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ORIGINAL ARTICLE

Cold alkaline extraction of Elephant grass for optimal subsequent extraction of hemicelluloses and energy production

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Abstract

There is growing scientific and industrial interest in obtaining useful substances by fractionating lignocellulosic biomass from non-food plant crops for use by the bioenergy industry. The primary goals are to ensure process sustainability and to comply with the principles of circular economy. In this work, we optimized energy production from Elephant grass by previously using cold alkaline extraction to remove its hemicellulose fraction. Elephant grass contains a high proportion of lignin (20%) and hemicelluloses (27.4%), and therefore is an excellent alternative to wood materials for energy production by direct burning. Energy production was optimized to identify the best operating conditions among those tested, namely: alkali concentrations of 80–120 g NaOH L^{-1}, temperatures of 20–40 °C, and treatment times of 30–90 min. Using the optimum conditions thus established (viz., 100 g NaOH L^{-1}, 30 °C, and 30 min) raised the high heating value (HHV) to 19.151 MJ kg^{-1} (i.e., by 4% relative to the starting material). Also, it allowed the content in elemental C to be preserved, that in H increased by 4.86% and, more environmentally significant, most sulphur (46.9%) to be removed from the solid phase upon treatment. Cold alkaline extraction of the raw material additionally enabled relatively selective separation of the hemicellulose fraction from the cellulose and lignin fractions. Thus, 30.1% of all hemicellulose was dissolved in the treatment liquor and made valorizable while 93.0% of cellulose and 82.1% of lignin present in the raw material remained in the solid phase.

Keywords Biorefinery · Cold alkaline extraction · Elephant grass · Hemicelluloses · Bioenergy

1 Introduction

Biorefining is becoming increasingly essential to ensure sustainable development, especially when using raw materials based on non-food plants such as perennial grasses, which escape the food versus fuel dilemma [1–3]. In fact, using biomass as a renewable energy source can be useful to fulfil the goals of the current European energy policy, which is increasingly favouring the establishment of sustainable energy crops not compromising food production [4, 5]. A fraction of 17.9% of all energy used in the word in 2018 was obtained from renewable sources—and 67% of it from biomass—2.2% from nuclear power and 79.9% from fossil fuels [6]. The European Union has set the goal that, by 2030, 37% of all energy should be produced from renewable sources [7, 8]. In the process, fossil fuels should be gradually replaced with renewable energy sources to reduce greenhouse gas emissions, diversify energy supply, and alleviate the currently strong dependence on oil markets while creating new jobs, fostering industrial entrepreneurship, and boosting economic growth [9, 10].

Energy from biomass can be used in the traditional manner (i.e., by burning, which uses 57.7% of lignocellulosic biomass) or as "modern bioenergy," which uses the remainder portion of biomass (specifically, 20.8% for industrial heating, 10% for home heating, 8.3% for transportation, and 3.3% for electricity production) [6].

A number of plant species are currently being used for energy production worldwide. Especially prominent among the non-wood species are perennial grasses such as giant reed (*Arundo donax*, L.), miscanthus (*Miscanthus x giganteus*), energy cane (*Saccharum spontaneum hybrids*), switchgrass (*Panicum virgatum L.*), bulbous canary grass

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(*Phalaris aquatica L.*), elephant grass (*Pennisetum purpureum* Schum.) [11–13], and bagasse from eating sugarcane [14, 15]. These plants are especially suitable for energy production by virtue of their low cropping costs, and also of their ability to grow in a variety of environments or even on marginal land [16]. Also, they produce large amounts of biomass [17], use water and nutrients highly efficiently [18, 19], contain more cellulose and lignin than deciduous species, and have a relatively low moisture content at the end of their growth cycle [2, 20, 21]. Even more important, they have the potential for use on marginal land and hence for improving the social and economic status of some rural areas [22].

Pennisetum purpureum Schum is a perennial grass of the family Poaceae from tropical Africa. Popularly known by the names "Elephant grass," "giant zacate," "fake sugarcane," "Napier grass," "merkeron," "Uganda grass" and "king grass," this plant has traditionally been used as fodder in tropical and subtropical countries. Each plant of Elephant grass has a number of branching stems radiating from the same stand. Its stems can be up to 8 m tall and 2.5 cm thick, and resemble those of sugarcane in appearance [1, 23]. Elephant grass grows well in temperate, subtropical, and tropical regions with a dry season. Also, it can grow in many types of land, but especially on thick, light-textured, well-drained soils [24], as well as on acid soils previously amended with biochar [25]. This species is highly resistant to disease and tolerant of drought [26]. Its ability to grow on contaminated soils has allowed its cropping in engineered wetlands to exploit its phytoremediating effects and use the resulting biomass for ethanol and butanol production [27]. Among others, elephant grass has additionally been used to reclaim water bodies contaminated with high concentrations of arsenic [21].

Like miscanthus and switchgrass, Elephant grass is a C4-type plant whose high photosynthetic efficiency leads to also high lignocellulose production [17, 28–30]. In fact, Elephant grass produces in the region of 45 t dry matter ha^{-1} $year^{-1}$ [1, 23, 31, 32] depending on the particular genotype, soil fertility, and crop management practices. Thus, [33] and [34] reported yields of 12–150 and 45–67 t ha^{-1} $year^{-1}$, respectively, so high biomass production yields, and the fact that Elephant grass can be harvested up to four times each year, make it especially attractive for energy production [1, 33].

Industrially, Elephant grass has been used to obtain chemicals such as cellulose and xylanase [35], monosaccharides and ethanol [36], xylitol and ethanol [37], hemicelluloses [38, 39], lignin nanoparticles [40], cellulose pulp [32, 41], and corrosion inhibitor extracts [42]. Also, it has been used to obtain fuels such as ethanol [33, 34, 43–45] or methane [24, 46–48], pyrolysis products [34, 49–52], thermal and electrical energy [17, 24, 53–58], and densified solid biofuels [19]. These uses testify to the high potential of Elephant grass for energy production and have fostered research into genetic breeding methods for improving its properties as a raw material and optimizing its use [24, 53, 59–61].

