Proceedings of the Canadian Society for Mechanical Engineering International Congress
32nd Annual Conference of the Computational Fluid Dynamics Society of Canada
Canadian Society of Rheology Symposium
CSME-CFDSC-CSR 2025
May 25–28, 2025, Montréal, Québec, Canada

# Characterization of the physicochemical properties of densified fuels produced from the co-processing of sawdust, miscanthus, and wheat straw. Analysis of the impact of the compaction force, the particle size and the feedstock mixing ratio

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Abstract—This work aims at investigating the effects of feedstock properties and compaction force on the physicochemical properties of densified fuel briquettes issued from the co-processing of 3 biomass types (namely wood sawdust (S), miscanthus (M) and wheat straw (WS)) using a hydraulic press machine. To that end, we built a design of experiments integrating two compaction forces (225 and 450 kN), two particle sizes (<1.25 mm and comprised in the 1.25-2.5 mm range) and five proportions of each biomass (ranging from 0 to 100%) in S/M, S/WS and M/WS blends. The intrinsic quality of the obtained fuels was assessed by determining their apparent density, and ash, volatile matter, and fixed carbon contents. The results obtained showed that all the above densification parameters influence the apparent density of the produced briquettes. Values ranging from 512 to 1121 kg·m<sup>-3</sup> were measured, with the highest one determined when considering a high compaction force, small wood particles, and a wood proportion of 100 wt%. Increasing the wood content from 0 to 100 wt% was, moreover, shown to decrease the proportion of ash in the produced briquettes while enhancing their wt% in volatile matters, thus translating into increased fuel energy contents as exemplified by computed net calorific values going from 15.22 to 19.12 MJ·kg<sup>-1</sup>.

Keywords: Biomass; Co-processing; Fuel briquettes, Densification parameters

#### I. Introduction

Growing concerns over the depletion of fossil fuels driven by global energy demand and the increasing emissions of greenhouse gases (GHG) has surged the search for alternative fuels issued from biomass. The overexploitation of traditional biomass resources for domestic use induced by the rapid increase of the global population, however, leads to deforestation, and in turn, contributes to climate change. Beneficiating biomass wastes or energy crops thus represents an interesting alternative. These resources are indeed renewable, carbon-neutral, exhibit low nitrogen and sulfur contents, and are cost-effective and potentially widely distributed. The direct combustion of lignocellulosic biomass, however, presents some challenges due to its low thermal efficiency. Furthermore, biomass typically has a low bulk density (ranging from 80–200 kg·m<sup>-3</sup>, depending on the biomass type [1]), which makes it more complex to handle, transport and store in loose form, thus limiting its economic viability. In this context, densification has proven to be a promising route to convert dried waste (typically containing between 5 and 10% of moisture) into a uniform solid fuel having improved properties (e.g., high energy density) [1], to meet household, commercial and industrial energy needs while substituting firewood and charcoal.

Several densification technologies have been developed to conduct the briquetting process. One can notably cite hydraulic and mechanical presses, which are generally classified according to their compaction pressure [2], which significantly impacts the densification process and the quality of the solid fuels produced [1,2]. As for the feedstock type, wood chips have been considered as the primary raw material for briquette production for several decades [3]. However, due to growing demand and competition limiting access to these chips, forestry and agricultural wastes (e.g., sawdust, straw or rice husks) as well as energy crops (e.g., miscanthus) have been increasingly used to produce densified fuels (see [4-6], as examples).

Past studies demonstrated that the type of biomass, the feedstock properties (notably the particle size) and the densification operating parameters all significantly influence the physicochemical properties of the briquettes produced. As a general trend, the finer the particles when optimal compaction conditions are considered, the stronger and denser the fuel briquettes [6,7]. Using biomass wastes having low ash and high fixed carbon contents, moreover, leads to the production of higher-quality fuels (see [6,7], as examples) whose oxidation exhibits a better combustion efficiency, hence minimizing the emission of air pollutants, notably for cooking applications, as exemplified in [6]. As far as the operating parameters governing the densification process are concerned, increasing the compaction force typically promotes the binding of the particles, which undergo elastic and plastic deformation to form solid

bridges (see [1,2,8] and references therein). Note also that the co-processing of different biomass types can represent an interesting option for producing high-quality solid fuels. Doing so indeed allows to increase the proportion of some binding agents (such as lignin, proteins, and starch) while leading to densified fuels having improved physicochemical properties notably in terms of energy density [2]. The quality of the produced briquettes, however, strongly depends on the nature of the biomass considered and on the compaction conditions [2,8]. Further research, based on a systematic and thorough characterization of the density, composition and energy content of the fuels issued from the co-processing of various biomass wastes must therefore be conducted to elucidate the influence of the biomass properties and densification parameters on the quality of the briquettes produced.

