162 mgI₂/g. The experimental methodology of Plackett & Burman was used for the methylene blue adsorption test, with the following variables: test temperature (20–30 °C), dye concentration (5–15 mg/L), adsorption time (20–60 min), adsorbent mass (0.5–1 g), shaking table speed (71–167 rpm), pH of the dye solution (6.5–8.5) and activated charcoal particle size (0.45–2 mm). The yield of adsorbent material was approximately 16% at 650 °C. The maximum adsorption capacity of the carbon was approximately 1.5 mg/g. The iodine number of the material produced in this research was much higher than the value established by the standards for adsorbent materials.

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✉ M. M. Pedroza
mendes@ifto.edu.br

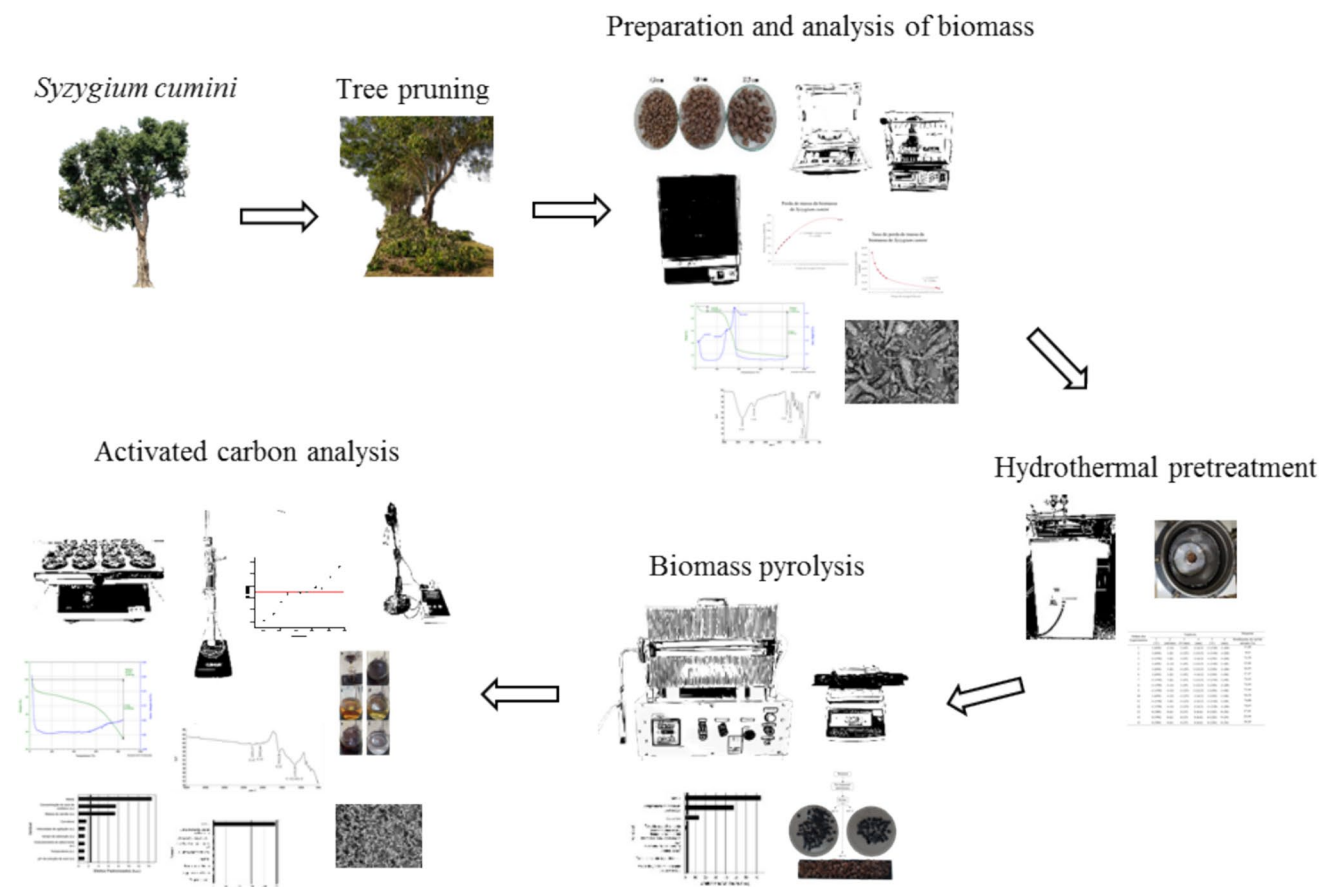
¹ Department of Environmental Engineering, Federal University of Tocantins (UFT), Palmas, Brazil

² Department of Electrical Engineering, Federal Institute of Tocantins (IFTO), Palmas, Brazil

³ Institute of Chemistry, University of Ribeirão Preto (UNAERP), Ribeirão Preto, Brazil



Graphical abstract



Keywords Pyrolysis of tree prunings · Adsorbent material · Plackett–Burman methodology · Chemical dye removal

Introduction

Waste from pruning and removing urban trees in public and private areas located in urban developments can generate serious urban problems when they are not properly used, being discarded in inappropriate places such as illegal landfills, or sent to sanitary landfills, contributing to operational overload and increasing management costs, as well as loading sanitary landfills with this organic waste, which requires a shredding procedure (Calahorra et al. 1992; Martín-Lara et al. 2019). Waste resulting from pruning public trees includes trunks, logs, branches, stumps, roots, and plant waste from urban centers resulting from the maintenance of parks and gardens.

Syzygium cumini is a species native to India, which has been introduced to several countries in Africa, and Latin America (Ayyanar and Subash-Babu 2012; Araga et al. 2017; Singh and Tirkey 2021). In Brazil, it is found in all regions of the country. In addition, it is cultivated

in several subtropical regions worldwide, especially California, Florida, Algeria, and Israel (Thiagamani et al. 2024), resulting in significant volumes of pruning residues that remain underexplored from a technological and environmental perspective.

Tree prunings can be subjected to heat treatment to produce activated carbon, mainly through thermochemical conversion processes such as pyrolysis, a material with physical–chemical characteristics that develop large surface areas ideal for adsorbing pollutants in liquid and gaseous media (Menéndez et al. 2004; Gu et al. 2014; Sarvade et al. 2016; Sharma et al. 2024). This process enables the transformation of lignocellulosic residues into value-added carbonaceous materials, contributing to waste valorization strategies.

Activated carbon has a variety of possible uses, in which it can be used as a filter bed to remove toxic substances and heavy metals from domestic and industrial effluents as well as organic contaminants and dyes from aqueous solutions (Mamaní et al. 2019; Li et al. 2008). The adsorption



performance of activated carbon is strongly dependent on both the properties of the precursor biomass and the operational conditions applied during its production.

Carbonaceous materials related to the solid fraction of the thermochemical conversion process have high carbon contents, ideal for adsorbing inorganic and organic compounds. The production of activated carbon from plant biomass is well-suited for metal removal in medicine (adsorption of poison), gas cleaning, water purification, and the food industry (Viotti et al. 2019). Several studies have reported the successful use of agricultural and forestry residues as precursors for activated carbon production, demonstrating their potential for environmental remediation applications (Román et al. 2006; Fischer et al. 2019).

Despite these advances, limited attention has been given to urban tree pruning residues, particularly those derived from *Syzygium cumini*. Moreover, there is a lack of systematic studies that simultaneously evaluate multiple pyrolysis parameters and biomass pretreatment conditions using statistical experimental designs, as well as their direct influence on adsorption performance (Chafyq et al. 2021; Ahmad et al. 2023).