In this scenario, our group envisaged the development of a process for obtaining energy from Elephant grass and using its lignocellulose fraction as a source of chemicals. This is the way biorefining schemes are typically used to fractionate lignocellulosic materials stepwise for optimal use of the ensuing fractions. In this work, we used cold alkaline extraction (CAE) prior to fractionation of Elephant grass on the grounds of its operational and environmental advantages over other alkaline and hydrolysis treatments [26, 62]. The most salient advantage of CAE over alternative biomass fractionation methods is its ability to separate hemicelluloses in a selective manner for their subsequent alcoholic precipitation with minimal degradation to furfural [63–67]. In addition, CAE requires no high temperatures or pressures, and hence uses energy sparingly. In fact, most CAE-based processes are conducted at mild temperatures (20–40 °C) [68]. However, the efficiency of the CAE treatment is strongly dependent on the particular raw material and greater with grassy lignocellulosic materials than with woody plants (i.e., it allows lignocellulose to be more efficiently extracted from grasses) [63, 69–71].

With the aims of ensuring sustainability in the process and complying with the principles of circular economy, in this work, we optimized the efficiency of Elephant grass as an energy crop by subjecting the raw material to cold alkaline extraction in order to remove its hemicellulose fraction in the treatment liquor and use the resulting solid phase for energy production with increased economic and environmental efficiency in the overall process.

2 Materials and methods

Figure 1 shows the sequential scheme of the experimental work carried out with the Elephant grass.

2.1 Chemical and energy characterization

The material studied was elephant grass grown at an experimental station of the Pulp and Paper Laboratory of the Federal University of Viçosa (Minas Gerais, Brazil) that was ground by hand to obtain fragments of variable length and 1–4 cm in width. The material was characterized in chemical and energy terms by using the same methods as with the CAE liquor and solid phase.

For chemical analysis, samples were ground to variably sized particles in Wiley mill. The particles were sieved according to TAPPI T257-cm85 and those ranging from 40 to 60 mm in size retained. Moisture was determined by

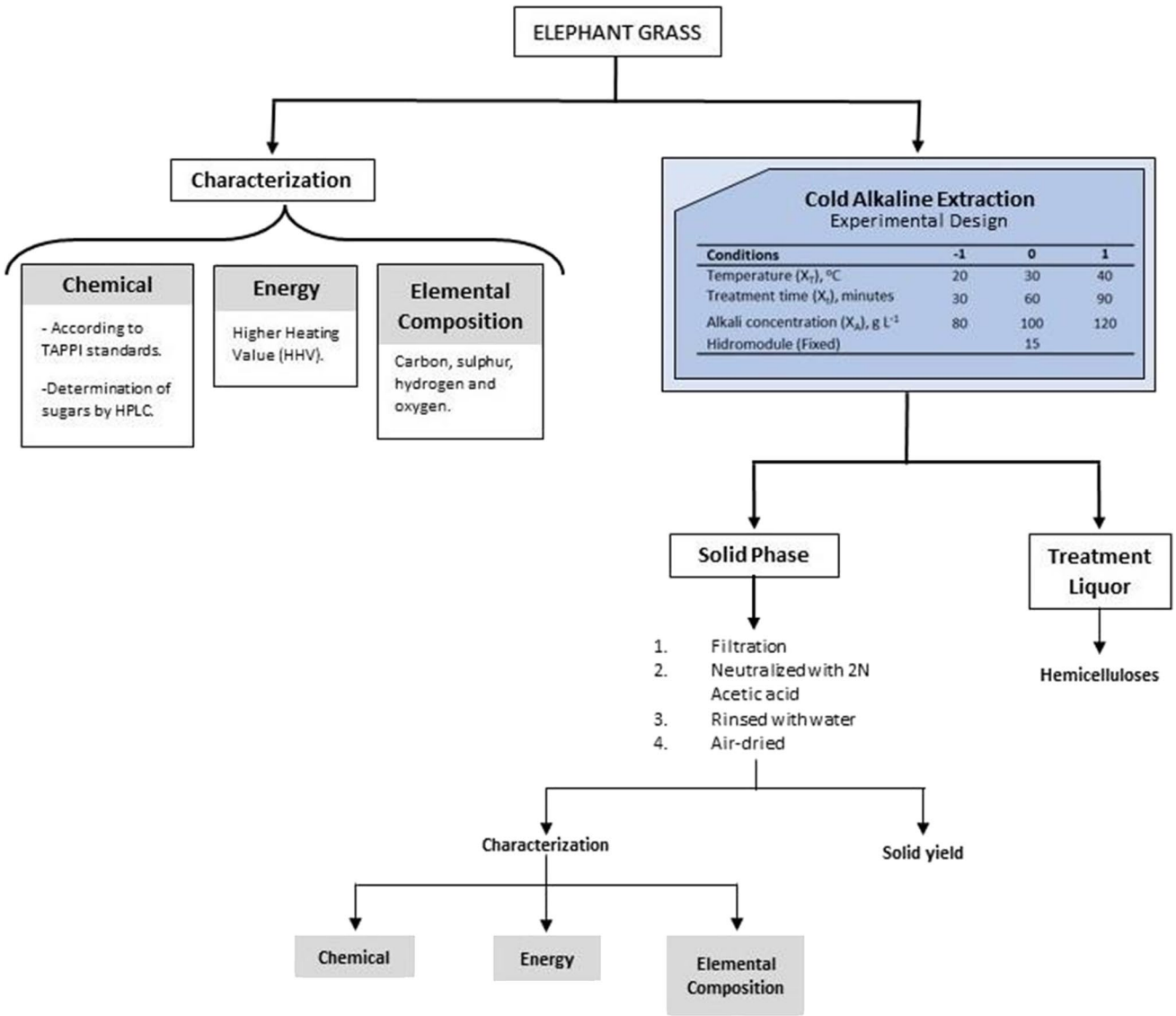


Fig. 1 Scheme of the work carried out

drying to a constant weight at 105 °C (TAPPI T-264-cm-07). The samples, raw material, and CAE solid phase were all hydrolysed with 5 mL of 72% H_2SO_4 acid at 30 °C for 1 h and the -hydrolysed by adding water to reduce the acid concentration down to 4% while heating at 121 °C for 60 min according to TAPPI T 249 cm-09. The resulting solid residue was recovered by filtration and labelled Klason lignin (TAPPI T 222 om-06). Soluble lignin was determined according to TAPPI T UM 25, and the hydrolysate used to determine, glucan, and hemicelluloses (xylan, araban, galactan, and mannan) in the material on an Agilent 1100 HPLC instrument equipped with a BioRaden ion-exchange column and a refractive index detector. The working temperature was 50 °C, and the mobile phase consisted of 0.005 M H_2SO_4 and was circulated at 0–0.6 mL/min. All measurements were made in triplicate and the resulting coefficient of variation was less than 4%.