Given the foregoing, the present work aims at assessing the physicochemical properties of solid fuels obtained from the densification of various biomass types. The objective is notably to evaluate the influence of the compaction force, the particle size and the feedstock mixing ratio on the density and chemical properties of the resulting briquettes. Although we already conducted a similar study in [6], only two types of biomass were considered therein, versus three in the present work. Here, we considered widely varying feedstocks, namely, wood sawdust, miscanthus and wheat straw, to represent woody biomass, energy crops, and agricultural residues, respectively, which are largely available in Canada. Each briquette sample was produced using a hydraulic press machine following the methodology described in section II, which also details the procedure implemented to characterize the resulting briquette samples. The obtained results were then analyzed, as detailed in section III, in order to determine the formulation leading to the production of the most interesting densified fuels.

## II. METHODOLOGY

# II-1. Feedstock and sample preparation

As mentioned above, the three types of biomass considered in this work are wood sawdust (referred to as "S"), miscanthus ("M"), and wheat straw ("WS"). These biomaterials, selected for their abundance/availability in Quebec, were provided from wood sawmills and local farms in the province. As for the sample preparation, each feedstock was ground using a cutting mill machine (Retsch, SM300 model) to obtain particles having a size less than 2.5 mm. Note that these particles were then sieved to obtain two different size fractions (< 1.25 mm and between 1.25 and 2.5 mm). The proximate analysis and the heating value of the tested samples are provided in Table 1.

Table 1. Chemical properties of tested biomass samples

	Particle size/mm	Proximate analysis/ wt%				Calorific
Samples		Moisturea	$Ash^b$	Volatiles <sup>b</sup>	Carbon <sup>b,c</sup>	value/ MJ·kg <sup>-1</sup>
S	< 1.25	7.43	1.99	78.48	19.53	18.87
	1.25 - 2.5	8.54	0.99	78.74	20.27	19.12
M	< 1.25	7.39	4.62	74.59	20.79	17.05
	1.25 - 2.5	8.25	2.94	74.67	22.39	17.41
WS	< 1.25	6.61	6.91	74.84	18.25	15.22
	1.25 - 2.5	8.09	3.31	74.92	21.77	16.11

<sup>&</sup>lt;sup>a</sup> as received (ar); <sup>b</sup> dry basis (db); <sup>c</sup> calculated by difference

As can be seen by looking at the reported results, the moisture contents of the considered feedstock globally lie

between 5 and 10%, which is consistent with the recommendations that this parameter not exceed 10% in order to obtain high-quality densified briquettes (see section 1 and references therein).

## II-2. Design of experiments (DoE)

A full factorial design of experiments (DoE) comprising three variables was used to study the impact of the compaction force (CF), the particle size (PS) and the feedstock mixing ratios on the properties of the densified fuels. To that end, two CF values were set (225 and 450 kN), two PS were used (less than 1.25 and between 1.25 and 2.5 mm) and five weight percentages were selected (0, 25, 50, 75, and 100 %) for each feedstock in the blends. Of note, these parameter levels were defined based on preliminary trials which allowed finding specific compaction force ranges leading to densified briquettes that were rigid enough to be manipulated for analysis purposes. Furthermore, the mixing ratios were set to obtain compositions going from pure S to pure M and WS. The DoE resulted in 96 briquette formulations, designated using the following nomenclature: CF'a'-PS'b'-PS'c'-Biomass1/Biomass2'd'/'e', where indicates the level associated with the compaction force (1 for 225 kN and 2 for 450 kN), 'b' and 'c' denote the particle sizes of the first and second feedstocks of the blends (1 for less than 1.25 mm and 2 for within 1.2-2.5 mm), while 'd' and 'e' represent the weight proportion of each biomass type in the blends, respectively.