This research aims to obtain activated carbon from urban tree pruning of the species *Syzygium cumini* in a fixed-bed reactor, addressing this research gap, aiming at the optimization of variables related to the pyrolysis processes: (1) pyrolysis temperature, (2) water vapor flow rate, (3) heating rate of reactor, (4) biomass particle diameter, (5) heating temperature of the biomass in steam humidity, and (6) contact time of biomass with wet steam. The activated carbon produced was subsequently applied in the adsorption of a chemical dye, with an experimental design using the Plackett–Burman Matrix being applied during the tests, in order to identify the most influential factors and clarify the novelty of applying a multivariable optimization approach to steam-assisted pyrolysis and dye adsorption using *Syzygium cumini* residues. This study was conducted in the city of Palmas, state of Tocantins, Legal Amazon Region, Brazil, in 2024.

Materials and methods

Biomass preparation

The waste used in this study was tree pruning of the species *Syzygium cumini*, collected from public flowerbeds in the city of Palmas—TO, resulting from urban cleaning procedures in the city. The biomass samples were divided into three diameters, for the execution of the experimental planning as one of the independent variable factors, following the granulometric sequence of 6 mm

(MESH—1/4"), 8 mm (MESH—5/16") and 12 mm (MESH—1/2") (Elhenawy et al. 2024).

Biomass pretreatment and pyrolysis

Biomass from *Syzygium cumini* pruning underwent a thermochemical pretreatment using a steam-based hydrothermal environment, herein referred to as steam pretreatment. Unlike conventional hydrothermal carbonization, the biomass was not in contact with liquid water, being exposed exclusively to water vapor under autogenous pressure.

The process was conducted in a vertical autoclave (PHOENIX, model AV), with a maximum allowable pressure of 2.8 kgf/cm² and a total capacity of 100 L. Water occupied approximately 1/5 of the autoclave volume, generating saturated steam, while the biomass remained physically separated from the liquid phase throughout the treatment.

Operational parameters such as temperature, pressure, and residence time were selected based on previous studies involving steam-assisted thermal pretreatment of lignocellulosic biomass (Gollakota et al. 2018; Scapini et al. 2021).

Biomass was inserted into the reactor according to the granulometry established in the experimental planning adopted for each pyrolysis test. Thermal conversion was performed in a stainless steel fixed-bed reactor measuring 60 cm in length and with an external diameter of 5.0 cm. The reactor was heated by a reclining split furnace (FLYEVEER brand, model FE50RPN and line 05/50), and operated in batch mode. The pyrolysis atmosphere was established by the continuous injection of superheated water vapor, which acted as a non-oxidizing carrier medium and heat-transfer agent, characterizing a steam-assisted pyrolysis process. The water vapor was generated in an autoclave at 133 °C under a pressure of 2 kgf/cm² (Fig. 1). The reactor temperature, carrier vapor flow rate, and heating rate were adopted according to the experimental design described above. The duration of pyrolysis for each experiment was programmed with an initial time of 15 min at a temperature of 150 °C and 30 min for the temperatures of the experimental design adopted (Zhang et al. 2018, 2021).

The chemical characteristics of the biomass and charcoal obtained in the process were determined through the analytical techniques presented in Table 1.

A Plackett–Burman (PB) experimental design was used to evaluate the effects of six independent variables on the production of activated carbon during biomass pyrolysis (Chafyq et al. 2021; Ahmad et al. 2023). The independent variables are: (a) pyrolysis temperature (350, 500 and 650 °C), (b) carrier vapor flow rate in the pyrolysis reactor (4, 6 and 8 mL/min), (c) heating rate of the pyrolysis reactor



Fig. 1 Parts of the biomass pyrolysis system used in this research

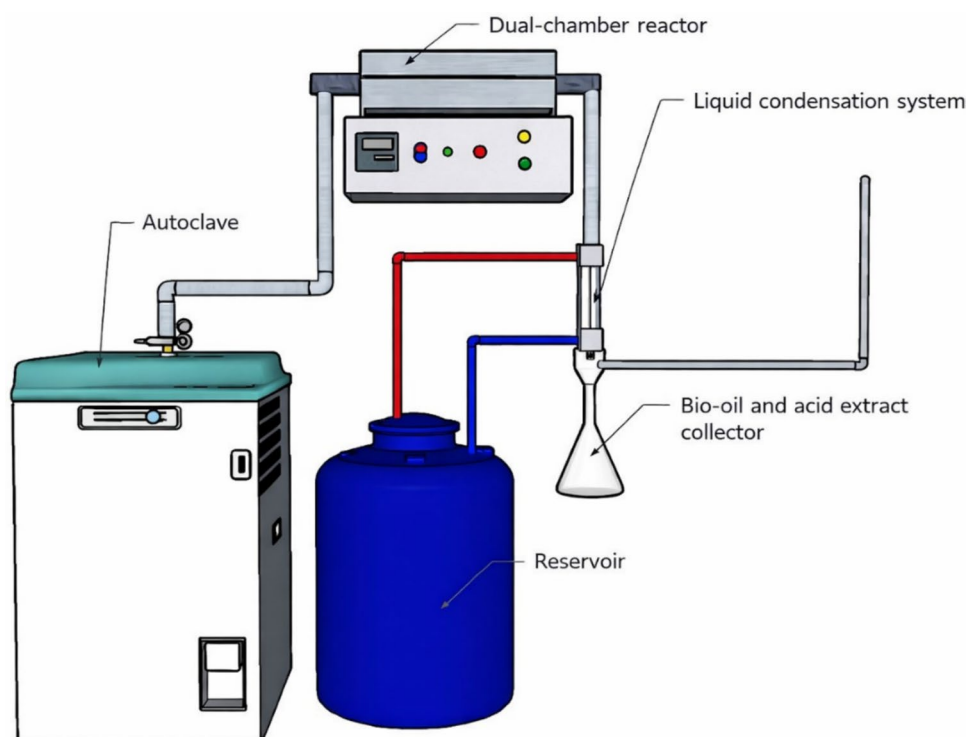


Table 1 Analytical techniques used in the characterization of biomass and activated carbon

Analytical parameter	Analytical method
Moisture (%)	ISO-589–1981
Volatile matter (%) ^a	ISO-5623–1974
Ash (%) ^a	ISO-1171–1976
Fixed carbon (%) ^a	By difference
Carbon (%) ^b	Elemental analysis
Hydrogen (%) ^b	Elemental analysis
Oxygen (%)	By difference
Nitrogen (%) ^b	Elemental analysis
Determination of the Zero Load Point	Volumetric analysis
Iodine index (mg/ g)	NBR 11834:1991
Thermal characterization	TGA/DTG
Functional groups (%)	FTIR
Micrographs	Scanning Electron electron microscopy (SEM)

^aDry basis

^bDry and ash free basis

(25, 35 and 45 °C/min), (d) biomass particle diameter (6, 8 and 12 mm), (e) biomass heating temperature in wet steam (110, 120 and 130 °C) and (f) contact time of the biomass with wet steam (20, 30 and 40 min).

Adsorption test

An experimental design was carried out in this study to verify the effects of seven independent variables on the methylene blue adsorption test (Pernyeszi et al. 2019; Mohammadzadeh et al. 2023; Wang et al. 2024). The independent variables are: (a) temperature (20, 25 and 60 °C), (b) methylene blue concentration (5, 10 and 15 mg/L), (c) adsorption time (20, 40 and 60 min), (d) activated carbon mass (0.5, 0.75 and 1 g), (e) stirring speed (71, 119, 167 rpm), (f) pH of the methylene blue solution (6.5, 7.5 and 8.5) and (g) adsorbent particle size (0.4, 1.2 and 2 mm). The effect of seven factors on the methylene blue adsorption test under study was verified through a variable selection design. An experimental design was applied using the Plackett–Burman Matrix (Chander et al. 2024; Borham et al. 2024), with 12 tests and 03 repetitions at the central point, totaling 15 experiments. The following responses were considered in this experimental design: (a) removal efficiency, (b) filtrate absorbance and (c) adsorption ratio Q .



