

## Article

# Improving the Physical, Mechanical and Energetic Characteristics of Pine Sawdust by the Addition of up to 40% *Agave durangensis* Gentry Pellets

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**Abstract:** *Agave durangensis* Gentry biomass, as a residue from the mezcal production process, may be an interesting bioenergy alternative; however, its high ash content limits its application. In this study, pellets were generated with agave fiber mixed with *Pinus* species sawdust in the following six proportions (%): 100–0 (control), 80–20, 60–40, 40–60, 20–80 and 0–100 (control). The physical, chemical and energetic properties of the pellets were evaluated according to the UNE-EN ISO 17225-6, UNE EN ISO 17827-2, UNE-EN ISO 17828, UNE-EN ISO 18122, UNE-EN ISO 18123, UNE-EN ISO 18125, and UNE-EN ISO 18134-1 standards. The results showed significant statistical differences ( $p < 0.05$ ) among the treatments tested. The percentage of volatile material and fixed carbon ranged from 86.53 to 89.96% and 4.17 to 8.16%, respectively; the ash content ranged from 0.27 to 10.06%, and the calorific value ranged from 17.33 to 18.03 MJ/kg. Bulk density ranged from 725.76 to 737.37 kg/m<sup>3</sup> and the impact-strength index was in the range of 69.33 to 126.66. The mechanical hardness and compressive strength were found to be in the ranges of 50.5 to 68.4% and 0.90 to 36.65 N/mm, respectively. Pellets generated with *Agave* residue mixture  $\leq 40\%$  were identified as promising biobased resources for the sustainable production of renewable energy.

**Keywords:** *Agave durangensis*; pellets; *Pinus* spp.; physical and energetic properties



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## 1. Introduction

Global climate change and greenhouse gas emissions are the result of environmental pollution derived from human activities. The generation of electrical energy based on the burning of fossil fuels, land use change and forest fires are among the main causes of such trends [1].

The instability of the fossil-fuel market, concerns about fossil-fuel depletion, environmental protection, and the energy dependence of countries with scarce conventional energy resources have driven the development of renewable energies [1].

Bioenergy production with biomass from agricultural and forestry residues is a sustainable activity that supplies energy to millions of people, and offers income and employment opportunities in rural areas where small producers can improve their economy through the sale and/or production of biomass and bioenergy [2]. Biofuels represent a potential source of renewable energy, have the potential to generate new and large markets for agricultural and forestry producers, and can replace the consumption of traditional fossil fuels such as oil and coal [3]. Furthermore, the products that can be generated from biorefineries are sources of natural carbon, can reduce the threat of climate change and meet the demands for fuels, materials and energy [4]. The products obtained from biorefinery are diverse,

can be incorporated into noodles, snacks, beverages, pasta, wine, breakfast mueslis, candy, cosmetic products, bio-fertilizers, pigments, as well as nutraceuticals, and can also improve the nutritional profile of products used in food and animal feed [5,6].

Pellets generated with forest biomass provide an important alternative for energy generation; however, due to the accelerated growth in demand and the impossibility of producing more wood waste, it is necessary to evaluate different sources of biomass that meet certain international quality standards [7].

Industries of small to medium scale in developing countries that are based on traditional technologies depend on biomass to generate heat to process and dry the final product. Most of them buy their biofuel, but some also collect it from free or relatively low-cost sources, which lowers their cost and solves logistical problems if it is densified on site [8].

Agro-industrial agave residue has gained great interest worldwide as a raw material for the generation of various products [9]. It has high bioenergy potential due to its high productivity in semiarid ecosystems [10]. It is a residual fibrous material resulting from the extraction of the fermented agave pineapple, and approximately 40% of the total weight of agave corresponds to bagasse (residues) [11]. During the process of obtaining mezcal, 15 to 20 kg of this type of waste is produced for each liter [12].

In Mexico, the main mezcal-producing states are Durango, Guanajuato, Jalisco, Nayarit, Oaxaca, San Luis Potosí, Tamaulipas and Zacatecas. Durango has a moderate number of producers and in 2015 alone, total mezcal production amounted to 243,900 L, generating approximately 3659 tons of bagasse (wet basis) [13]. Notwithstanding the high availability of agave fiber and other similar biomass sources, some characteristics of the material may limit its use as an energy source. *Agave durangensis* fiber, from which mezcal is produced, has a holocellulose content of 44.72%, 7.63% lignin content and 12.65% ash content, which could limit its use as pellets [14].

Pellets with adequate physical, mechanical and energetic characteristics made from various biomass sources must also meet other requirements established in international standards such as a low percentages of ash due to the high risk of slag formation [15]. The search for opportunities to utilize agave residues requires the physical and energetic characterization of such materials to establish their quality in order to define the most appropriate technologies for their utilization and their subsequent use as a source of renewable fuels.

An alternative to reducing the amount of wastes produced is to transform them into bioenergy products and thus reduce their environmental impact. The densification of biomass into pellets involves compacting them to reduce their volume and to maximize their efficiency as an economic and ecological energy source, making mixtures with other residues such as those resulting from the transformation of pine wood (sawmilling). The objective of this research was to improve the physical, mechanical, and energetic characteristics of pine sawdust via the addition of *Agave durangensis* Gentry residues.

## 2. Materials and Methods

### 2.1. Collection and Treatment of Material

*Agave durangensis* residues were collected from the mezcal producer Hacienda Dolores S.P.R. de R.L. located in the city of Durango, Mexico. This material is composed of  $35.57 \pm 0.41\%$  cellulose,  $9.42 \pm 0.49\%$  hemicellulose and  $16.08 \pm 0.15\%$  lignin [16], the content of the total extracts (cyclohexane, acetone, methanol, hot water) is  $32.44 \pm 0.25\%$ , the pH is  $5.84 \pm 0.08$  and lignin accounts for  $7.62 \pm 0.31\%$ . The inorganic elements concentration is  $2.21 \pm 0.29$ , undetected,  $58.84 \pm 1.13$ , undetected,  $1.42 \pm 0.1$  and  $11.90 \pm 0.16\%$  for aluminium, sulphur, calcium, chlorine, phosphorous, and magnesium, respectively [14,17]. Pine sawdust was collected from a sawmill located in the city of El Salto, Durango, Mexico. The biomass was conditioned at room temperature in a tarp for approximately 2 to 3 weeks until reaching moisture content of less than 12%. Subsequently, the material was ground in a hammer mill (TFS 420) with a 3.15 mm mesh. Six mixtures were created with different proportions of agave-pine residues (Ti, %); in two of them, only *Agave durangensis* biomass