## II-3. Briquette production

The two-component fuel blends were prepared by weighing the feedstocks using a digital scale (Sartorius, BCE6202 - 1S) in order to obtain the desired mixing ratios. A total mass of  $10\pm0.1$  g of sample was then used to produce the briquettes by means of an automatic hydraulic press machine (MATEST S.p.A. TREVIOLO 24048) equipped with a cylindrical die (having an inner diameter of 50 mm and a height of 50 mm) delivering a maximum compression force of 500 kN.

II-4. Characterization of the briquette samples and statistical analysis of the DoE results

The characterization of the physicochemical properties of the briquettes includes the measurement of their apparent density ( $\rho$ ), the characterization of their volatile matter, ash and fixed carbon contents, and the assessment of their net calorific value. Of note, the measurements were repeated three times. Mean values are thus presented next, noting that error bars in the graphs account for the dispersion of the experimental data around the mean values.

To determine the apparent density ( $\rho$ , expressed in kg·m<sup>-3</sup>), the mass of the sample was measured using a digital balance (Sartorius, BCE6202 – 1S), while the diameter ( $D_s$ ) and height ( $H_s$ ) were measured using a digital caliper. Finally, the density was calculated as follows:

$$\rho = \frac{\text{mass of the briquette sample}}{\pi \times (D_s^2/4) \times H_s} \tag{1}$$

As far as the proximate analysis is concerned, the following procedures were implemented. To determine the ash content (AC, wt%), 1 g of dried fuel was placed in a crucible and

introduced in a furnace (Labtech LEP-230P) at 550 °C for 2 hours until the carbon was eliminated. Thereafter, the sample was removed and cooled down in a desiccator before the ash mass was weighed. The ash content (AC, wt%) was then calculated as follows:

$$AC = \frac{m_{ash} - m_{crucible}}{m_{od}} \tag{2}$$

where  $m_{ash}$ ,  $m_{crucible}$  and  $m_{od}$  represent the mass of the crucible containing the ash after oxidation, the mass of the crucible, and the mass of the dried fuel sample, respectively. For the assessment of the volatile matter content (VMC, wt%), 1 g of sample was placed in a covered crucible, oven-dried at 105 °C using a furnace (VWR 1305U), and then placed into a furnace at 550 °C for 10 minutes. Next, the sample was removed and weighed after being cooled down in a desiccator. The volatile matter content was finally calculated as per (3):

$$VMC = 100 \times \frac{m_i - m_f}{m_i - m_c}$$
 (3)

with  $m_c$ ,  $m_i$  and  $m_f$  being the mass of the crucible, the mass of the crucible containing the oven-dried sample, and the mass of the crucible containing the sample heated at 550 °C, respectively. The estimation of the fixed carbon content of the briquette sample was finally calculated by difference.

Regarding the net calorific value (*NCV*), it was computed using an additivity rule of type:

 $NCV_{briquette} = wt\%B1 \times NCV_{B1} + wt\%B2 \times NCV_{B2}$  (4) where  $NCV_{briquette}$ ,  $NCV_{B1}$ , and  $NCV_{B2}$  represent the net calorific value of the briquette, the first biomass in the blend, and the

second one, while wt%B1 and wt%B2 denote their respective weight percentages in the considered formulation.

A comparative analysis of variance (ANOVA) with a posthoc Tukey honestly significant difference test was conducted to statistically analyze the impact of the production parameters (CF, PS, and mixing ratio) on the density and chemical properties (ash, volatile matter, and fixed carbon contents) of the produced briquettes. All these analyses were performed using the Minitab statistical software version 22, while setting the significance level to 0.05.

#### III. RESULTS AND DISCUSSION

This section presents the results obtained in analyzing the impact of the densification parameters on the properties of the produced briquettes. Note that the discussion below will mainly focus on the F and p values, which represent the variance ratio and the probability factor indicating whether or not the difference between group means is statistically significant (verified when p < 0.05), further noting that for brevity, the detailed results of the statistical analysis of the DoE will not be reported.

III-1. Impact of the densification parameters on the apparent density of the obtained briquettes

The results obtained when measuring the apparent density of the tested briquette samples are summarized in Figure 1.