Table 2 Results of gravimetric and elemental analyses of *Syzygium cumini* biomass

Biomass	Proximate analysis				Ultimate analysis				References
	moisture (%)	Volatile matter (%)	Fixed carbon (%)	Ash (%)	C (%)	H (%)	N (%)	C/H	
<i>S. cumini</i> pruning	5.97	81.90	17.44	0.66	45.04	5.57	0.19	8.09	This research
Coconut fiber	7.6	81.5	8.92	1.98	45.1	6.8	47.7	6.63	(Paz et al. 2021)
Tree pruning	6.65	77.05	13.01	3.29	48.52	6.95	0.24	6.98	(Mamaní et al. 2019)
Olive tree pruning	3.80	72.62	21.89	1.69	51.16	6.70	1.12	7.64	(Gu et al. 2014)
Olive tree pruning	5.36	82.53	6.84	5.27	51.27	6.75	0.70	7.60	(Ronda et al. 2016)
<i>S. cumini</i> seeds	53.49	78.98	21.02	2.09	44.5	4.98	0.77	8.94	(Singh et al. 2021)
Açaí seeds	—	76.31	21.18	2.51	48.21	6.69	1.60	7.21	(Sato et al. 2020)

Results and discussion

Chemical characterization of biomass (*Syzygium Cumini*)

The characterization of the biomass under study was achieved through gravimetric methods (moisture, volatile material, fixed carbon and ash), and instrumental methods (Fourier transform infrared spectroscopy—FTIR, ultimate analysis, and thermogravimetric—TG and DTG). Table 2 presents the results obtained in the characterization of *Syzygium cumini* pruning.

The results of moisture (5.97%), volatile matter (81.90%), and fixed carbon (17.44%) of *Syzygium cumini* pruning are within the range reported for other lignocellulosic biomasses. The ash content (0.66%), however, is considerably lower than most values found in the literature, which is advantageous for thermochemical conversion processes.

Syzygium cumini pruning presented the following elemental composition: carbon (45.04%), hydrogen (5.57%) and nitrogen (0.19%), values comparable to those of other forest residues. The low nitrogen content is particularly beneficial, as it minimizes the formation of nitrogen-containing gaseous compounds during pyrolysis. The C/H ratio of 8.09 is consistent with values reported for lignocellulosic precursors commonly used for carbonaceous materials.

The high volatile matter content (> 80%) is a key property for activated carbon production, since the release of volatile compounds during pyrolysis promotes the development of pore structures through the formation of micro- and mesopores. In addition, the carbon content of approximately 45% ensures the formation of a stable carbonaceous matrix capable of withstanding subsequent activation treatments. Furthermore, low ash content reduces the likelihood of pore blockage and improves the accessibility of active sites. Therefore, the combined characteristics of high volatile matter, adequate carbon content and low ash content indicate

that *Syzygium cumini* pruning is a suitable and promising precursor for activated carbon production.

The thermogravimetric analysis of the biomass of *Syzygium cumini* was performed using nitrogen gas at a flow rate of 40 mL/min and a heating rate of 10 °C/min. The analysis lasted 83.04 min until the temperature of 850 °C exceeded 80% mass loss.

In the temperature range of 25 to 149 °C, moisture and light volatile materials were lost, reducing 0.2856 mg (8.837%) of the initial mass (Fig. 2). The moisture content obtained in the proximate analysis (5.97%) is within the margin of error of ± 2.3 of the moisture loss reported in the thermogravimetric analysis.

From the temperature of 154 °C, the biomass begins another mass loss curve, increasing a reduction of 2.386 mg, which corresponds to 73.82% of the initial mass. Thus, the initial mass of the thermogravimetric process is 3.232 mg and the final mass is 0.5582 mg at a temperature of 846 °C, such that the residue corresponds to 17.27% of the initial mass. Regarding the immediate analysis of the volatile material and moisture content of the *Syzygium cumini* biomass, it resulted in 86.97% and the total percentage of mass loss from the thermogravimetric analysis was 82.657%.

The maximum derivative of mass loss occurred between 357 °C and 600 °C. The initial mass loss, up to approximately 148.86 °C, was due to moisture loss. The other peaks of the DTG curve are attributed to the degradation of hemicellulose and cellulose. In this case, the decomposition of hemicellulose and cellulose was observed in two peaks, the first of which is related to hemicellulose, whose thermal decomposition occurs at lower temperatures. The second peak in this temperature range is attributed to the decomposition of cellulose, where due to its greater thermal stability, decomposition occurs at higher temperatures (Martín-Lara et al. 2019). Lignin degradation occurred between temperatures of 357.40 °C and 846 °C due to its greater stability than the other biomass components (Menéndez et al. 2004; Sutar et al. 2017).



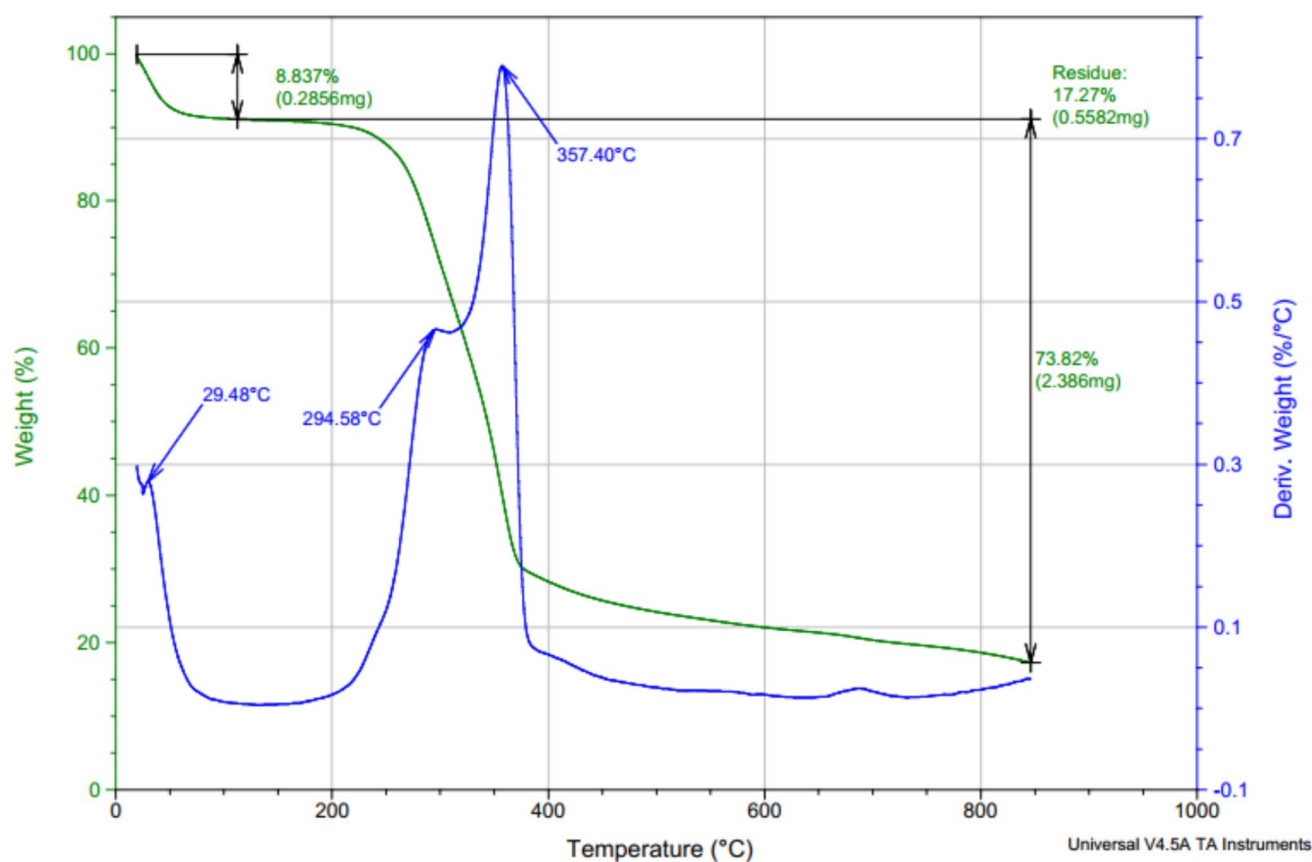


Fig. 2 TG and DTG curves of *Syzygium cumini* biomass

Table 3 Temperature range and mass variation obtained from TG/DTG of *Syzygium cumini* pruning