The raw materials and the solid phases were analysed for carbon, sulphur, hydrogen, and oxygen on an Eltra Helios CHS analyser. Also, they were used to determine their higher heating value (HHV) at a constant volume in accordance with CEN/TS 14,918 (2005) and UNE 16,001 EX (2005) on a Parr 6300 automatic isoperibol bomb calorimeter.

2.2 Cold alkaline extraction and determination of hemicelluloses

Alkaline extraction was done by immersing the raw material in a bath containing an NaOH solution, using a constant solid/liquid ratio on a dry basis of 15 in all tests. The

independent variables of the process were the soda concentration, temperature, and treatment time.

The solid fraction was recovered by filtration, neutralized with 2 N acetic acid, rinsed with water, and air-dried for weighing in order to estimate yield. The proportions of glucan and hemicelluloses from the raw material present in the extraction liquor were determined as described in the previous Sect. (2.1). The solid fraction was also subjected to the determinations described in it.

2.3 Multiple regression models and experimental design of cold alkaline extraction

The CAE process was modelled in order to maximize extraction of hemicellulose derivatives while ensuring that the resulting solid would have a high heating value for energy production. A $2n$ factorial design with a central point was used to reduce the number of tests needed and also to avoid significant covariances between the dependent variables. The number of tests needed, N, was calculated as $2^n + 2n + n_C$, where 2^n is the number of points constituting the design, $2n$ that of axial points and n_C that of central points.

The independent variables were normalized by using the following Eq. (1):

$$X_n = \frac{X - \mathrm{X}}{(X_{\max} - X_{\min})/2} \tag{1}$$

where X is the absolute value of the variable concerned, and $\bar{X}$, $X_{\max}$, and $X_{\min}$ are its mean, maximum, and minimum value, respectively.

As can be seen in Table 1, each independent variable was examined at three different values. The results were used to establish second-order polynomials for each dependent variable, the polynomials including linear and quadratic terms of the independent variables, and their mutual interactions, and being fitted by multiple regression:

$$Y = a_0 + \sum_{i=1}^{n} b_i X_{ni} + \sum_{i=1}^{n} c_i X_{ni}^2 + \sum_{i=1 i=1}^{n} d_{ij} X_{ni} X_{nj} (i < j) \tag{2}$$

where X denotes independent variables, Y dependent variables, and coefficients a_o, b_i, c_i, and d_{ij} are constants for each model obtained from the experimental results. The dependent variables were yield, glucan, xylan, araban, galactan, mannan, Klason lignin, and soluble lignin, and the independent variables were soda concentration, temperature, and time.

Table 1 Conditions of CAE experimental design

Conditions	−1	0	1
Temperature (X_T), °C	20	30	40
Treatment time (X_t), minutes	30	60	90
Alkali concentration (X_A), g L^{-1}	80	100	120
Hidromodule (fixed)	15		

The independent variables present in the polynomials are those which exhibited statistically significant coefficients. In fact, no term with $p < 0.05$ as per Student's t-test or spanning a confidence interval narrower than 95% was included.

The results were processed with the software Statistica v. 10.0 (StatSoft, Inc., Tulsa, OK, USA) and additionally used to calculate R^2 and Snedecor's F-value, those with $R^2 > 0.85$ or $F > 5$ being deemed acceptable.

3 Results and discussion

As noted earlier, the primary aims of this work were to optimize the use of Elephant grass by subjecting the raw material to cold alkaline extraction (CAE) in order to remove its hemicellulose fraction in the treatment liquor and facilitate energy production from the resulting solid phase under the assumption that CAE would increase the heat capacity and decrease the sulphur content of the solidphase while enabling selective extraction of hemicellulose derivatives for their valorization. For this purpose, the starting material was characterized in chemical and energy terms, and the CAE process was modelled for optimal energy production. Finally, the efficiency with which hemicellulose derivatives were extracted, and its dependence on the operating conditions, were assessed.

3.1 Chemical and energy characterization

Based on recent studies [24], Elephant grass can store dry matter more efficiently than other plant species. This results in favourable quality-related biomass properties including high heating value (HHV), and cellulose, hemicellulose, lignin, and ash contents. These properties make elephant grass an effective alternative to lignocellulosic raw materials for energy production by burning. Table 2 shows the chemical composition of Elephant grass as determined by our group and other authors. As can be seen, coefficients of variation were relatively high (viz. 136% for cellulose, 105% hemicellulose, 69% for lignin, and 25% for ash), which can be ascribed to differences in plant growth environment. In any case, our results fall within the chemical composition ranges previously reported by other authors. Also, the differences in composition can have arisen from differences among the 85 genotypes studied [33], which spanned cellulose and lignin contents over the range 38.5–48.9% and 6.4–12.4%, respectively.

The chemical composition of Elephant grass is compared with those of other grass species and a reference material (*Eucalyptus globulus*) used for bioenergy production and

Table 2 Chemical characterization of Elephant grass

Component (%)	This work	Oluwasina [38]	Gomes [32]	Pandey [21]	Cardona [72]	De Conto [49]
Glucan	41.7	55.33	38.2	36.34	22.6	30.37
Xylan	23.1	–	9.6	–	–	–
Arabinan	2.9	–	0.2	–	–	–
Galactan	0.95	–	0.8	–	–	–
Manan	0.45	–	0.6	–	–	–
Hemicelluloses	27.4	16.6	–	34.12	20.9	31.31
Klason lignin	19.3	2.79	–	–	–	–
Soluble lignin	1.56	24.0	2.2	–	–	–
Lignin	20.9		18	30.40	19.4	26.02
Holocelluloses	69.1	69.60	–	–	–	–
Ash	–	8.87	–	–	11.1	–

other industrial purposes in Table 3. The cellulose content of the plant material used here (41.7%) was higher than that of other grasses such as switchgrass (31.0%) and sugarcane bagasse (39.0%), but somewhat lower than that of *Miscanthus giganteus* (48.6%) and eucalyptus wood (48.1%). The material had a high content in hemicellulose (27.4%), which is especially useful for biorefining by selective extraction of the hemicellulose fraction. This content exceeds the combined xylan and araban content reported by Oluwasina [38] for the same species (17.6%) and is comparable to the upper ends of the ranges for other materials such as rice straw (23–28%), wheat straw (26–32%), sugarcane bagasse (27–32%), and bamboo (15–26%) [80]. Xylan was the major component of the hemicellulose fraction and present in considerably higher proportions (23.1%) than in other materials (e.g., 8.4% in eucalyptus wood or 17.3% in sugarcane bagasse) [81]. The proportion of lignin in Elephant grass (20.9%) was higher than those in other residual grass materials such as wheat straw, tricolor sorghum, or corn stover (18.6%) [73], but slightly lower than those of grass species such as *M. Giganteus* and sugarcane bagasse, and wood species such as eucalyptus (26.9%), poplar (25.2%), and leucaena (24%) [75, 77, 82, 83]. Finally, the lignin contents of elephant straw were similar to those of other raw materials such as rice straw (15–25%) [84, 85] and fell in the overall ranges for grass species (10–30%) [73].