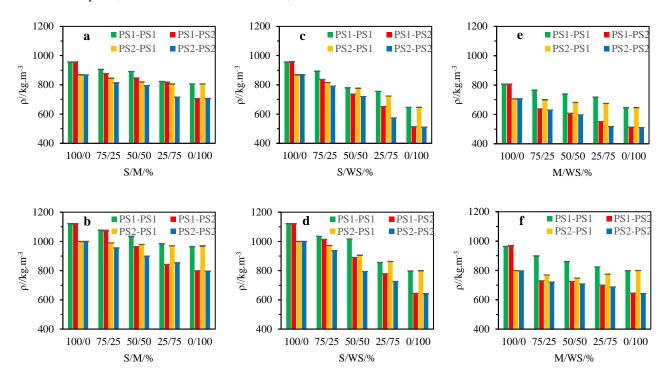


Figure 1. Variation of the apparent density noted 'ρ' as a function of particle size (PS1 = < 1.25 mm and PS2 = 1.25–2.5 mm) and of the S/M (a and b), S/WS (c and d), M/WS (e and f) mixing ratios for compaction force of 225 kN (a, c, and e) and 450 kN (b, d, and f)

As can be seen by comparing the density values reported in Figures 1a, c, e with those issued from Figures 1b, d, f, the

compaction force significantly influences the apparent density of the produced briquettes regardless of the considered feedstocks. This observation is corroborated by the analysis of variance showing that the impact of this parameter on  $\rho$  is statistically significant (F(1,112)=422.11, p<0.001, F(1,112)=464.35, p<0.001, and F(1,112)=646.03, p<0.001 for the S/M, S/WS, and M/WS briquettes, respectively). This trend is actually in line with the conclusions drawn by [9], who traced the rise of  $\rho$  with increasing CF to the elastic and plastic deformation undergone by the particles during their compaction [1,2,8], hence resulting in the formation of strong bridges when high CF are used.

As for the effect of the particle size of the feedstocks on  $\rho$ , the analysis of variance leads to conclude that this parameter significantly impacts the density of the obtained briquettes (F(1,112)=68.70, p<0.001 and F(1,112)=63.38, p<0.001 for Sin the S/M and S/WS blends, respectively, versus F(1,112)=81.21, p<0.001 and F(1,112)=33.81, p<0.001 for M in the S/M and M/WS blends and F(1,112)=129.00, p<0.001 and F(1,112)=682.97, p<0.001 for WS in the S/WS and M/WS blends). More specifically, the data plotted in Figure 1 show that the lower the PS, the higher the apparent density. Note that we previously observed a similar behavior in [6], where the apparent density of briquettes produced from wood and rice husk was shown to increase as the wood particle size was decreased. As mentioned in [6], this phenomenon can be traced to the reduction of the void spaces and to an increase of the contact surfaces between the particles composing the briquettes when finer particles are considered.

Regarding the influence of the S/M, S/WS, and M/WS mixing ratios on the briquette density, it is also shown to be significant (F(4,112)=80.53,statically p < 0.001, F(4,112)=318.03, p<0.001 and F(4,112)=148.92, p<0.001 for S/M, S/WS, M/WS blends, respectively). This is particularly exemplified by the data in Figure 1, which show that p monotonically increases with the S content for the S/M and S/WS blends while it rises with the M content in the case of the M/WS blends. This trend actually agrees with the observations by [4] and [5], who respectively showed that the density of pellets produced from S/M and S/WS blends increases with the S content. This can be explained by the higher lignin content of S (26.5 wt%) as compared to M and SW (between 12.3 and 16.9 wt% according to [10]), noting that lignin especially acts as an in-situ binder [1].

Of note, the apparent density of the duplicated samples (those produced from pure S, M, and WS, as is the case of the CF1-PS1-PS1-S/M100/0 and CF1-PS1-PS2-S/M100/0 formulations, for example) are identical, similarly to the other properties measured and presented in sect. III-2 below. This therefore confirms the good reproducibility of the production tests performed in the present work. Note, however, that for brevity, this point will not be further emphasized below. To conclude, it is noteworthy that the average density values obtained herein are globally comprised between 511.86±0.35 and 1120.57±1.64 kg·m<sup>-3</sup>, with the lowest and highest values being obtained from pure WS and S, respectively. Since, the apparent density gives an indication of the transportation requirement per unit of energy, a higher density is thus preferable, meaning that briquettes containing high proportions of wood sawdust should preferentially be considered.

III-2. Impact of densification parameters on the chemical properties of the obtained briquettes

As mentioned in sect. II-4, the ash, volatile matter, and fixed carbon contents of the densified fuels produced when varying the CF, PS and biomass mixing ratios were measured to elucidate the effects of these parameters on the chemical properties of the obtained briquettes. Figure 2 reports the data obtained when considering S/M and S/WS blends with a compaction force of 225 kN as examples. Since the main trends highlighted in the following sections also apply to the M/WS blends as well as to the tests performed with a CF of 450 kN, corresponding results will not be detailed for brevity.