Events	T_i (°C)	T_{max} (°C)	T_f (°C)	ΔT_v (°C)	ΔW_v (mg)	DTG_{max} (%/°C)
1	25	29.48	148.86	123.86	0.2856	0.2787
2	154.16	294.58	362.36	208.2	1.7697	0.4651
3	357	357.4	600	243	0.7438	0.7898

T_i , Initial temperature; T_f , Final temperature; T_{max} , Temperature at which the derivative of the function is zero; ΔT_v , Temperature difference, $\Delta T_v = T_f - T_i$; ΔW_v , Mass loss in the event; DTG_{max} , Maximum mass loss rate of the event

Table 4 Temperature ranges for devolatilization of organic compounds and groups

Component groups	Moisture	Carboxylic group	Phenolic group	Ether	Cellulose	Other groups with oxygen
Temperature range (°C)	up to 150	150–600	300–600	up to 600	up to 650	150–900

Table 3 shows the temperature range and mass variation, which shows that the second event maintained the highest loss, reaching 54.75% with a loss rate of 0.4651%/°C, while the third event had 23.01% mass loss with a rate of 0.7898%/°C.

In the thermal test, through the thermogravimetric curve, it is observed that the devolatilization of organic compounds, functional groups, and other compounds are related to defined temperature ranges, as observed in Table 4. The biomass of *Syzygium cumini* is made up of several components, such as moisture, cellulose, hemicellulose,



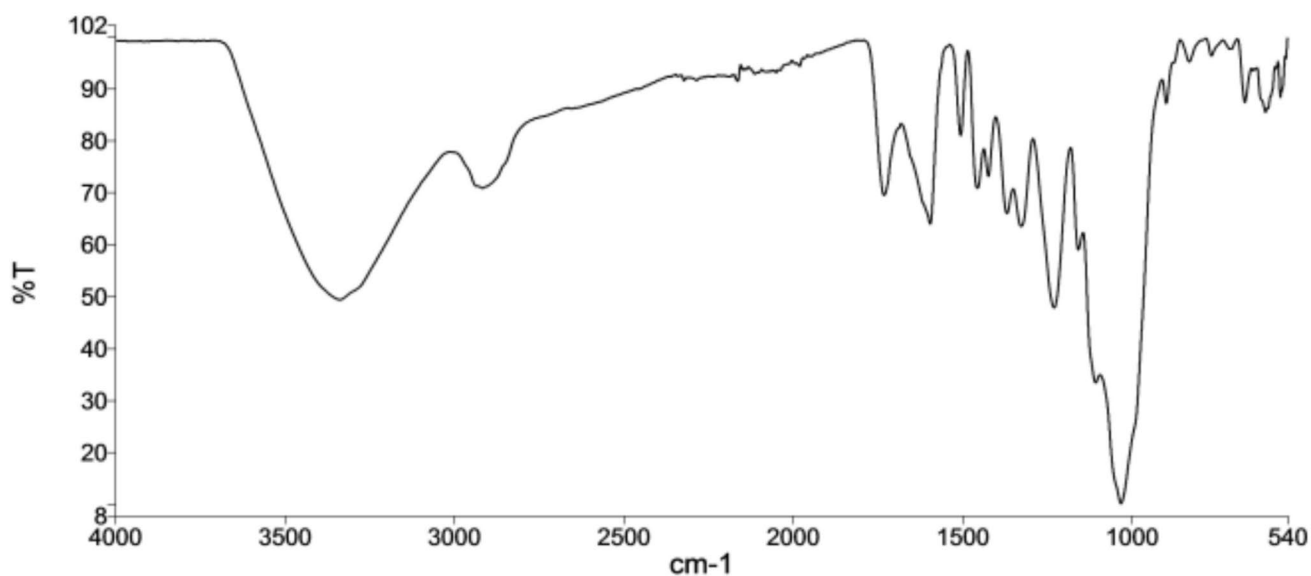


Fig. 3 FTIR spectrum of *Syzygium cumini* biomass

lignin, as well as inorganic salts responsible for the ash content detected in the material.

During the infrared spectroscopy instrumental test, spectral bands between 4000 and 400 cm^{-1} were detected (Fig. 3), with 24 peaks being observed with evidence of the functional groups found in the structure of the *Syzygium cumini* biomass, allowing the investigation and identification of its chemical composition.

A broad band of 3336.32 cm^{-1} was detected due to the functional group of axial deformation OH originating from water, phenols, carboxylic acids, and alcohols [Rodríguez et al. 2019]. The same can be confirmed in the thermogravimetric analysis of the biomass, which shows an event of devolatilization of moisture, and other elements of origin of hemicellulose, cellulose, and lignin. Another band at 2913.98 cm^{-1} , which is typical of axial deformation CH found in the structure of cellulose, hemicellulose and lignin. The analytical test of the *Syzygium cumini* biomass recorded deformations in the 1800–1600 cm^{-1} wavelength regions and transmittance around 69% referring to the axial deformation of C=O and C=C of the aromatic ring, groups found in hemicellulose, and lignin.

The functional groups identified in the instrumental infrared spectroscopy analysis are among the alcohols, phenols, alkenes, alkanes, ethers, and carboxylic acids present in the chemical structure of cellulose, hemicellulose, and lignin, elements belonging to lignocellulosic materials.

Experimental design plackett & burman: biomass pyrolysis

Table 5 presents the results of the Plackett–Burman (PB) experimental design for the thermochemical conversion of *Syzygium cumini* pruning biomass, with charcoal yield from the pyrolysis process considered as the response variable. The design was conducted by a PB of 12 tests and 3 at the central point, considering 6 factors, namely: pyrolysis reactor temperature, carrier vapor flow rate in the pyrolysis reactor, pyrolysis reactor heating rate, biomass particle diameter, biomass heating temperature in wet steam and biomass contact time with wet steam.

The yields of charcoal obtained in the pyrolysis process of *Syzygium cumini* pruning varied between 11.86 and 75.99%. The experiments carried out at a reactor temperature of 350 °C resulted in high solid yields, above 70%. At this temperature, which can be classified as severe torrefaction or the transition zone between torrefaction and low-temperature slow pyrolysis, a low degree of devolatilization of the main biomass components (cellulose, hemicellulose and lignin) was observed. This behavior indicates the good thermal stability of the biomass under mild thermal conversion conditions.

At a pyrolysis reactor temperature of 650 °C, an average charcoal yield of 15.87% was observed, a value well below that obtained during the pyrolysis of olive prunings (32% charcoal) at a temperature of 600 °C (Mamaní et al. 2019; Ronda et al. 2016), which demonstrates that the temperature of the pyrolysis reactor is a relevant factor for the yield and characteristics of the activated carbon.