In terms of energy potential, the high heating value (HHV) of Elephant grass, calculated as described under Materials and Methods, was 18.44 MJ kg^{-1}, which is very similar to that reported by Reza et al., [50] (18.55 MJ kg^{-1}) but higher than that obtained by Tsai [86] (16.3 MJ kg^{-1}). These differences are quite normal because HHV changes with plant age (e.g., from 16.96 MJ kg^{-1} at 4 months to 22.04 MJ kg^{-1} at 6) [87]. HHV for Elephant grass was similar to the values for a number of wood species (in the region of 20 MJ kg^{-1}) according to [88], and also similar to or slightly greater than those of short-lifecycle plant species used for energy production such as *Leucaena leucocephala* (19.3–20.6 MJ kg^{-1}) [83], switchgrass (18.8 MJ kg^{-1}), or wheat straw (18.55 MJ kg^{-1}) [74].

An elemental composition analysis conducted as described under Materials and Methods provided the C, O,

Table 3 Chemical composition of selected lignocellulosic crops of industrial and bioenergetic interest

Component (%)	Switchgrass [73, 74]	*Miscanthus* x *giganteus* [75, 76]	Sugarcane bagasse [32]	Eucalyptus [73, 77, 78]	Wheat straw [73, 79]	Bicolor sorghum [73]
Glucan	30.97, 38	48.64, 39.5	39.01	48.07, 49.4, 46.8	32.64, 41.8	34.01
Xylan	20.42, 22.8	nd, 19.0	22.05	10.42, 12, 16.6	19.22, 25.4	14.14
Arabinan	2.75, 3.7	nd, 1.8	2.06	0.3, 0.3, 0.5	2.35, 2.5	1.65
Galactan	0.92, 1.3		0.46	0.74, 1.2	0.75, 0.7	0.52
Mannan	0.29		0.35	1.23, 0.9	0.31	0.2
Hemicelluloses	24.55	18.67				
Lignin	17.56, 22.1	27.2	23.09	26.91, 27.2, 22.9	16.85	16.09
Holocellulose		67.31		nd, nd, 66.9		
Ash	5.76, 3.7	5.49	3.66	1.22	10.22	5.04
Extractives	16.99		3.76	4.15	12.95	22.03

H, and S contents that are compared with those reported by Reza et al. [50] and those for various other raw materials in Table 4. The chemical composition of a raw material can be expected to influence that of the gases it releases and the amount of heat it produces upon burning. The fact that the C and H contents of Elephant grass were somewhat higher than those of other materials suggested that it might be more efficient for energy production. On the other hand, the S content of Elephant grass was similar to those of other lignocellulosic raw materials and substantially lower than those of fossil fuels.

Table 4 Elemental composition of Elephant grass and other lignocellulosic materials

Species	C (%)	H (%)	S (%)	O (%)
Elephant grass[(1)]	48.999	6.790	0.12125	44.09
Elephant grass[(2)]	43.23	5.8	0.11	50.86
Clon AF2 Populus[(1)]	52.101	6.023	0.016	41.86
Leucaena[(1)]	50.623	6.117	0.024	43.236
Robinia[(1)]	51.38	6.301	0.023	42.296
Eucalyptus globulus[(1)]	51.924	6.326	0.01	41.74
Olmo[(1)]	52.916	6.406	0.017	40.661
Pino[(1)]	57.746	6.942	0.12	35.192
Ethiopian rapeseed[(3)]	46.3	6.1	0.49	47.11
Populus[(3)]	48.19	5.9	0.05	45.86

[(1)]This work, [(2)] [50], [(3)] [89]

3.2 Modelling and optimization of the higher heating value and elemental composition of the post-CAE liquor and solid phase

Table 5 shows the HHV and elemental composition (C, H, S, and O) of the solid fraction from the CAE treatment as determined under variable operating conditions selected in accordance with the experimental design used. The polynomial equations of the design are shown in Table 6, which includes one for CAE solid yield. As can be seen, the differences between observed and calculated values never exceeded 10% and correlation coefficients were all higher than 0.95. Table 5 also includes CAE yield.

As can be seen from Eq. 1 in Table 6, HHV for the post-CAE solid phase from Elephant grass was strongly dependent on the quadratic terms of the process variables. Clearly, the values and signs of the coefficients for the treatment time suggest that times in the lower end of the operating range should result in increased HHV levels. Also clearly, using alkali concentrations and temperatures at the ends of the operating ranges would detract from HHV as the very likely result of poor extraction of hemicelluloses into the CAE liquor at low temperatures and excessive extraction of polyphenols (lignin) at high temperatures. As confirmed by Fig. 2A, it was therefore advisable to use medium alkali concentrations and temperatures. Thus, Eq. 1 in Table 6 predicts HHV = 19.151 MJ kg^{-1}, which was nearly 4% higher than the value for the starting material.

Table 5 HHV and elemental composition of the post-CAE solid phase obtained under different operating conditions

T (°C), t (min), NaOH concentration (g L^{-1})	C (%)	H (%)	S (%)	O (%)	HHV (MJ/kg)	Yield (%)
0, 0, 0	47.581	7.089	0.0865	52.333	18.745	74.6
0, 0, 0	47.656	7.061	0.0827	52.261	18,863	75.3
1, 1, 1	47.185	6.747	0.0019	52.813	18.200	66.9
1, 1, –1	46.170	6.724	0.0018	53.828	17.777	71.8
1, –1, 1	47.027	6.862	0.0040	52.969	18.449	79.4
1, –1, –1	46.866	6.931	0.0035	53.131	18.045	82.8
–1, 1, 1	47.052	6.755	0.0084	52.940	18.122	75.3
–1, 1, –1	46.956	6.703	0.0118	53.032	18.278	72.1
–1, –1, 1	46.956	6.810	0.0384	53.006	18.429	84.7
–1, –1, –1	47.585	6.850	0.0384	52.377	18.374	86.8
1, 0, 0	46.172	6.975	0.0232	53.805	18.435	70.5
–1, 0, 0	46.263	6.869	0.0520	53.685	18.402	76.1
0, 1, 0	48.388	7.056	0.0613	51.551	18.866	70.6
0, –1, 0	48.871	7.067	0.0891	51.040	19.244	82.5
0, 0, 1	46.891	6.982	0.0831	53.026	18,590	77.4
0, 0, –1	47.077	6.975	0.0844	52.839	18.470	80.6
Raw material	48.999	6.790	0.12125	57.912	18.440	