and pine sawdust were used separately as the controls (T1 and T6, respectively). The treatments for making pellets were as follows: T1 = 100–0, T2 = 80–20, T3 = 60–40, T4 = 40–60, T5 = 20–80 and T6 = 0–100. The pelletizer (ZLSP-R300) had a flat disc with channels of 8 mm in length and 6 mm wide, which produced pellets at a rate of 400 kg h. For pellet manufacturing, 20 kg of each mixture was placed in rubber bags and mixed homogeneously; then, the material was mechanically transported to feed the pelletizer so as to form the pellets at a temperature of 110 °C according to the procedure of Núñez-Retana et al. [18], and around 15 kg of pellets per mixture was formed. The procedure began by palletizing the pine sawdust to increase the temperature of the rollers and plate of the pelletizing machine until reaching a temperature of 110 °C, which was measured with a dual laser thermometer type K. Once the pelletizing temperature was reached with the pine sawdust, the pelletizing of the treatment (T6) containing 100% pine sawdust was started, so as not to abruptly change the type of material. After pelletizing all the material for T6, pine sawdust was pelletized again so that the pelletizer could stabilize once more, then the temperature of the rollers was checked and the pelletizer continued with T5, at a mixture of 20:80. After pelletizing this treatment, pine sawdust was used again to stabilize the pelletizer, after which pelletizing continued with T4 until reaching T1—pine sawdust was used between the pelletizing of each treatment. The pellets were cooled to room temperature for 24 h and stored in plastic bags for later analyses.

## 2.2. Characterization of the Base Material

The treatments were analyzed to determine the particle-size distribution according to the UNE-EN ISO 17827-2 standard [19]. The moisture content was determined by placing 1 g of biomass from each treatment in a Petri dish and then in a muffle for 4 h at  $105 \pm 2$  °C; the values were calculated according to the UNE EN ISO 18134 [20] standard.

Volatile materials were identified with 1 g of biomass from each treatment in a muffle at  $900 \pm 10$  °C for 7 min; the values were calculated according to the UNE EN ISO 18123 [21] standard.

The ash content was determined according to the UNE EN ISO 18122 standard [22]. Then, 1 g of the sample was placed in a muffle at 250 °C for 1 h, after which the temperature was increased to 550 °C and the sample was maintained in this condition for 2 h.

Fixed carbon was determined by calculating the difference from 100 of the moisture content, ash, and volatile-matter fraction values [23].

The percentages of particles of each sieve size and the results of the characterization of the treatments are presented in Tables 1 and 2, respectively.

**Table 1.** Particle-size distribution of the raw material in *Agave durangensis* fibers and *Pine* spp. sawdust mixtures (Sd = standard deviation).

Sieve Number	T1		T2		T3		T4		T5		T6	
	%	Sd										
12	0.30	0.11	0.18	0.12	0.09	0.02	0.11	0.02	0.06	0.03	0.01	0.01
14	0.21	0.04	0.33	0.02	0.42	0.01	0.83	0.19	1.36	0.40	0.85	0.07
20	2.25	0.08	9.70	0.80	20.02	0.36	32.86	3.74	47.81	2.62	48.26	0.53
30	6.67	0.27	11.45	0.66	14.94	1.23	16.11	1.33	14.22	1.56	18.57	0.86
40	18.00	0.32	16.02	0.63	15.85	0.25	14.97	0.27	13.51	1.08	13.82	0.25
60	20.80	0.58	18.78	0.02	14.60	1.01	11.89	1.26	8.86	0.69	8.81	0.63
100	16.50	1.23	15.11	0.21	11.94	1.00	8.35	0.80	5.29	0.24	4.32	0.33
>100	35.26	1.46	28.42	1.37	22.14	1.57	14.89	1.01	8.89	0.47	5.37	0.58

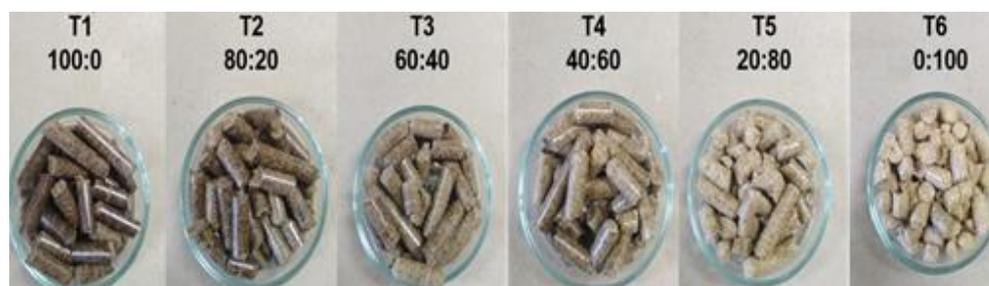
**Table 2.** Proximal analysis of *Agave durangensis* fibers and *Pine* spp. sawdust mixtures (Sd = standard deviation).

Treatment	Moisture Content (%)		Volatile Material (%)		Ash (%)		fixed Carbon (%)		Higher Heating Value (MJ/kg)	
	Average	Sd	Average	Sd	Average	Sd	Average	Sd	Average	Sd
T1	6.54	0.12	82.28	1.09	10.57	1.14	7.16	1.12	14.50	0.52
T2	5.97	0.15	86.24	2.59	6.50	0.61	7.26	1.99	14.08	0.12
T3	6.42	0.05	87.91	0.52	5.82	0.48	6.27	0.95	14.42	0.10
T4	6.67	0.06	88.09	2.28	4.76	0.65	7.15	2.27	14.92	0.11
T5	6.64	0.11	86.82	1.76	4.22	0.50	8.96	1.36	15.85	0.09
T6	7.70	0.10	88.34	1.73	0.25	0.11	11.91	1.78	18.88	0.99

The degree of slagging was developed according to the rapid test developed by [24], where approximately 5 g of particles of each base material was placed in a crucible, heated in a muffle to a temperature of 1100 °C at a constant rate of 5 K/min, and after the final temperature was reached, it was maintained for 30 min. The sample was removed from the furnace and cooled to room temperature. The procedure was carried out in triplicate. The degree of slagging was determined by a visual inspection of the treated sample and values of 0 (does not form slag) and 1 (if it forms slag) were allocated. We found that *Agave durangensis* fiber does not form slag, while pine sawdust does form slag.