## III-2-1. Ash

Ash represents the solid residue resulting from the oxidation of biomass. In addition to reducing the net calorific value of solid fuels, elements in ash (including heavy metals) can cause problems arising from the deposition of slag and ash in combustion facilities, thus explaining why the lower the ash content, the better the briquette quality. As shown by the results depicted in Figures 2a and d, the lower the particle size, the higher the ash content, as confirmed by the analysis of variance (F(1,112)=34.16, p<0.001 and F(1,112)=17.42, p<0.001 for S inS/M and S/WS blends, respectively, versus F(1,112)=66.51, p<0.001 for M in the mixtures composed of S and M and F(1,112)=169.27, p<0.001 for SW in the S/WS blends). In addition to agreeing with the conclusions drawn in [6], the fact that the selection of finer granulometries leads to producing briquettes having lower ash contents can be related to the lower proportions of ash contained in the feedstocks sieved to obtain a size fraction between 1.25 and 2.5 mm as exemplified in Table 1.

The analysis of variance, moreover, indicates that the mixing ratio significantly influences the ash content of the briquettes (F(4,112)=83.80, p<0.001, F(4,112)=85.54, p<0.001, and F(4,112)=10.82, p<0.001 for the S/M, S/WS and M/WS blends), as can be seen by looking at the graphs of Figures 2a and d. Specifically, the greater the M and WS wt%, the larger the ash content of the briquettes, which is in line with expectations, since agricultural residues and energy crops generally contain more ash than woody biomass, as confirmed by the data reported in Table 1. Note also that a similar trend was also observed by [11], who noted that the ash content of densified fuels produced from wood and miscanthus increased with the M content.

To conclude, one can note that the ash content of the densified fuels produced in the present work falls between  $0.99\pm0.01$  and  $7.00\pm0.04$  wt% with the lowest values obtained when considering PS comprised in the 1.25-2.5 mm range and relatively high proportions of S, which thus represents the conditions recommended for producing quality briquettes.

## III-2-2. Volatile matters

Volatile matters denote the volatile fuels and flammable hydrocarbons which are emitted when the fuel is heated to high temperatures. Since the burning of these volatile matters typically accounts for about two-thirds of the total energy released by a wood fire, the higher the VMC, the more reactive and easily ignited the fuel. While the plots of Figures 2b and e show that the PS does not influence the VMC of the briquettes,

they conversely show that the feedstock mixing ratio significantly impacts this property, as corroborated by the results of the analysis of variance (F(4,112)=131.12, p<0.001 and F(1,112)=169.16, p<0.001 for the S/M and S/WS blend, respectively). The VMC is notably shown to monotonically decrease from  $78.74\pm0.21$  wt% to  $74.67\pm0.43$  wt%, versus

74.33±0.27 wt% when the proportions of M and WS in the S/M and S/WS blends goes from 0 to 100 wt%. This trend, which is consistent with the fact that the VMC in M and WS is lower than that in S (see Table 1), thus indicates that a high proportion of S should preferentially be considered in producing reactive fuel briquettes.

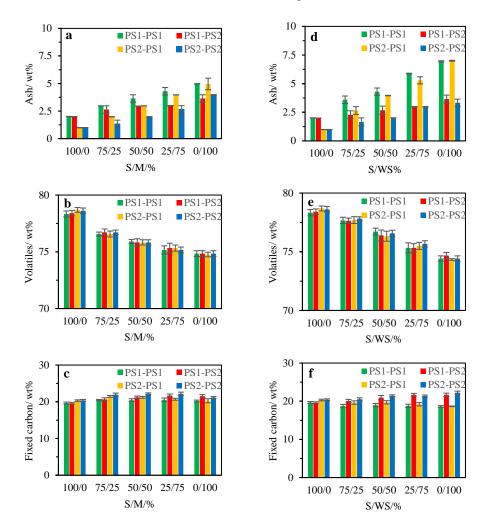


Figure 2. Evolution of the ash (a and d), volatile matters (b and e), and fixed carbon (c and f) contents of S/M (a, b and c) and S/WS (d, e and f) briquettes produced using a compaction force of 225 kN with different particle sizes (noted PS1 and PS2)