Y_{HHV} high heating value (HHV). Y_C, Y_H, and Y_S content in carbon, hydrogen, and sulphur (%). Y_{yield} CAE yield (%). X_A, X_T, X_t normalized alkali concentration, temperature, and treatment time, respectively

Table 6 Polynomial models for HHV, C, H, S, and yield

Eq	Polynomial	r^2	F
1	$Y_{HHV} = 18.850 + 0.0938 \cdot X_A - 0.121 \cdot X_t - 0.070 \cdot X_T - 0.346 \cdot X_A^2 + 0.179 \cdot X_t^2 - 0.458 \cdot X_T^2 + 0.116 \cdot X_A \cdot X_T$	0.940	34.5
2	$Y_C = 47.513 - 0.279 \cdot X_t - 0.302 \cdot X_T - 0.476 \cdot X_A^2 + 1.170 \cdot X_t^2 - 1.242 \cdot X_T^2$	0.945	52.6
3	$Y_S = 0.0887 - 0.0097 \cdot X_t - 0.0112 \cdot X_T - 0.0070 \cdot X_A^2 - 0.0156 \cdot X_t^2 - 0.0532 \cdot X_T^2 + 0.0070 \cdot X_T \cdot X_t$	0.982	140.4
4	$Y_H = 7.073 - 0.047\ X_t + 0.035 \cdot X_T - 0.104 \cdot X_A^2 - 0.160 \cdot X_T^2 + 0.0316 \cdot X_A \cdot X_t$	0.956	66.9
5	$Y_{yield} = 75.56 - 2.35\ X_T - 5.96\ X_t - 1.04\ X_A - 1.94\ X_T^2 + 3.78\ X_A^2 - 1.19\ X_t X_A$	0.987	57.61

Y_{HHV} high heating value (HHV). Y_C, Y_H, and Y_S content in carbon, hydrogen, and sulphur (%). Y_{yield} CAE yield (%). X_A, X_T, X_t normalized alkali concentration, temperature, and treatment time, respectively

Equations 2 and 4 in Table 6 show the modelled results for the C and H contents of the post-CAE solid phase. The conditions needed to maximize HHV and both contents were identical. Again, the quadratic terms were especially influential and interaction terms virtually uninfluential. As with HHV, maximizing the C and H contents of the post-CAE solid phase would require using medium alkali concentrations and temperatures in combination with short treatment times. Under these conditions, Eqs. 2 and 4 in Table 6 predict a content in C of 48.961% and one in H of 7.120%. While the C content was similar to that of the raw material, the H content was 4.86% higher. The response surface for the C content, not shown, was similar to that of Fig. 2A.

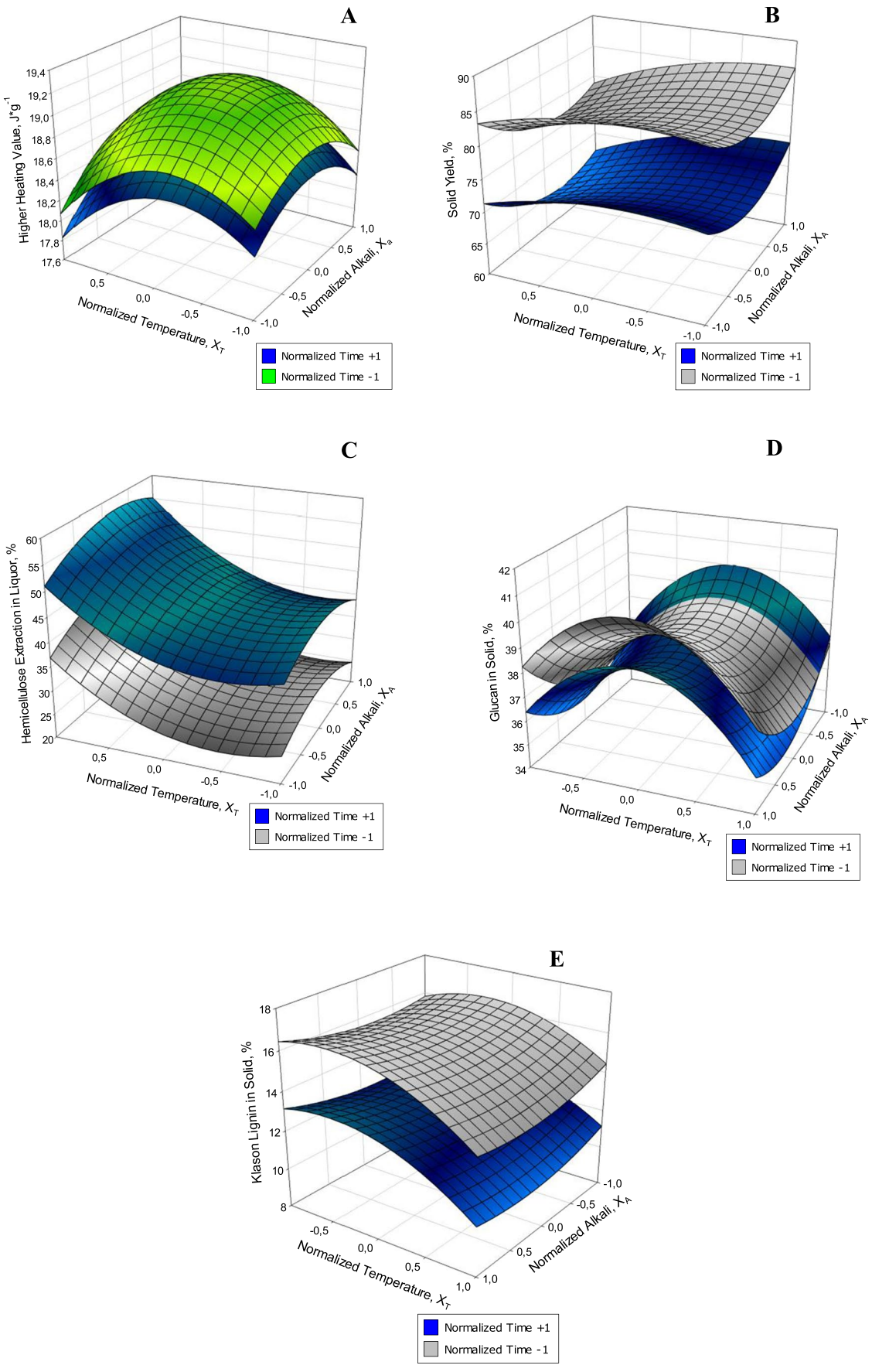
Based on the predictions of Eq. 3 in Table 6, the operating conditions maximizing energy production, and the C and H contents, should lead to increased S contents in the post-CAE solid phase. Although this might be an unwanted outcome of the treatment, the S content of the starting biomass was so low that it was unlikely to raise environmental concerns. Also, the CAE treatment considerably reduced the S content by 26.6–98.5% depending on the particular conditions (46.9% with the optimum settings). In this way, CAE facilitated use of the material for energy production with little emission of sulphur dioxide. This is an added advantage to the above-mentioned increased energy yield and valorization of the hemicellulose fraction relative to burning of the raw material. Also, the increased energy yield need not be accompanied by an excessive decrease in solid yield. In fact, hemicellulose extraction should be selective providing most cellulose and lignin remain in the post-CAE solid phase.