### 2.3. Pellets Processing

Pellets were made with a Mill model ZSLP-R300 flat die pelletizer with a capacity of 250–350 kg/h, following the recommendations of Núñez-Retana et al. [18]. After processing, the pellets were cooled room temperature, sieved to remove dust particles and impurities and stored in airtight plastic bags. The pellets were colored in different shades depending on the proportion of treatments (Figure 1).

**Figure 1.** Colors taken by the pellets made with different mixtures of *Agave durangensis* fibers (T1) and *Pine* spp. sawdust (T6) [Ti = *Agave durangensis* fibers: *Pinus* spp. Sawdust].

### 2.4. Pellet Characterization

#### 2.4.1. Proximal Analysis

The values of the variables of moisture content, volatile material, ash content and fixed carbon of the pellets were determined with the procedure reported in Section 2.2.

#### 2.4.2. Physical and Mechanical Properties

The bulk density of the pellets was determined according to the UNE-EN ISO 17828 standard [25], while particle density was determined 1 and 7 days after processing, once they reached hygroscopic equilibrium in a conditioning room at 20 °C and 60% relative humidity. Then, the pellets were weighed and measured (diameter and length) with a digital vernier; the calculation of particle density consisted of dividing the total weight of the pellet by the volume. The reduction in density (the parameter that determines the quality of the pellet) was determined by the difference in the percentage between the

density of the pellet at the time of processing (day 1) and 7 days later [26]. In the same way, the energetic density was calculated according to Garcia et al. [27].

Mechanical hardness was estimated using the drop test, which is a parameter used to determine the hardness of the pellets since it establishes their ability to withstand different conditions and consisted of determining the weight retained in each pellet when dropped twice from a height of 1.85 m onto a concrete floor. Fifteen replicates per treatment were performed. The impact-resistance index (IRI) was calculated by Richards [28].

#### 2.4.3. Calorific Value

The calorific value of the treatments was determined in accordance with the provisions of the UNE-EN ISO 18125 standard [29], for which a semi-automatic AC600 isoperibol calorimeter was used.

### 2.5. Statistical Analysis

The response variables of moisture content, volatile material, ash content, fixed carbon, the calorific value, bulk density, particle density, mechanical hardness (1 and 7 days) and the impact-resistance index (1 and 7 days) were analyzed using a completely randomized design. In cases where statistically significant differences were observed among treatments ( $p < 0.05$ ), Tukey's mean comparison test was used.

The response variables that showed noncompliance in any of the variance normality assumptions (normality of data and equality of variance), according to the Shapiro–Wilk or Lillie tests, were statistically analyzed with the Kruskal–Wallis nonparametric test. All analyses were performed with the “*Im*” package implemented in R [30].

## 3. Results

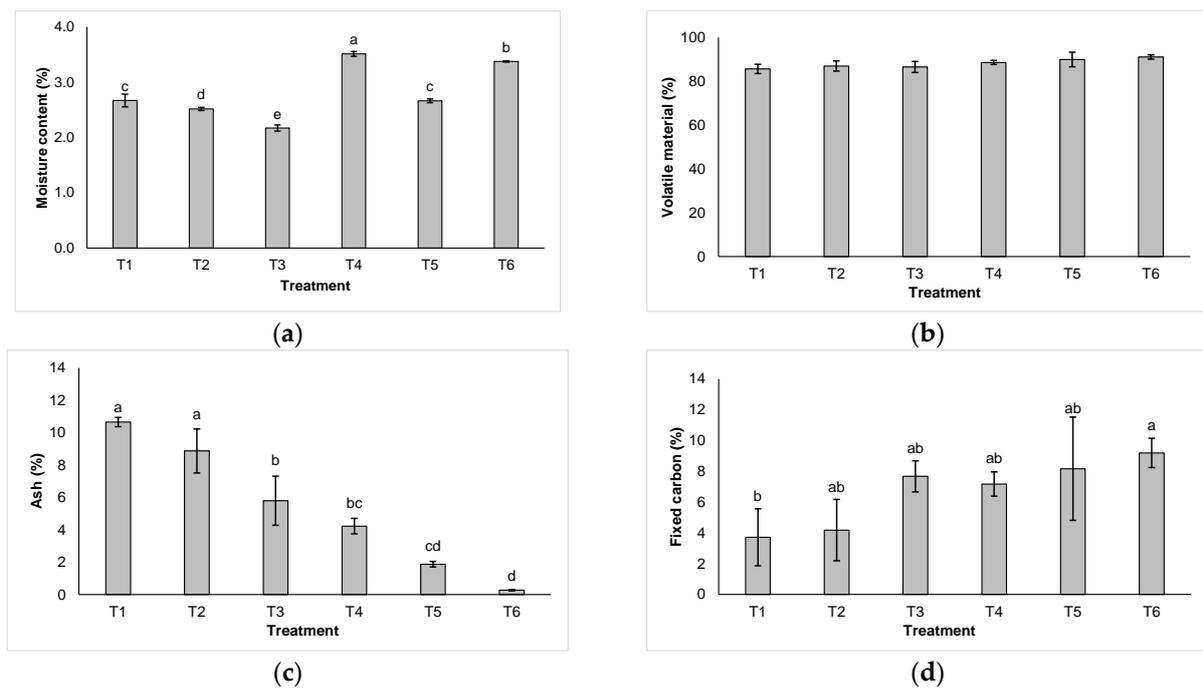
### 3.1. Proximal Analysis

The results of the normality tests and analysis of variance (ANOVA) are presented in Table 3. With the exception of moisture content, the rest of the variables showed a normal distribution of their values ( $p > 0.05$ ). On the other hand, except for volatile material content, all variables showed statistically significant differences ( $p < 0.05$ ), as shown in Table 3.