Table 5 Results of the PB design experiments for pyrolysis of *Syzygium cumini* biomass

Essays	Variables						Planning response
	1 (°C)	2 (mL/min)	3 (°C/min)	4 (mm)	5 (°C)	6 (min)	Activated carbon yield (%)
1	1 (650)	−1 (4)	1 (45)	−1 (6.3)	−1 (110)	−1 (20)	11.86
2	1 (650)	1 (8)	−1 (25)	1 (12.5)	−1 (110)	−1 (20)	16.9
3	−1 (350)	1 (8)	1 (45)	−1 (6.3)	1 (130)	−1 (20)	71.59
4	1 (650)	−1 (4)	1 (45)	1 (12.5)	−1 (110)	1 (40)	15.84
5	1 (650)	1 (8)	−1 (25)	1 (12.5)	1 (130)	−1 (20)	16.93
6	1 (650)	1 (8)	1 (45)	−1 (6.3)	1 (130)	1 (40)	17.47
7	−1 (350)	1 (8)	1 (45)	1 (12.5)	−1 (110)	1 (40)	74.43
8	−1 (350)	−1 (4)	1 (45)	1 (12.5)	1 (130)	−1 (20)	72.93
9	−1 (350)	−1 (4)	−1 (25)	1 (12.5)	1 (130)	1 (40)	75.99
10	1 (650)	−1 (4)	−1 (25)	−1 (6.3)	1 (130)	1 (40)	16.24
11	−1 (350)	1 (8)	−1 (25)	−1 (6.3)	−1 (110)	1 (40)	74.08
12	−1 (350)	−1 (4)	−1 (25)	−1 (6.3)	−1 (110)	−1 (20)	78.65
13	0 (500)	0 (6)	0 (35)	0 (8.0)	0 (120)	0 (30)	27.01
14	0 (500)	0 (6)	0 (35)	0 (8.0)	0 (120)	0 (30)	25.94
15	0 (500)	0 (6)	0 (35)	0 (8.0)	0 (120)	0 (30)	26.26

Variables: (1) Reactor temperature, (2) carrier vapor flow rate, (3) Reactor heating rate, (4) Biomass diameter, (5) Biomass temperature in wet steam, (6) Contact time of biomass in wet steam.

Table 6 Regression coefficients of the PB design with 95% confidence level of the pyrolysis of *Syzygium cumini* biomass

Name	Effect	Standard error	Calculated t	p-value
Mean	45.24	0.61	74.45	0
Curvature	−37.68	2.72	−13.86	0
Reactor temperature (x_1)	−58.74	1.22	−48.33	0
Inert gas flow rate (x_2)	−0.02	1.22	−0.02	0.9884
Reactor heating rate (x_3)	−2.44	1.22	−2.01	0.0842
Biomass diameter (x_4)	0.52	1.22	0.43	0.6807
Biomass temperature in wet steam (x_5)	−0.10	1.22	−0.08	0.9357
Contact time of biomass in wet steam (x_6)	0.86	1.22	0.71	0.4997

Regarding the central point of the experiment, at a reactor temperature of 500 °C, an average charcoal yield of 26.40% was determined; another study with lignocellulosic materials at this temperature obtained a 24.03% charcoal yield (Mamaní et al. 2019).

The pyrolysis reactor temperature obtained a p -value equal to 0 (zero), but the other factors had p -value estimates above the significance level of 0.05. The effect of the reactor temperature factor was considered as the main effect of the PB variable selection planning, followed by the pyrolysis



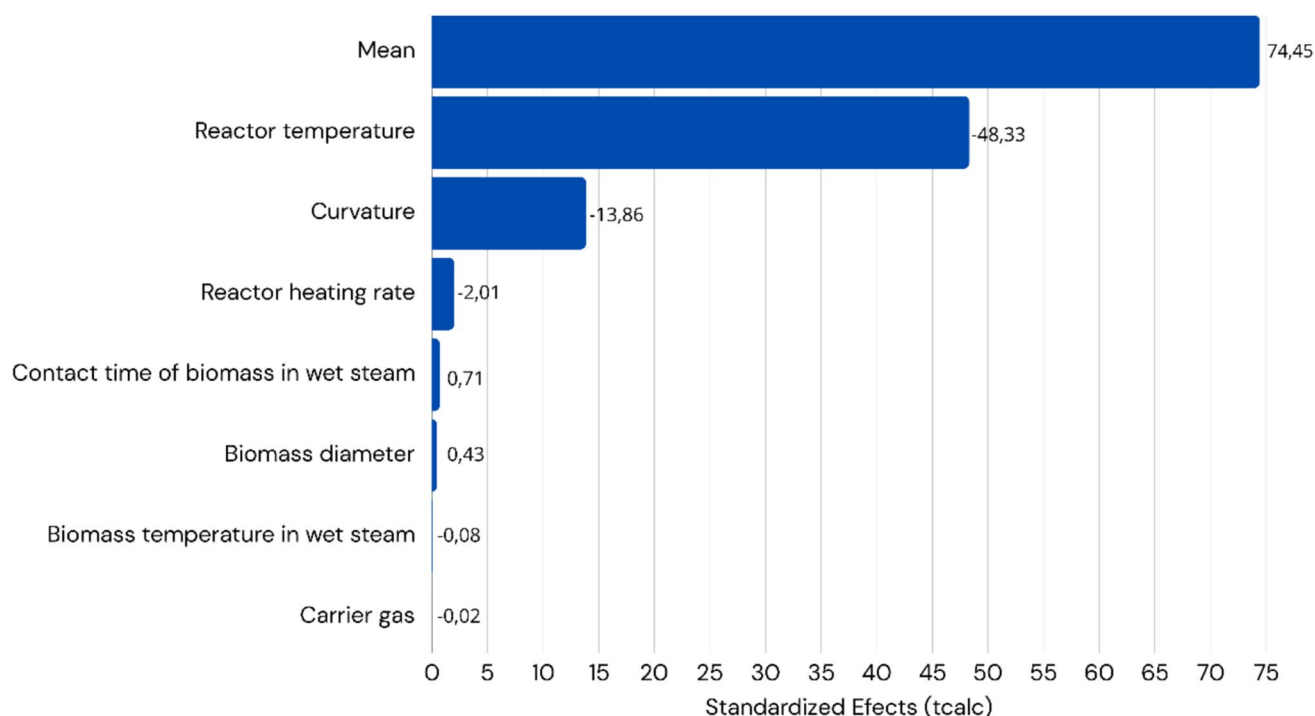


Fig. 4 Representation of the Pareto Diagram obtained in the PB planning with a 95% confidence level of the pyrolysis of the biomass

Table 7 Results of proximate and ultimate analysis of activated carbon from *Syzygium cumini*

Pyrolysis temperature (°C)	Proximate analysis				Ultimate analysis				References
	Moisture (%)	Volatile material (%)	Fixed carbon (%)	Ash (%)	C (%)	H (%)	N (%)	C/H	
350	9.4	81.8	7.9	0.9					This research
500	5.5	44	49.1	1.4					
650	7.7	25	64.8	2.5	83.02	2.67	0.17	31.09	
400	4.52	24.99	54.77	15.72	61.27	5.47	1.53	11.20	(Martín-Lara et al 2019)
500	5.21	12.65	59.42	22.72	63.52	3.44	1.23	18.47	(Martín-Lara et al 2019)
400	3.93	38.54	50.22	7.31	59.18	5.96	1.07	9.93	(Rodríguez et al. 2019)
600	6.04	35.58	53.88	4.50	83.58	2.85	0.29	29.33	(Calahorra et al.1992)

reactor heating rate (p -value of 0.0842), the closest value to the 95% confidence level of significance in relation to the other non-statically significant factors. The highest p -values of the planning were for the carrier vapor flow rate in the pyrolysis reactor (0.9884), and biomass heating temperature in wet steam (0.9357) factors, as shown in Table 6.