As can be seen from Table 5, the CAE treatment resulted in little biomass loss. Thus, solid yields ranged from 66.9 to 86.8% and exceeded those obtained by García et al. [63] treating wheat straw (viz., 61.1% by using an NaOH concentration of 100 g L^{-1}, a temperature of 40 °C, and a treatment time of 90 min). The yield from Elephant grass under similar temperature and time conditions amounted to 71.8 and 66.9% with an NaOH concentration of 80 and 120 g L^{-1}, respectively. There were no substantial changes in yield with temperature or the alkali concentration, however. By contrast, time considerably influenced the results. Thus, the highest yields were obtained at short times (30 min, normalized value –1) and, with little difference, low temperatures (20 °C, –1) and high alkali concentrations (120 g NaOH L^{-1}, + 1). As can be seen from Fig. 2B, using short treatment times and medium temperatures (normalized value 0) provided the highest solid yields. Based on Eq. 5 in Table 6, using medium temperatures and alkali concentrations in combination with short times (–1) would lead to a yield of 81.5% in the post-CAE solid phase. This is quite acceptable if one considers that the highest possible yield was 85.6% (Eq. 5, Table 6).

3.3 Modelling and optimization of monosaccharide contents in the post-CAE liquor and solid phase

Table 7 shows the contents in Klason and soluble lignin, individual monomers from the polysaccharide fraction, their combination, and monomers from the hemicellulose fraction (xylan, arabinan, galactan, and mannan) of the post-CAE solid fraction as a function of the operating conditions used. The results were used to calculate the proportion of each fraction that was extracted into the CAE liquor. Table 8 shows the proportions such fractions but relative to the percent content of each polymer in the starting material.

Not all chemical characterization results shown in Tables 7 and 8 were modelled. In fact, some components were present in too small amounts in both the raw material and the post-CAE solid phase for any useful conclusions to be drawn. Rather, we used multiple regression on the major dependent variables suggested by the experimental design in addition to total hemicellulose in order to consider two minor saccharides (mannan and galactan). On the other hand, the liquid fraction was modelled for extracted lignin, extracted glucan, and total extracted hemicellulose (viz., the combination of all monomers listed in Table 8 except glucan). The models used, and their statistical figures of merit, are shown in Table 9. As can be seen, the differences between experimental and calculated values never exceeded 10% and correlation coefficients (R^2) were all higher than 0.95.


A
Higher Heating Value, J*g-1
19,4
19,2
19,0
18,8
18,6
18,4
18,2
18,0
17,8
17,6
0,5
0,0
-0,5
-1,0
Normalized Temperature, XT
1,0
0,5
0,0
-0,5
-1,0
Normalized Alkali, XA
Normalized Time +1
Normalized Time -1
B
Solid Yield, %
90
85
80
75
70
65
60
0,5
0,0
-0,5
-1,0
Normalized Temperature, XT
1,0
0,5
0,0
-0,5
-1,0
Normalized Alkali, XA
Normalized Time +1
Normalized Time -1
C
Hemicellulose Extraction in Liquor, %
60
55
50
45
40
35
30
25
20
0,5
0,0
-0,5
-1,0
Normalized Temperature, XT
1,0
0,5
0,0
-0,5
-1,0
Normalized Alkali, XA
Normalized Time +1
Normalized Time -1
D
Glucan in Solid, %
42
41
40
39
38
37
36
35
34
-0,5
0,0
0,5
1,0
Normalized Temperature, XT
-1,0
-0,5
0,0
0,5
1,0
Normalized Alkali, XA
Normalized Time +1
Normalized Time -1
E
Klason Lignin in Solid, %
18
16
14
12
10
8
-0,5
0,0
0,5
1,0
Normalized Temperature, XT
-1,0
-0,5
0,0
0,5
1,0
Normalized Alkali, XA
Normalized Time +1
Normalized Time -1

◂**Fig. 2** **A** HHV response surface. **B** CAE yield response surface. **C** Hemicellulose extraction in the CAE liquor. **D** Percent cellulose content, as glucan, in the post-CAE solid phase. **E** Percent Klason lignin in the post-CAE solid phase

The values of the dependent variables of Table 9 were used to construct response surfaces in order to facilitate comparisons and identification of the optimum CAE conditions. The *z*-axis represents each of the most influential variables at two different response levels (+1 and –1).

CAE allowed hemicelluloses to be relatively selectively extracted in the form of highly pure polymers [90]. In fact, only 6.6–20.1% of all glucan present in the raw material—which accounted for an average 11.3% in it—was removed (i.e., 2.8–84% of all starting material). By contrast, 25.4–56.0% of the whole hemicellulose fraction (viz., 7.0–15.3% of raw material) was extracted. CAE additionally resulted in substantial delignification. Thus, 16.3–46.4% of the initial amount of Klason lignin (3.2–9.0% of all raw material) was extracted into the CAE liquor.