**Table 3.** Normality tests and analysis of variance of proximal analysis of pellets made with *Agave durangensis* fibers and *Pinus* spp. sawdust mixtures.

Variable	Shapiro–Wilk		ANOVA		Kruskal–Wallis
	<i>w</i> Value	<i>p</i> Value	<i>f</i> Value	<i>p</i> Value	
Moisture content (%)	0.88	0.026	-	-	<0.01
Volatile material (%)	0.98	0.984	2.77	0.0686	-
Ash (%)	0.91	0.087	64.18	<0.001	-
Fixed carbon (%)	0.95	0.467	4.2	<0.05	-

The moisture content was statistically different ( $p < 0.01$ ) among the treatments. The highest value was found for T4 (3.51%), while the lowest value was found for T3 (2.17%) (Figure 2a). However, as can be seen in Figure 2b, there was a positive trend in moisture content as the percentage of pine sawdust in the treatments increased, starting with a value of 85.63% in T1 and reaching a maximum value of 91.10% in T6. The ash content was statistically different ( $p < 0.001$ ) among the evaluated treatments; the lowest value occurred in T6 (0.27%), while the highest value was present in T1 (10.06%) (Figure 2c). Finally, fixed carbon also showed significant statistical differences among treatments, with T1 presenting the lowest value (3.71%) and T6 the highest value (9.20%) (Figure 2d).



**Figure 2.** Proximate analysis of pellets produced from mixtures of *Agave durangensis* and *Pine* spp. sawdust: (a) moisture content (%); (b) volatile material; (c) ash (%), and (d) fixed carbon (%). Different letters indicate statistically significant differences among treatments by the Tukey or Kruskal–Wallis tests ( $p < 0.05$ ).

### 3.2. Physical and Mechanical Properties

The Shapiro–Wilk and Lillie normality tests for the variables characterizing the physical and mechanical properties of the pellets confirm that the bulk density and hardness on day 7 followed a normal distribution ( $p < 0.05$ ), while particle density (day 1 and 7), hardness (day 1) and the impact-resistance index (day 1 and 7) rejected the hypothesis of following a normal distribution (Table 4). On the other hand, apart from hardness on day 7, all variables showed significant statistical differences among treatments ( $p < 0.01$ ) (Table 4).

**Table 4.** Normality tests and analysis of variance of physical and mechanical properties of pellets made with *Agave durangensis* fibers and *Pinus* spp. sawdust mixtures.

Variable	Shapiro–Wilk		Lillie Test		ANOVA		Kruskal–Wallis	
	$w$ Value	$p$ Value	$d$ Value	$p$ Value	$f$ Value	$p$ Value	Chi-Square	$p$ Value
Bulk density	0.899	0.055		.	8.34	<0.01	-	-
Particle density (Day 1)	-	-	0.21	<0.001	-	-	75.2	<0.001
Particle density (Day 7)	-	-	0.16	<0.001	-	-	83.8	<0.001
Relaxed particle density	-	-	0.20	<0.001	-	-	-	-
Hardness (Day 1)	-	-	0.11	<0.01	-	-	8.1	0.15
Hardness (Day 7)	-	-	0.07	0.339	24.65	<0.001	-	-
IRI (Day 1)	-	-	0.32	<0.001	-	-	44.6	<0.001
IRI (Day 7)	-	-	0.22	<0.001	-	-	62.3	<0.001

The bulk density in the treatments was higher than established ( $>600 \text{ kg/m}^3$ ) by the UNE-EN ISO 17225-6 standard and within the range of  $600$  to  $750 \text{ kg/m}^3$  [31]. In the treatments containing agave fiber, the value was higher ( $726$ – $737 \text{ kg/m}^3$ ) than for T6, in which only pine sawdust was used ( $693 \text{ kg/m}^3$ ) (Table 5). The particle density evaluated on days 1 and 7 increased as the proportion of agave fiber in the treatments increased. The value of the particle relaxation index (RPD) was higher when only pine sawdust was used (T1, 13.13%) compared to the treatments with a mixture of both residues. When observing

the behavior of the value of this index, a tendency for its value to decrease was observed as the percentage of agave fiber increased in the treatments, with the lowest value reported in T1 (0.61%) (Table 5).

**Table 5.** Physical and mechanical properties of pellets made with *Agave durangensis* fibers and *Pinus* spp. sawdust mixtures.

Treatment	BD (kg/m <sup>3</sup> )		PD (Day 1) (g/cm <sup>3</sup> )		PD (Day 7) (g/cm <sup>3</sup> )		RPD (%)		Hard (Day 1) (%)		Hard (Day 7) (%)		IRI (Day 1)		IRI (Day 7)								
	Average	Sd	Average	Sd	Average	Sd	Average	Sd	Average	Std.	Average	Sd	Average	Sd	Average	Sd							
T1	728	12	a	1.4	0.35	a	1.3	0.05	a	0.6	2.1	d	51.4	8.8	48.5	9.5	c	114.7	28.0	a	126.5	25.7	a
T2	726	11	a	1.3	0.04	b	1.3	0.05	b	1.6	1.3	cd	52.3	15.6	48.7	16.1	c	116.2	50.7	a	133.8	50.7	ab
T3	737	7	a	1.3	0.04	c	1.2	0.05	c	2.5	1.7	bc	54.9	10.9	53.5	9.5	c	111.8	30.8	a	109.4	29.4	ab
T4	736	9	a	1.2	0.04	d	1.2	0.04	d	3.3	1.9	b	58.5	7.9	51.8	12.0	c	102.9	29.2	ab	110.0	33.3	b
T5	735	11	a	1.2	0.06	e	1.1	0.07	e	9.8	2.2	a	43.9	20.7	68.0	7.4	b	98.2	54.1	b	70.0	17.4	c
T6	694	7	b	1.0	0.06	f	0.9	0.06	f	13.1	4.1	a	53.1	12.6	80.5	7.3	a	64.1	8.9	c	54.1	9.9	d