## III-2-3. Fixed carbon

The fixed carbon is the mass remaining after the release of the VM, excluding the ash and moisture contents. While the data in Figures 2c and f show that FC contents ranging between 18.21±0.23 wt% and 22.25±0.39 wt% are obtained, depending on the selected operating parameters, the statistical analysis of the DoE results indicate that both PS (F(1,112)=10.38, p=0.002 and F(1,112)=8.84, p=0.004 for S in S/M and S/WS blends, respectively, versus F(1,112)=35.41, p<0.001 for M in the mixtures composed of S and M and F(1,112)=107.68, p<0.001 for SW in the S/WS blends) and the mixing ratio (F(4,112)=11.03, p<0.001 and F(4,112)=1.61, p=0.007 for the S/M and S/WS blends) significantly influence the FC content of the produced briquettes. These results could still have been anticipated since the ash content is impacted by both the

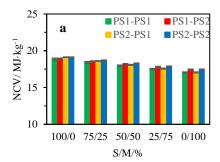
granulometry and the mixing ratio, the latter parameter having been shown to also influence the VMC (see sects. III-2.1 and III-2.2). Given that the fixed carbon content is computed by difference based on the proportion of ash and volatile matters contained in the briquette samples, it is therefore logical that the densification parameters affecting the ash and volatile matter contents also influence the fixed carbon content.

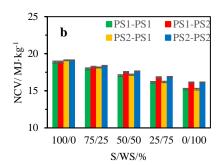
## III-2-4. Net calorific value

The net calorific value represents the amount of heat released by a unit quantity of fuel during combustion in the presence of oxygen, without considering the energy given off by water vapor during condensation. As mentioned in sect. II-4, this property was computed based on the NCV of the biomass samples, using Equation (4). As can be seen by looking at the results detailed in Figure 3, the NCV of the produced briquettes

increases from 15.22 $\pm$ 0.11 and 17.05 $\pm$ 0.08 MJ·kg<sup>-1</sup> to 19.12 $\pm$ 0.05 MJ·kg<sup>-1</sup> when the S content in the S/M and S/WS goes from 0 to 100 wt%, respectively. Note that a similar trend is observed when increasing the proportion of M in the briquettes produced from M/WS mixtures. This observation is consistent with the fact that the NCV of the considered feedstocks decreases in the following order: S > M > WS, as illustrated in Table 1. To conclude, it is noteworthy that the

NCV of the densified fuels issued from the use of fine particles is slightly lower than that of the briquettes obtained while considering coarser ones. This observation is still consistent with the fact that the NCV is influenced by both the ash and volatile matter contents of the fuels [6]. As a result, briquettes containing more ash, for instance (i.e., those produced considering fine particles, as detailed in section III-2-1), will tend to have a lower net calorific value.





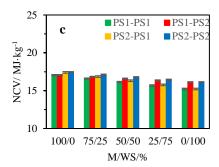


Figure 3. Variation of the net calorific value (NCV) of the produced briquettes as a function of the particle size (PS1 and PS2) for the S/M (a), S/WS (b), and M/WS (c) blends

#### IV. CONCLUSION

The present study aimed at investigating the influence of various densification parameters on the quality of fuel briquettes produced from blends containing woody biomass, agricultural waste and energy crop. The results obtained as part of this work in progress showed that the compaction force, the particle size, and the mixing ratio all significantly influence the briquette density, whose values were found to vary between 512 and 1121 kg·m<sup>-3</sup>. Specifically, higher compaction forces, finer particles and higher proportion of S (in the case of S/M and S/WS blends) and M (in the case of M/WS blends) proved to be more adapted for producing briquettes exhibiting a higher density. As for the proximate analysis of the densified fuels produced, we observed that both the granulometry and the feedstock mixing ratio significantly influence the ash content of the briquettes, while only the latter factor proved to impact the VMC. Finally, NCV between 15.22 and 19.12 MJ·kg<sup>-1</sup> were found, noting that the finer the particles, the higher the ash content and the lower the NCV. Although the results presented herein tend to show that using biomass residues blended with forestry biomass can be an interesting option for producing alternative fuels meeting household and industrial needs while providing an eco-friendly waste management option, further work is still necessary, and is thus in progress in our lab, to characterize the water and impact resistance indexes, as well as the combustion properties of the produced densified fuels.

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