The post-CAE liquor contained various monomers, xylo-oligomers, minerals, proteins, degradation products (furfural and 5-hydroxymethylfurfural), gluco-oligomers, arabanoligomers, extractables, dissolved lignin, and non-volatile solids [83]. Overall, hemicelluloses were the most efficiently extracted lignocellulosic fraction (especially xylan). Galactan and mannan monomers supplied only small amounts of monosaccharides to the post-CAE liquor owing to their low proportion in the raw material (less than 1% and 0.5%, respectively). Also, araban was less markedly extracted into the CAE liquor than was xylan. As expected, extraction peaked under the most drastic operating conditions.

Figure 2C illustrates the dependence of hemicellulose extraction into the CAE liquor on the operating conditions. As can be seen, the highest proportion of hemicellulose in the liquor (57.1%) was obtained by using a medium alkali concentration (100 g NaOH L^{-1}, normalized value = 0), a high temperature (40 °C, + 1), and a long treatment time (90 min, + 1). In any case, the first variable was scarcely influential. Other authors previously reported similar hemicellulose extraction values. Thus, Sun et al. [91] dissolved 89.3% of all hemicelluloses in poplar wood by using a similar alkali concentration (8.5% vs 10% here) but a much longer treatment time (16 h). Cheng et al. [65] obtained high xylan extraction yields (> 90%) from corn stover by using an NaOH concentration of 10%, a temperature of 75 °C and a time of 2 h. Under optimum conditions (viz., an alkaline mass fraction of 9.5%, 78 °C, and 252 min), Shao [92] obtained a hemicellulose extraction yield of 52.8% from poplar wood

In this work, we chose to restrict hemicellulose extraction in order to avoid cellulose depolymerization and extensive delignification—which would have diminished the high heating value (HHV) of the post-CAE solid phase. In fact, biomass for thermal energy production by burning should contain high proportions of lignin and cellulose, and possess a high C/N ratio, and high heating value but low moisture, ash, and nitrogen contents [57]. Thus, although the glucan fraction supplied monosaccharides to the CAE liquor, cellulose should ideally remain in the solid phase as a polymer in order to preserve its heating value—and so should lignin, which possesses a high thermal potential. Specifically, cellulose, hemicellulose, and lignin have an HHV of 17.36, 17.54, and 25.8 MJ kg^{-1}, respectively (i.e., cellulose and hemicellulose have a similar HHV which, however, is 48% lower than that of lignin) [93]. Using the operating conditions needed to maximize HHV in the post-CAE solid phase resulted in a hemicellulose extraction yield of 30.1%, which was lower than that obtained under the most drastic conditions (55.1%) but allowed relatively selective extraction of hemicelluloses over glucan and lignin.

As can be seen from Fig. 2D, minimizing the proportion of cellulose dissolved in the post-CAE liquor would require using high alkali concentrations, medium temperatures, and short treatment times.

It is necessary to take into account the synergy of the operating variables on the extraction of glucan. In our case, time and temperature are much more influential than the alkali concentration (see Eq. 2 in Table 9 or Fig. 2D). As the NaOH concentration is scarcely influential, so it allowed medium values to be used instead of high levels (i.e., the most suitable conditions for maximizing energy production). The percentages of glucan in solid can also rise if the extraction process is more selective towards the other fractions. As predicted by Eq. 2 in Table 9, such conditions would lead to a proportion of 38.7% of cellulose (glucan) in the post-CAE solid phase (i.e., 93% of all cellulose present in the starting material would remain in that phase). Also, as predicted by Eq. 7 in Table 9, only 7.15% of the initial amount of glucan would be dissolved in the CAE liquor (compared to 30.1% of all hemicellulose). These numbers testify to the ability of CAE to selectively extract hemicelluloses over cellulose.

Restricting delignification in order to allow as much lignin as possible to remain in the solid phase (Fig. 2E) would require using low temperatures and short treatment times with any alkali concentration. As can be seen, however, normalized temperatures over the range –1 to 0 scarcely influenced the lignin content of the post-CAE solid phase. As predicted by Eq. 6 in Table 9, the conditions needed to maximize HHV would result in 18.0% of all lignin being extracted into the CAE liquor.

Based on the foregoing, increasing the heating value of the post-CAE solid phase while ensuring adequate, selective extraction of hemicelluloses would require using the following, optimum conditions: a medium alkali concentration

Table7 Composition of the solid resulting from the CAE in percentage with respect to the raw material

T(°C), *t*(min), Conc. NaOH (g/L)	Lignin, %		Carbohydrates, %						Total hemicellulose
	Klason	Soluble	Glucan	Xylan	Arabinan	Galactan	Mannan	Total CHO	
0 0 0	13.6	0.8	36,3	13,3	2,2	0,7	0,2	52,8	16,4
0 0 0	13,4	0.8	36.7	13.5	2.2	0.7	0.2	53.2	16.6
1 1 1	10.4	0.6	36.0	9.3	1.8	0.6	0.3	48.1	12.1
1 1 – 1	11.3	0.6	36.8	10.7	1.9	0.7	0.3	50.4	13.5
1 – 1 1	14.0	0.9	37.0	12.8	2.5	0.9	0.4	53.7	16.6
1 – 1 – 1	14.3	1.0	37.2	13.3	2.6	0.9	0.4	54.4	17.2
– 1 1 1	13.5	0.8	36.3	13.4	2.4	0.8	0.3	53.1	16.8
– 1 1 – 1	12.4	1.0	36.9	13.0	2.5	0.8	0.3	53.5	16.6
– 1 – 1 1	16.1	1.4	38.9	16.4	2.8	0.9	0.3	59.3	20.4
– 1 – 1 – 1	15.9	1.5	35.8	16.0	2.7	0.9	0.4	55.8	20.0
1 0 0	11.2	0.9	33.3	9.7	2.0	0.7	0.3	46.0	12.7
– 1 0 0	13.3	1.2	33.9	13.6	2.3	0.8	0.3	50.9	17.0
0 1 0	12.2	0.5	37.9	12.3	2.1	0.7	0.1	53.1	15.2
0 – 1 0	16.2	0.8	38.8	15.7	2.5	0.9	0.2	58.0	19.2
0 0 1	13.8	0.8	37.7	13.7	2.5	0.7	0.3	54.8	17.1
0 0 – 1	14.2	0.8	38.5	14.6	2.3	0.8	0.3	56.5	18.0

Table 8 Percentages of extraction in liquid phase relative to the percent content of each polymer in the starting material