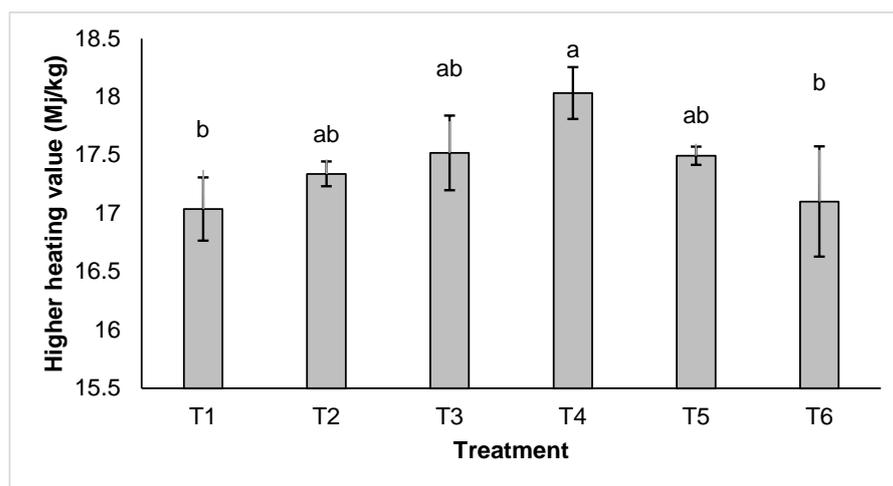
BD = Bulk density, PD = Particle density, RPD = Relaxed particle density, Hard = Hardness, IRI = Impact resistance index, Sd = Standard deviation. Different letters in columns indicate statistically significant difference among treatments by the Tukey and Kruskal–Wallis tests ( $p < 0.05$ ).

The hardness evaluated on day 1 was statistically similar for all treatments; however, when evaluated on day 7, it was higher in those treatments with a higher percentage of pine sawdust. The impact resistance index evaluated on days 1 and 7 showed significant statistical differences; in both cases there was a marked tendency for its value to increase as the percentage of pine sawdust increased in the treatments (Table 5).

On the other hand, the IRI was statistically different among treatments. The value of this index decreased as the proportion of pine sawdust increased; for day 1 the value ranged from 114.72 to 64.12 for T1 and T6, respectively, while for day 7 the value was in the range of 126.47 to 64.14 for T1 and T6, respectively.

### 3.3. Calorific Value

The calorific values revealed statistical differences among the treatments ( $p < 0.05$ ) (Table 6). The highest value was recorded in T4 (18.03 MJ/kg), followed by T3, T2 and T5 with values of 17.52, 17.49 and 17–34 MJ/kg, respectively. In contrast, the lowest values were recorded in treatments T6 and T1 (17.10 and 17.04 MJ/kg, respectively) (Figure 3).



**Figure 3.** Highest heating value of pellets produced from mixtures of *Agave durangensis* and *Pine* spp. sawdust. Different letters indicate statistically significant difference among treatments by Tukey test ( $p < 0.05$ ).

**Table 6.** Normality and analysis of variance tests of the highest heating value of pellets made with *Agave durangensis* fibers and *Pinus* spp. sawdust mixtures.

Variable	Shapiro–Wilk		ANOVA	
	w Value	p Value	f Value	p Value
Highest calorific value (MJ/kg)	0.979	0.9373	4.95	<0.05

The comparison of means with Tukey's test indicated that treatments T2, T3 and T5 presented statistically similar values (ab), while T1 and T6 (b) were also similar and, finally, T4 (a) did not present any equality, registering the highest calorific value (Figure 3).

Finally, the energetic density decreased from 12.4 to 11.9 GJ/m<sup>3</sup> for T1 to T6.

## 4. Discussion

### 4.1. Proximal Analysis

The moisture content is one of the most important parameters that determines many of the properties of pellets including their mechanical durability [32]. Similarly, a high moisture content prevents the material from reaching the required combustion temperature, generates a large amount of steam and CO<sub>2</sub> emissions, and causes damage to the combustion equipment. The moisture content of the pellets made with different mixtures of agave fiber and pine sawdust, despite statistical differences, was relatively low in all treatments (2.17 to 3.33%). In all treatments, moisture content values of less than 10% were recorded, which is a parameter established as the maximum admissible value in the European UNE EN ISO 17221-2 standard [31,33], and they were also lower than the 10.28% reported for pellets based on residual wood sawdust and anthracite coal in a 50–50 ratio by Boada and Vargas [34]. These authors additionally highlighted that the moisture content in the pellets had a negative influence on the net calorific value and on the combustion efficiency and temperature. On the other hand, once pellets are cooled, they can present moisture values of between 8 and 10% because they reach equilibrium with the temperature and relative humidity of the environment [35].

Moreover, the content of volatile material was high and did not present significant statistical differences among treatments ( $p > 0.05$ ). The volatile material content can vary from 70 to 90% depending on the type of biomass from which the pellet is made, with higher values providing a higher thermal capacity [36]. The high values provide a greater thermal capacity in the combustion processes, which favors a quick ignition but short duration of the combustion process [37]. The values obtained coincide with those reported by Ragland et al. [36] and were also similar to those reported by Castillo et al., (2014) with values between 84.49 and 89.28% for pellets made with mixtures of husk and oil palm fiber with different proportions [38]; however, they were higher at 46.23% and 53.15% with respect to pellets made with ground pecan (walnut) husk biomass [39,40]. Similarly, Romo-Ortega et al. (2011) detected 73.63% volatile matter for coffee stalks in mixtures of oak sawdust and pine chips with values of 86.3% and 72.2%, respectively [40].

The highest ash content (10.65%) was recorded in T1 (100–0), on the other hand, treatments T5 and T6 had lower values. The ash content value of T1 (12.1%) coincides with the value reported for *Agave durangensis* bagasse by Gurrola-Armendáriz [41]. A high ash content in fuels can affect combustion equipment and increase maintenance costs due to the required cleaning processes and slag formation [42]. However, in this work, the ash content was reduced to acceptable values of 8.87, 5.79, 4.22 and even 1.86% when the pellets were made with pine sawdust at proportions of 20, 40, 60 and 80%, respectively (Figure 2c). These values are in agreement with those reported by [34] for pellets made with sawdust from *Pinus leiophylla*, *P. montezumae* and *P. pseudostrabus* (0.30%, 0.22% and 0.24%), respectively.