The reactor temperature was the only factor that presented a significant negative effect on charcoal production (−58.74), as shown in Fig. 4. Pyrolysis reactor heating rate (−2.44), biomass contact time with wet steam (0.86), biomass particle diameter (0.52), biomass heating temperature in wet steam (−0.10) and carrier vapor flow rate in the pyrolysis reactor (−0.02) did not interfere significantly in charcoal production

through the thermochemical conversion of biomass used in this research.

Chemical characterization of activated carbon

Proximate and ultimate analysis of activated carbon

The activated carbon from *Syzygium cumini* pruning was characterized using laboratory procedures in triplicate for moisture, volatile matter, fixed carbon, and ash contents.



Table 7 presents the results of the proximate analysis as well as the carbon, hydrogen, and nitrogen contents.

At a temperature of 350 °C, the raw material did not undergo devolatilization of the elements, maintaining a volatile material content of 81.8%, a value close to that resulting from biomass, which was 81.9%. The fixed carbon and ash contents were 7.9 and 0.9%, respectively.

The moisture contents of the activated carbons produced in this study were 5.5% at 500 °C and 7.7% at 650 °C, both below the maximum value of 8% established by NBR 11834:1991. These values are also consistent with those reported for activated carbons in other studies.

The ash content represents the inorganic residues after the elimination of oxygen, carbon, hydrogen and nitrogen (Menéndez et al. 2004). The ash is composed of aluminum, silicon, magnesium, calcium, potassium, sodium, zinc and other minerals (Gu et al. 2014). The charcoal produced in this research obtained ash contents lower than 2.6%, values well below those achieved in other studies with charcoal from lignocellulosic materials such as olive pruning (Martín-Lara et al. 2019; Calahorra et al. 1992).

The charcoal obtained from the biomass of *Syzygium cumini* maintained the proportional increase in carbon from 45.04% of the biomass to 83.02% of the activated carbon. The other elements suffered a reduction due to devolatilization.

The carbon/hydrogen ratio (C/H) for the elemental analysis of charcoal produced at 650 °C was 31.09, a value close to that identified in another study (C/H of 29.33) that performed pyrolysis of olive pruning biomass at 600 °C (Calahorra et al. 1992). An increase in the C/H ratio was observed when compared with the result for *Syzygium cumini* biomass (C/H equal to 8.09), which indicates the occurrence of the aromatization process and chemical alteration of the biomass during the thermal process (Soares et al. 2014).

Hydrogen potential (pH)

The proportionality of hydroxide (OH^-) and hydronium (H^+) in the biomass of *Syzygium cumini* resulted in a pH of 6.64, a value higher than that obtained in studies of other species such as Ipê, Jatobá, Tauari, Muiracatiara and Guajuvira, with pH values ranging from 4.53 to 5.9.

With the pyrolysis process, the activated carbon obtained an increase in pH in relation to the biomass of *Syzygium cumini*, going from an acidic material to the basic pH range, which can be confirmed in the C/H ratio, which increases the concentration of carbon and reduces hydrogen. The carbon produced at a temperature of 500 °C obtained a pH of 8.02, and at a temperature of 650 °C, a pH of 9.02, due to the carbon maintaining a reduction in oxygen content. Activated carbon is in alkaline zones allowing the adsorption surface

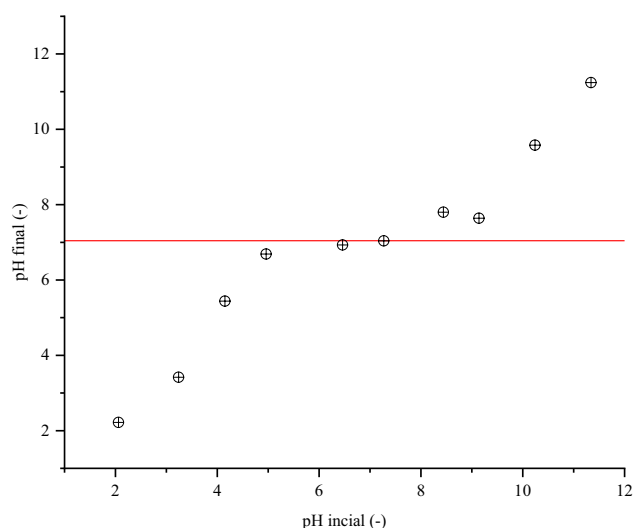


Fig. 5 Obtaining the pHPCZ of activated carbon produced at 650 °C from *Syzygium cumini* biomass

to maintain behavior for anion exchanges (Mamaní et al. 2019).

Point of zero charge (pH_{PCZ})

Point of Zero charge (pH_{PCZ}) of activated carbon shows the types of electrical charges present in the material. The method used consists of determining, in 11 tests, the final pH versus initial pH values (Mamaní et al. 2019) for carbon produced at 650 °C, in which the neutral surface charge of the activated carbon is determined (Fig. 5).

The carbonaceous material from the pyrolysis of *Syzygium cumini* pruning presented a pHPCZ in the pH range around the neutral of 7.042 ± 0.003 , a result obtained by drawing a straight line perpendicular to the ordinate axis, keeping the neutral charge point balanced for the 11 experiments. Other studies on the characterization of charcoal obtained a Point of Zero charge on average around 9.43, with the adsorbent material obtained at temperatures of 400, 450, 500 and 550 °C using lignocellulosic residues [20, 26]. For solutions with pH lower than the Point of Zero charge of activated charcoal, the surfaces of the material become positively charged, allowing anion adsorption. However, for medium pHs higher than the pHPCZ of charcoal, the surface will be negatively charged, allowing cation adsorption. And this chemical action occurs through the electrostatic attraction between the charges on the surface of the activated carbon, and the anionic or cationic groups in the medium (Viotti et al. 2019; Moruzzi et al. 2014).



Table 8 Result of iodine number of activated carbon from *Syzygium cumini*

Characteristic of activated carbon	NBR 11834:1991 (minimum limit)	Pyrolysis temperature (°C)	
		500	650
Iodine number (mg de I ₂ /g)	600	1162.07	1189.43

Iodine number

The iodine number test followed the procedures established by NBR 12073:1991 to determine the amount of iodine adsorbed in milligrams per gram of activated carbon obtained by the thermochemical treatment of *Syzygium cumini* pruning. The carbon was inspected to verify whether the material meets the specific conditions of NBR 11834:1991, which establishes limits for the adsorption characteristics of the material.

The analyzed carbons were obtained at the central point temperature of the experiment (500 °C) and at the upper level (650 °C). The product obtained at 500 °C had an iodine adsorption capacity of 1162.07 mgI₂/g. The carbon at 650 °C maintained an iodine number 3% higher than the carbon at 500 °C, reaching 1189.43 mgI₂/g (Table 8). NBR 11834:1991 establishes a minimum value of 600 mgI₂/g for the iodine number as the required characteristics for activated carbon used in the adsorption of impurities.

Although increasing pyrolysis temperature resulted in a decrease in activated carbon yield, this behavior is consistent with the enhanced devolatilization and structural rearrangement of the carbon matrix at higher temperatures. At lower temperatures (e.g., 350 °C), the process resembles

torrefaction, leading to higher solid yield but limited pore development due to incomplete thermal decomposition of lignocellulosic components.