T(°C), *t*(min), Conc. NaOH (g/L)	Extraction in percentage in liquid phase with respect to the initial polymer (%)							% Total hemicellulos extracted in liquid
	Klason lignin	Soluble lignin	Glucan	Xylan	Arabinan	Galactan	Mannan	
0 0 0	29.5	49.5	12.8	42.3	25.4	24.4	53.9	40.0
0 0 0	30.8	50.4	11.9	41.8	24.8	24.9	51.1	39.5
1 1 1	46.4	64.1	13.5	59.6	36.6	33.1	40.6	55.9
1 1 – 1	41.2	62.2	11.6	53.8	34.4	28.9	36.2	50.6
1 – 1 1	27.7	44.9	11.1	44.7	12.4	3.9	11.8	39.3
1 – 1 – 1	26.0	33.9	10.8	42.3	10.0	4.1	17.2	37.1
– 1 1 1	30.3	50.7	12.9	42.0	18.2	20.7	33.1	38.6
– 1 1 – 1	35.5	35.6	11.5	43.9	13.0	12.8	36.0	39.4
– 1 – 1 1	16.8	10.4	6.6	29.2	3.1	1.9	24.7	25.4
– 1 – 1 – 1	17.7	0.8	14.1	30.7	5.7	4.0	22.8	27.0
1 0 0	42.1	40.2	20.1	58.0	30.7	25.8	36.4	53.7
– 1 0 0	31.2	21.5	18.7	41.1	20.0	19.9	32.4	38.0
0 1 0	36.7	71.2	9.1	46.7	29.2	22.0	76.5	44.4
0 – 1 0	16.3	49.1	7.0	32.0	14.7	4.6	63.4	29.8
0 0 1	28.4	50.0	9.6	40.9	14.6	26.8	31.2	37.4
0 0 – 1	26.3	51.5	7.7	36.8	19.4	19.4	28.4	34.3

(100 g NaOH L^{-1}, normalized value = 0), a medium temperature (30 °C, 0), and a short treatment time (30 min, –1). These conditions are milder than those used by García et al., (2013) [63] to obtain a hemicellulose-rich valorizable liquor and a solid fraction suitable for producing cellulose pulp (viz., 100 g NaOH L^{-1}, 40 °C, and 90 min, which allowed them to extract all hemicellulose present in the raw material and 10.3% of lignin). As predicted by Eq. 8 in Table 9, these conditions would allow 55.1% of all hemicellulose and 46.8% of all lignin present in elephant grass to be extracted. Therefore, as suggested by the results of García et al. [63], (2017), the alkaline treatment was more efficient with elephant grass than it was with wheat straw. Also, based on the results of De Carvalho et al. [94], it was comparably efficient for extraction from sugarcane straw. Thus, using 110 g NaOH L^{-1}, 33 °C, and 60 min in combination allowed them

Table 9 Models for the dependent variables after the CAE process

No. Eq	Solid residue equations	r^2	*F*-Snedecor
1	$YligK = 13.48 - 1.00\ X_T - 1.65\ X_t - 1.25\ X_T^2 + 0.70\ X_t^2 + 0.54\ X_A^2 - 0.32\ X_T X_A$	0.984	92.69
2	$Yglu = 36.52 - 0.38\ X_t - 2.96\ X_T^2 + 1.78\ X_t^2 + 1.52\ X_A^2 - 0.44\ X_T X_A - 0.55\ X_t X_A$	0.942	24.41
3	$Yxil = 13.36 - 1.65\ X_T - 1.55\ X_t - 0.21\ X_A - 1.69\ X_T^2 + 0.69\ X_t^2 + 0.77\ X_A^2 - 0.33\ X_T X_A$	0.984	72.44
4	$Yara = 2.19 - 0.20\ X_T - 0.26\ X_t + 0.23\ X_A^2 - 0.07\ X_T \cdot X_t$	0.945	47.33
5	$Yhmt = 16.49 - 1.87\ X_T - 1.93\ X_t - 0.23\ X_A - 1.65\ X_T^2 + 0.74\ X_t^2 + 1.08\ X_A^2 - 0.34\ X_T \cdot X_A$	0.992	138.91
No. Eq	**Extraction liquor equations**	r^2	*F*-Snedecor
6	$Yligex = 30.16 + 5.18\ X_T + 8.55\ X_t + 6.50\ X_T^2 - 3.64\ X_t^2 - 2.81\ X_A^2 + 1.63\ X_T X_A$	0.984	92.69
7	$Ygluex = 12.33 + 0.90\ X_t + 7.11\ X_T^2 - 4.28\ X_t^2 - 3.66\ X_A^2 + 1.05\ X_T X_A + 1.32\ X_t X_A$	0.942	24.41
8	$Yhmtex = 39.80 + 6.83\ X_T + 7.04\ X_t + 0.83\ X_A + 6.01\ X_T^2 - 2.69\ X_t^2 - 3.95\ X_A^2 + 1.22\ X_T \cdot X_A$	0.992	138.91

YligK, klason lignin content in the solid; *Yglu*, glucan content in the solid; *Yxil*, xylan content in the solid; *Yara*, araban content in the solid; *Yhmt*, hemicelluloses conten in the solid; *Yligex*, lignin extracted; *Ygluex*, glucan extracted; *Yhmtex*, hemicelluloses extracted; *XA, XT, Xt*, normalized value of the alkali concentration, temperature, and time, respectively

to extract 52.5% of all hemicellulose in their raw material compared to 25.7–55.1% from elephant grass here.

4 Conclusions

Elephant grass has been deemed an excellent alternative to woody materials for energy production by direct burning, basically by virtue of its substantial contents in lignin (20.86%) and hemicelluloses (27.4%).

Using the optimal operating conditions for pre-burning cold alkaline extraction (CAE) of Elephant grass (viz., an alkali concentration of 100 g NaOH L^{-1}, a temperature of 30 °C, and a treatment time of 30 min) allowed its high heating value to be raised by 4% (to 19.151 MJ kg^{-1}) while preserving the proportion of elemental C and increasing that of H with respect to the raw material, and deriving the environmental advantage of reducing the sulphur content by 46.9% from the starting material to the post-CAE solid phase.

Cold alkaline extraction of the raw material additionally enabled relatively selective separation of the hemicellulose fraction from the cellulose and lignin fractions. Thus, 30.1% of all hemicellulose was dissolved in the treatment liquor and made valorizable while 93.0% of cellulose and 82.1% of lignin present in the raw material remained in the solid phase.

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Declarations

Conflict of interest The authors declare no competing interests.

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