The percentage of fixed carbon in the pellets increased proportionally to the increase in the percentage of pine sawdust. The highest value (9.19%) was recorded in T6 (100% pine sawdust), while the lowest value (3.71%) was recorded in T1 (100% agave fiber) (Figure 2d).

A low fixed carbon content increases friability and brittleness and decreases compressive strength and cohesion [43]. Therefore, the addition of pine sawdust had a positive influence on pellet quality.

#### 4.2. Physical and Mechanical Properties

Bulk density in all treatments was higher than  $600 \text{ kg/m}^3$ , a parameter established as a minimum value in the EN UNE ISO 17225-6 standard [33]. When comparing the values between T1–T5 they were found to be statistically similar ( $727\text{--}737 \text{ kg/m}^3$ ), while the lowest value ( $694 \text{ kg/m}^3$ ) was recorded in T6 where only pine sawdust was used (Table 5). These values can be attributed to the high number of small particles present in the agave fiber biomass (Table 1); the use of a small particle size increases bulk density [44], because the pellets formed have a higher content of matter and could be more durable [32]. A low bulk density negatively affects the energy capacity of biofuels (less energy per unit volume) and therefore increases the costs related to storage and transportation [45].

Regarding particle density, this variable presented statistical differences ( $p < 0.001$ ) among treatments, both in freshly processed pellets (day 1), as well as those conditioned for 7 days (Table 5). On day 1, values ranging from  $1.03$  to  $1.39 \text{ g/cm}^3$  were recorded, while on day 7 the values ranged from  $0.90$  to  $1.31 \text{ g/cm}^3$ . Notwithstanding this variation, these values are in the range reported by Gaitán-Álvarez et al. (2017) [46] for pellets made from oil palm ( $1.07\text{--}1.28 \text{ g/cm}^3$ ) but greater than the values reported by Castillo et al. [38] ( $0.87\text{--}0.77 \text{ g/cm}^3$ ) for pellets made from coconut palm. These values are also higher than the  $0.82\text{--}0.97 \text{ g/cm}^3$  found for pellets made from mixtures of pod husk with cassava starch [47]. In the present study, an increase in particle density was observed as the percentage of agave fiber increased in the treatments; this can be attributed to the higher percentage of small particles contained in the pellets where agave fiber was included (Table 1). The effect of particle size and moisture percentage on particle density was also reported by Mani et al. [48].

When comparing the relaxation index values, statistical differences were found among treatments, with the highest value (13.13%) recorded in T6. These values decreased as the percentage of agave fiber in the pellets increased until reaching 0.61% in T1 (Table 5); this behavior can be attributed to the volume occupied by the *A. durangensis* fiber [49].

The variable pellet hardness at day 1 was statistically similar in all treatments (Table 4). The values were in the range of 43.92 to 58.45%, i.e., during free fall approximately half of the weight of each pellet was lost regardless of the proportions of agave fiber and pine sawdust with which they were made. This behavior can be attributed to the fact that as the pellet cools it solidifies, which could increase its hardness, and its relationship to the proportion of agave fibers. This argument can be verified with the results reported at 7 days of conditioning, as the values showed significant statistical differences ( $p < 0.001$ ) among the treatments. Treatment T6 showed the highest value (80.55%), which means that the hardness was lower compared to pellets containing agave fiber. These values are similar to those reported by Núñez-Retana et al. for pellets made with mixtures of *Quercus* sp. and *Pinus durangensis* wood sawdust in different proportions [18]. These authors obtained values for *Q. conzattii* of 69.1%, followed by *Q. laeta* (63.7), *Q. sideroxyla* (61.4) and *Q. rugosa* (61%); they also highlighted that the lowest durability occurred in those pellets made only with pine sawdust. Similar hardness values (66.27, 61.74 and 49.49%) were reported for pellets made with *Acacia wrightii*, *Ebenopsis ebano* and *Havardia pallens* wood, respectively, by Carrillo et al. [23].

The impact-resistance index identified statistically significant differences among treatments ( $p < 0.05$ ) evaluated at day 1 and 7 (Table 4). On day 1, the highest value (114.71) was recorded in T1, followed by T2 (116.18) and the values continued to decrease as the proportion of pine sawdust increased until reaching T6 (64.12) (Table 5). Similar behaviour was also observed when this variable was evaluated at 7 days of conditioning, where the values ranged from 133.83 to 126.47 for T1 and T6, respectively. In both conditioning periods, there was a marked tendency for the value to decrease as the proportion of pine

sawdust increased. Impact resistance is an important characteristic of pellets; high values generate fewer fine particles when handled both in storage and during transportation. The values obtained are similar to those reported for pine sawdust pellets made at moisture contents of 7.9 and 11.0%, respectively, by Núñez-Retana et al. [50]. In contrast, pellets made from *Cedrelinga catanaeformis* sawdust obtained values from 86 to 94 [51].

The calorific value of the pellets showed significant statistical differences among treatments ( $p < 0.05$ ). T2, T3 and T5 presented statistical equality (ab) in their average values; likewise, T1 and T6 (b) were similar, while T4 (a) did not present statistical equality with the rest of the treatments (Figure 2). T4 presented the highest calorific value as a result of the mixture of 40–60 agave fiber to pine sawdust. These values are similar to those reported by Soto et al. for pellets made with mixtures of charcoal and *Pinus radiata* wood sawdust [52].

According to estimates made by Tauro et al. [53], in developing countries such as Mexico, given the incipient use of biomass as an energy source, the potential market of pellets for energy use generated from agricultural waste is 131 PJ/year, with the total costs ranging between 6.3 and USD/GJ 12.8; additionally, these authors point out that the distance, means of transport and inherent costs in production affect the competitiveness of bioenergy products.

## 5. Conclusions

The pellets produced in different proportions with *Agave durangensis* Gentry fiber and pine sawdust had good physical, mechanical and energetic characteristics. The mixture of these two types of biomass affected some of their properties to different degrees; however, most of them complied with the parameters established in the UNE-EN ISO 17225-6 standard and those recommended by the European pellet council. As demonstrated by the physical, mechanical and energetic characteristics evaluated in this study, mixtures with a percentage of agave fiber  $\leq 40\%$  constitute an interesting source for the production of renewable energy and provide great possibilities of success in the market.