At higher temperatures (500–650 °C), the intensified release of volatile matter promotes the formation and widening of micropores, improving adsorption capacity as evidenced by the iodine number. This illustrates the classical trade-off between carbon yield and activation degree, where higher temperatures favor porosity development and adsorption performance at the expense of gravimetric yield.

The marginal increase in iodine number between 500 and 650 °C suggests that most of the microporous structure was already developed at 500 °C, with additional temperature primarily contributing to further burn-off rather than significant gains in adsorption capacity.

The activated carbon in this study demonstrated an average iodine number 48.5% higher than the minimum limit established as a requirement for acceptance as activated carbon for the treatment of water for public supply. Other studies with other lignocellulosic materials presented the following iodine numbers: 402.5 mgI₂/g (Fagnani et al. 2019), 990 mgI₂/g (Calahorra et al. 1992), 892 mgI₂/g (Moruzzi et al. 2014) and 831.1 mgI₂/g (Román et al. 2006). Materials with high iodine adsorption capacity have characteristics of activated carbon with a large number of micropores with diameters of less than 2 nm and mesopores of 2 to 50 nm.

The iodine number is an important parameter in defining the relative level of activation and indicative of the adsorption capacity. Fisher et al. (2019) determined the iodine number of commercial activated carbons from pine (P) and babassu coconut shell (CCB) residues, obtaining values between 672.1 and 831.1 mgI₂/g.

Fig. 6 SEM micrograph image of the activated carbon produced in this research

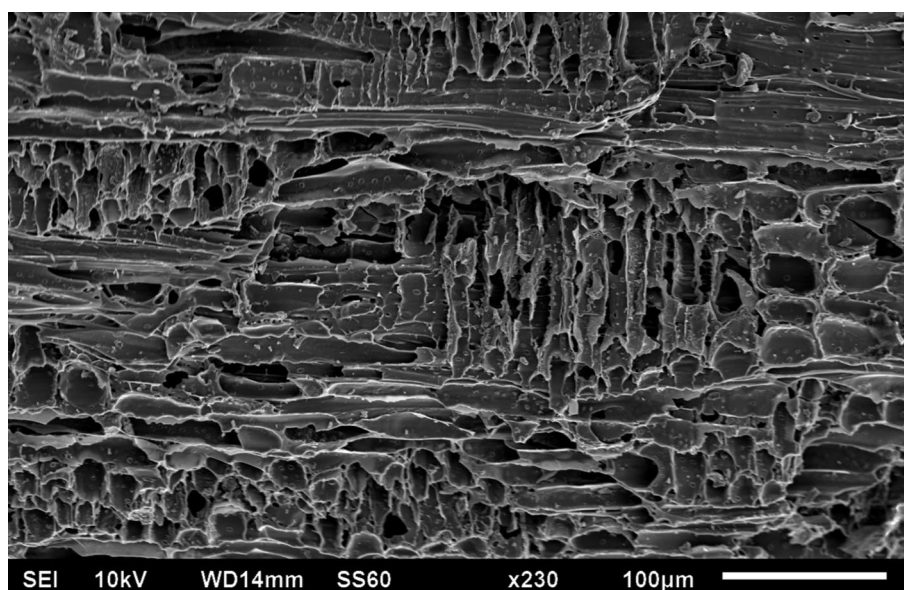


Table 9 Results of experiments used in PB planning—methylene blue adsorption test

Essays	Variables							Planning responses	
	1 (°C)	2 (mg/L)	3 (min)	4 (g)	5 (rpm)	6 pH	7 (mm)	Adsorption capacity (mg/g)	Efficiency (%)
1	30	5	60	0.5	71	6.5	2	0.48	95.95
2	30	15	20	1	71	6.5	0.5	0.49	98.61
3	20	15	60	0.5	167	6.5	0.5	1.48	98.49
4	30	5	60	1	71	8.5	0.5	0.15	91.71
5	30	15	20	1	167	6.5	2	0.49	97.28
6	30	15	60	0.5	167	8.5	0.5	1.48	98.49
7	20	15	60	1	71	8.5	2	0.49	97.48
8	20	5	60	1	167	6.5	2	0.16	97.16
9	20	5	20	1	167	8.5	0.5	0.16	93.65
10	30	5	20	0.5	167	8.5	2	0.48	96.19
11	20	15	20	0.5	71	8.5	2	1.46	97.16
12	20	5	20	0.5	71	6.5	0.5	0.47	93.41
13	25	10	40	0.8	119	7.5	1.2	0.49	97.61
14	25	10	40	0.8	119	7.5	1.2	0.49	97.19
15	25	10	40	0.8	119	7.5	1.2	0.48	96.70

Variables: (1) temperature, (2) methylene blue concentration, (3) adsorption time, (4) mass of activated carbon, (5) stirring speed, (6) pH of dye solution, (7) adsorbent particle size.

SEM micrographs (Fig. 6) of activated carbon reveal a fibrous and highly porous structure, with the presence of interconnected macropores and mesopores, resulting from the activation of the lignocellulosic precursor. The high surface roughness and the opening of longitudinal channels indicate a significant development of the porous structure, favoring the diffusion of the adsorbate. These results are consistent with the high iodine number obtained ($1162 \text{ mg I}_2 \cdot \text{g}^{-1}$), highlighting the predominance of micropores and the high adsorptive capacity of the material.

Methylene blue adsorption test

The results obtained for the adsorption capacity of activated carbon produced at 650°C from *Syzygium cumini* biomass ranged from 0.16 to 1.48 mg/g . Although the maximum adsorption capacity reached 1.48 mg g^{-1} , this value was obtained under experimental conditions in which a removal efficiency of 98.5% was achieved, indicating that the adsorbent was not saturated and still exhibited available active sites for further dye uptake. Therefore, the adsorption capacity reported here is strongly



Table 10 Regression coefficients for adsorption capacity (mg/g) of the methylene blue test

Name	Effect	Standard error	Calculated t	<i>p</i> -value
Mean	0.65	0.04	14.67	0.0000
Curvature	−0.33	0.20	−1.64	0.1516
Temperature (x_1)	−0.11	0.09	−1.22	0.2668
Methylene blue concentration (x_2)	0.67	0.09	7.51	0.0003
Adsorption time (x_3)	0.12	0.09	1.30	0.2415
Activated carbon mass (x_4)	−0.65	0.09	−7.36	0.0003
Stirring speed (x_5)	0.12	0.09	1.34	0.2296
pH of dye solution (x_6)	0.11	0.09	1.22	0.2668
Adsorbent particle size (x_7)	−0.11	0.09	−1.26	0.2538

dependent on the initial dye concentration and should not be directly compared with values obtained under saturation or high-concentration conditions. The experiments that resulted in the highest adsorption capacity were those related to the highest dye concentration and those that presented a solution pH higher than the pHPCZ (7.04). The highest adsorption capacities were in the experiments using the lowest amount of activated carbon. The adsorption efficiencies resulting from the experimental design of variable selection were all above 91.71%, as reported in Table 9.

Although high adsorption efficiencies (> 91.71%) were achieved, the reusability and regeneration of the activated carbon were not evaluated in this study. The primary objective of this work was the selection of significant variables affecting adsorption performance using a Plackett–Burman experimental design, serving as a proof-of-concept for the application of activated carbon derived from *Syzygium cumini* pruning. The reuse of carbon through cycles, however, has already been studied by other researchers. Albadarin and Al-Zawahreh (2025) conducted a review with 38 carbon-based adsorbents for phenol removal in aqueous media, including activated carbon, graphene, graphene oxide, multi-walled carbon nanotubes (MWCNTs), and carbonaceous composites. The most efficient materials in terms of reuse were activated carbons obtained from *Moringa oleifera* bark and lignin, as well as MWCNTs and graphene oxide, which maintained high efficiency for up to five cycles. Among the adsorbents evaluated, activated carbons derived from *Moringa oleifera* and lignin also stood out for their efficient removal of methylene blue dye.