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## References

1. González Ávila, M.E. Producción de bioenergía en el Norte de México: Tan lejos y tan cerca. *Front. Norte* **2009**, *21*, 177–183.
2. Şahin, B.; Aslan, S.; Ceylan, Z.D.; Karaoğlu, S.Y. Sustainability and socioeconomic impacts of bioenergy. *Bioenergy Stud.* **2021**, *1*, 37–42. [CrossRef]
3. Hernández, M.; Hernández, J. Verdades y mitos de los biocombustibles. *Elementos* **2008**, *71*, 15–18. Available online: <https://elementos.buap.mx/directus/storage/uploads/00000002099.pdf> (accessed on 9 May 2022).
4. Bhatia, S.K.; Kim, S.-H.; Yoon, J.-J.; Yang, Y.-H. Current status and strategies for second generation biofuel production using microbial systems. *Energy Convers. Manag.* **2017**, *148*, 1142–1156. [CrossRef]
5. Vukušić, J.L.; Millenautzki, T.; Cieplik, R.; Obst, V.; Saaïd, A.M.; Clavijo, L.; Zlatanovic, S.; Hof, J.; Mösche, M.; Barbe, S. Reshaping Apple juice production into a zero discharge biorefinery process. *Waste Biomass Valoriz.* **2021**, *12*, 3617–3627. [CrossRef]
6. Katiyar, R.; Banerjee, S.; Arora, A. Recent Advances in the Integrated biorefinery concept for the valorization of algal biomass through sustainable routes. *Biofuels Bioprod. Biorefin.* **2021**, *15*, 879–898. [CrossRef]
7. Forero Nunez, C.A.; Jochum, J.; Sierra Vargas, F.E. Characterization and feasibility of biomass fuel pellets made of Colombian timber, coconut and oil palm residues regarding European standards. *Environ. Biotechnol.* **2012**, *8*, 67–76.
8. Keles, S.; Bilgen, S.; Kaygusuz, K. Biomass energy source in developing countries. *J. Eng. Res. Appl. Sci.* **2017**, *6*, 566–576.

9. Morán, J.I.; Alvarez, V.A.; Cyras, V.P.; Vázquez, A. Extraction of cellulose and preparation of nanocellulose from sisal fibers. *Cellulose* **2008**, *15*, 149–159. [CrossRef]
10. Pérez-Pimienta, J.A.; López-Ortega, M.G.; Sanchez, A. Recent developments in agave performance as a drought-tolerant biofuel feedstock: Agronomics, characterization, and biorefining. *Biofuels Bioprod. Biorefin.* **2017**, *11*, 732–748. [CrossRef]
11. Arreola-Vargas, J.; Flores-Larios, A.; González-Álvarez, V.; Corona-González, R.I.; Méndez-Acosta, H.O. Single and two-stage anaerobic digestion for hydrogen and methane production from acid and enzymatic hydrolysates of agave tequilana bagasse. *Int. J. Hydrogen Energy* **2016**, *41*, 897–904. [CrossRef]
12. Chávez Guerrero, L. Uso de bagazo de la industria mezcalera como materia prima para generar energía. *Ingenierías* **2010**, *13*, 8–16.
13. Rosas Medina, I.; Colmenero Robles, A.; Naranjo Jiménez, N.; Rodríguez García, J.H. El Mezcal de Durango, México. *Vidsupra* **2013**, *5*, 113–117.
14. Moreno-Anguiano, O.; Carrillo-Parra, A.; Rutiaga-Quiñones, J.G.; Wehenkel, C.; Pompa-García, M.; Márquez-Montesino, F.; Pintor-Ibarra, L.F. Chemical composition of *Luffa aegyptiaca* mill., *Agave durangensis* Gentry and *Pennisetum* sp. *PeerJ* **2021**, *9*, e10626. [CrossRef]
15. Schön, C.; Feldmeier, S.; Hartmann, H.; Schwabl, M.; Dahl, J.; Rathbauer, J.; Vega-Nieva, D.J.; Boman, C.; Öhman, M.; Burval, J. New Experimental Evaluation Strategies Regarding Slag Prediction of Solid Biofuels in Pellet Boilers. *Energy Fuels* **2019**, *33*, 11985–11995. [CrossRef]
16. Carrillo-Parra, A.; Rutiaga-Quiñones, J.G.; Ríos-Saucedo, J.C.; Ruiz-García, V.M.; Ngangyo-Heya, M.; Nava-Berumen, C.A.; Núñez-Retana, V.D. Quality of pellet made from agricultural and forestry waste in Mexico. *BioEnergy Res.* **2021**, 1–10. [CrossRef]
17. Moreno-Anguiano, O.; Cloutier, A.; Rutiaga-Quiñones, J.G.; Wehenkel, C.; Rosales-Serna, R.; Rebolledo, P.; Hernández-Pacheco, C.E.; Carrillo-Parra, A. Use of *Agave durangensis* bagasse fibers in the production of wood-based medium density fiberboard (MDF). *Forests* **2022**, *13*, 271. [CrossRef]
18. Núñez-Retana, V.D.; Rosales-Serna, R.; Prieto-Ruiz, J.Á.; Wehenkel, C.; Carrillo-Parra, A. Improving the physical, mechanical and energetic properties of *Quercus* spp. wood pellets by adding pine sawdust. *PeerJ* **2020**, *8*, e9766. [CrossRef]
19. UNE-EN ISO 17827-2; Biocombustibles Sólidos. Determinación de la Distribución de Tamaño de Partícula para Combustibles sin Comprimir. Parte 2: Método Del Tamiz Vibratorio Con Abertura de Malla Inferior o Igual a 3.15 mm. AENOR: Madrid, España, 2016.
20. UNE-EN ISO 18134-3; Determinación del Contenido de Humedad. Método de Secado en Estufa. Parte 1: Humedad Total. Método de Referencia. AENOR: Madrid, España, 2016.
21. UNE-EN ISO 18123; Biocombustibles Sólidos. Determinación del Contenido en Materias Volátiles. AENOR: Madrid, España, 2016.
22. UNE-EN18122; Determinación del Contenido de Ceniza. AENOR: Madrid, España, 2016.
23. Carrillo-Parra, A.; Ngangyo Heya, M.; Colín-Urieta, S.; Foroughbakhch Pournavab, R.; Rutiaga Quiñones, J.G.; Correa-Méndez, F. Physical, mechanical and energy characterization of wood pellets obtained from three common tropical species. *PeerJ* **2018**, *6*, e5504. [CrossRef]
24. Junker, H.; Nikolaisen, L.; Møller, H.; Jensen, P.D.; Hjuler, K.; Dahl, J. Characterization of solid biofuels 2004-Development of Methods. DONG energy. *PSO Proj.* 2008. Available online: [https://www.teknologisk.dk/\\_/media/39484\\_Characterization%20of%20solid%20biofuels%202004%20-%20development%20of%20methods.%20PSO%20project%20nr%205297.pdf](https://www.teknologisk.dk/_/media/39484_Characterization%20of%20solid%20biofuels%202004%20-%20development%20of%20methods.%20PSO%20project%20nr%205297.pdf). (accessed on 9 May 2022).
25. UNE-EN ISO 17828; Biocombustibles Sólidos Determinación de la Densidad a Granel. AENOR: Madrid, España, 2016; 16p.
26. Lam, P.Y.; Lam, P.S.; Sokhansanj, S.; Bi, X.T.; Lim, C.J.; Melin, S. Effects of pelletization conditions on breaking strength and dimensional stability of douglas fir pellet. *Fuel* **2014**, *117*, 1085–1092. [CrossRef]
27. García, R.; González-Vázquez, M.P.; Rubiera, F.; Pevida, C.; Gil, M.V. Co-pelletization of pine sawdust and refused derived fuel (RDF) to high-quality waste-derived pellets. *J. Clean. Prod.* **2021**, *328*, 129635. [CrossRef]
28. Richards, S.R. Physical testing of fuel briquettes. *Fuel Process. Technol.* **1990**, *25*, 89–100. [CrossRef]
29. UNE-EN ISO 18125; Biocombustibles Sólidos. Determinación del Poder Calorífico. AENOR: Madrid, España, 2018.
30. R Core Team. *R: A Language and Environment for Statistical Computing*; R Foundation for Statistical Computing: Vienna, Austria, 2021. Available online: <http://www.Rproject.org/> (accessed on 12 December 2021).
31. UNE EN ISO 17225-6; Biocombustibles Sólidos—Especificaciones y Clases de Combustibles. Parte 6: Pélets No Leñosos Para Uso No Industrial. AENOR: Madrid, Spain, 2014; 16p.
32. Whittaker, C.; Shield, I. Factors affecting wood, energy grass and straw pellet durability—A review. *Renew. Sustain. Energy Rev.* **2017**, *71*, 1–11. [CrossRef]
33. Ahamed, I.; Ali, A.; Ali, B.; Hassan, M.; Hussain, S.; Hashmi, H.; Ali, Z.; Soomro, A. Pelletization of Biomass Feedstocks: Effect of Moisture Content, Particle Size and a Binder on Characteristics of Biomass Pellets. 2021. Available online: <https://www.researchsquare.com/article/rs-163994/v1.pdf> (accessed on 9 May 2022).
34. Boada, L.E.A.; Vargas, F.E.S. Caracterización físico-química de pellets producidos a partir de mezclas 50/50 Carbón Bituminoso/Madera Residual. *Inf. Técnico* **2015**, *79*, 18–25. Available online: [https://revistas.sena.edu.co/index.php/inf\\_tec/article/view/133](https://revistas.sena.edu.co/index.php/inf_tec/article/view/133). (accessed on 9 May 2022). [CrossRef]
35. Kofman, P.D. *The Production of Wood Pellets*; COFORD: Dublin, Ireland, 2007. Available online: [http://www.coford.ie/media/coford/content/publications/projectreports/cofordconnects/cnnpellet\\_production.pdf](http://www.coford.ie/media/coford/content/publications/projectreports/cofordconnects/cnnpellet_production.pdf) (accessed on 31 January 2021).
36. Ragland, K.W.; Aerts, D.J.; Baker, A.J. Properties of wood for combustion analysis. *Bioresour. Technol.* **1991**, *37*, 161–168. [CrossRef]