It is important to note that the precursor material is an abundant and low-cost lignocellulosic residue, which may partially offset the production costs associated with

high-temperature activation. Nevertheless, regeneration and multi-cycle adsorption studies are essential to fully assess the economic and operational feasibility of the adsorbent and should be addressed in future investigations.

The factors studied (dye concentration and mass of activated carbon used in the test) presented a *p*-value equal to 0.0003, however the other factors presented *p*-values above the significance level of 0.05. Thus, only the concentration of methylene blue and mass of carbon presented statistically significant effects, as shown in Table 10. The effect of the dye concentration was positive and equal to 0.67, which indicates that the change from the lower level (−) to the higher level (+) in this planning favored the increase in the value of the adsorption capacity of the carbon. However, the carbon mass factor presented a negative effect (−0.65), indicating that the increase in the mass of carbon used in the adsorption test favored a lower adsorption capacity of the dye used.

Pareto Diagram (Fig. 7) of the adsorption capacity demonstrates the factors with significance in the experiment, which were the concentration of methylene blue and the mass of activated carbon, as well as the sequence of the factors according to their level following stirring speed, adsorption time, adsorbent granulometry, temperature, and pH of the methylene blue solution.

Table 11 presents the regression coefficients obtained for the factors studied in this research using the efficiency of methylene blue removal in the adsorption tests as the response of the experimental design. The concentration of the dye used was the only factor that presented statistical significance in the test, with a positive effect equal to 3.24 (*p*-value = 0.0062), indicating that the increase in the dye concentration favors the increase in the removal of this chemical species in the adsorption process.

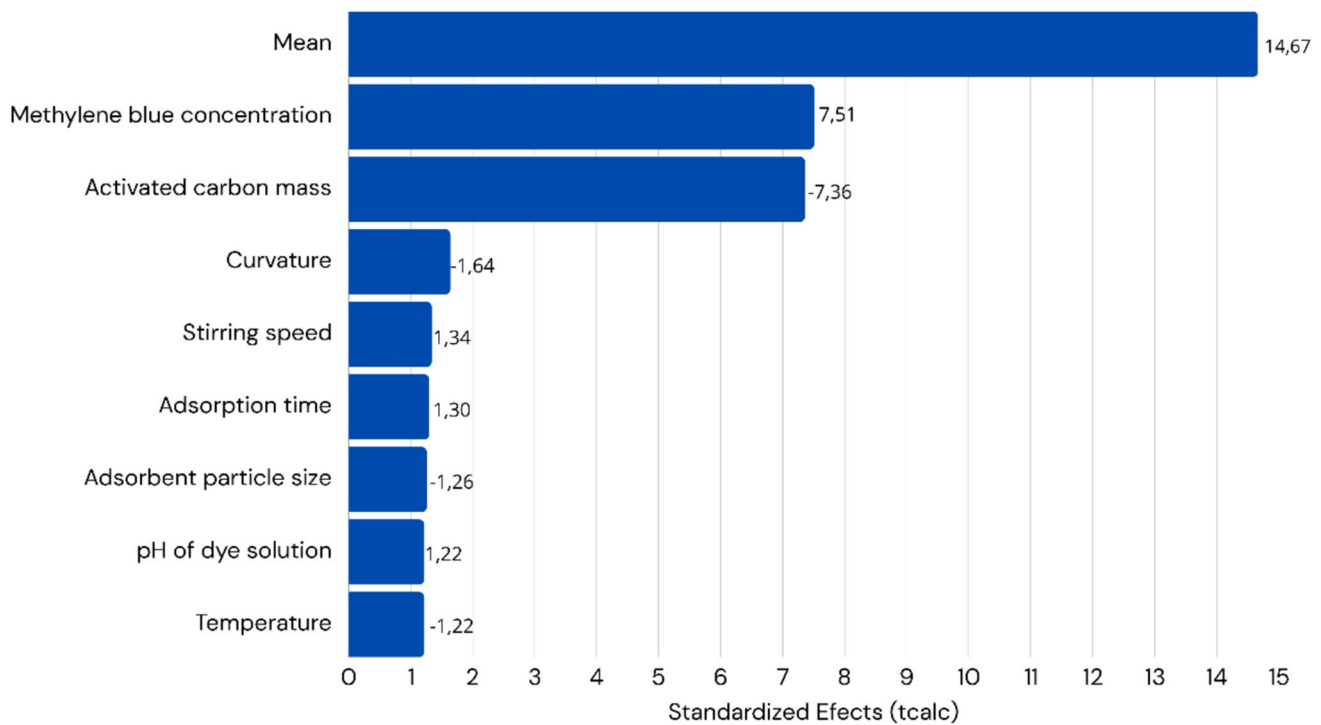


Fig. 7 Representation of the Pareto Diagram obtained in the PB adsorption capacity planning of the methylene blue test

Table 11 Regression coefficients for adsorption efficiency (%) of methylene blue test

Name	Effect	Standard error	Calculated t	p-value
Mean	96.30	0.39	245.34	0.0000
Curvature	1.74	1.76	0.99	0.3607
Temperature (x_1)	0.15	0.79	0.19	0.8579
Methylene blue concentration (x_2)	3.24	0.79	4.13	0.0062
Adsorption time (x_3)	0.50	0.79	0.63	0.5503
Activated carbon mass (x_4)	-0.63	0.79	-0.81	0.4506
Stirring speed (x_5)	1.16	0.79	1.47	0.1911
pH of dye solution (x_6)	-1.04	0.79	-1.32	0.2348
Adsorbent particle size (x_7)	1.14	0.79	1.46	0.1955

The increasing p -values followed the following sequence: (1) methylene blue concentration, (2) agitation speed, (3) adsorbent particle size, (4) pH of the blue solution, (5) carbon mass, (6) adsorption time and (7) temperature. The experimental design results for the dye adsorption test indicate that agitation speed, adsorption time, particle size, temperature, and solution pH are not statistically significant with respect to dye removal efficiency. Therefore, aiming at economy and process optimization, the test can be performed with agitation speed at low rotations in a shorter adsorption time (Popa and Visa 2017; Li et al. 2008).

Conclusion

The biomass studied is composed of high levels of volatile materials (81.9%), fixed carbon (17.44%) and carbon (45.04%), which indicates the possibility of using this material as a precursor in the production of carbonaceous products. Among the variables studied in the experimental planning related to biomass pyrolysis, only temperature had a significant negative effect (-58.74) on charcoal production at a significance level of 5%, which indicates that high



temperatures provide greater thermal degradation of the biomass. The charcoal obtained in the pyrolytic process presented the following characteristics: increased carbon and hydrogen ratio based on biomass, low ash content, alkaline pH, zero charge point around neutral, demonstrating a material with balanced electrical charges, iodine index above 1162.07 mgI₂/g, which indicates its application as an adsorbent material for micropollutants. The concentration of methylene blue and the mass of charcoal were significant in the variable selection planning. The concentration of methylene blue had a positive effect, demonstrating that with the increase in the concentration of the solution, the adsorption capacity of the activated charcoal will be greater. The mass of charcoal had a negative effect, showing that the lower this factor in this mass range, the greater the adsorption capacity in mg/g.

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Declarations

Conflict of interest All authors certify that they have no affiliations with or involvement in any organization or entity with any financial interest or non-financial interest in the subject matter or materials discussed in this manuscript.

Ethical approval Not applicable.

Consent for publication All authors agreed for publication.

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