37. Fernandes, E.R.K.; Marangoni, C.; Souza, O.; Sellin, N. Thermochemical characterization of banana leaves as a potential energy source. *Energy Convers. Manag.* **2013**, *75*, 603–608. [CrossRef]
38. Castillo, Y.A.G.; Rincón, S.N.R.; Vargas, G.C.; Isiordia, G.E.D.; Vargas, F.E.S. Caracterización de materiales lignocelulósicos residuales de palma de aceite y palma de coco para la fabricación de pellets. *Avances Investig. Ing.* **2014**, *11*, 83–91.
39. Barroso León, T.S. Elaboración de Pellets a Partir de Cáscara de Pecana Como Combustible Bioenergético-Cañete-2018. 2018. Available online: <https://hdl.handle.net/20.500.12692/24713> (accessed on 15 December 2021).
40. Romo Ortega, N.; Fernando Toro, A.; Flores Pardo, L.M.; Cañas Velasco, A. Evaluación de las propiedades fisicoquímicas y térmicas de tallos de café y su análisis económico para la producción de pellets como biocombustible sólido. *Ing. Recur. Nat. Ambient.* **2011**, *10*, 79–91. Available online: <https://www.redalyc.org/pdf/2311/231122666007.pdf> (accessed on 9 May 2022).
41. Gurrola-Armendáriz, D.L. Aprovechamiento Integral del Bagazo de Agave Mezcalero Cocido para su uso en la Agricultura y Ganadería. 2016. Available online: <https://www.repositoriodigital.ipn.mx/handle/123456789/24053> (accessed on 11 February 2022).
42. Correa-Méndez, F.; Carrillo-Parra, A.; Rutiaga-Quiñones, J.G.; Márquez-Montesino, F.; González-Rodríguez, H.; Jurado-Ybarra, E.; Garza-Ocañas, F. Moisture and inorganic substance content in pine timber products for use in pellets and briquettes. *Rev. Chapingo Ser. Cienc. For. Ambient.* **2014**, *20*, 77–88. [CrossRef]
43. Demirbaş, A. Sustainable cofiring of biomass with coal. *Energy Convers. Manag.* **2003**, *44*, 1465–1479. [CrossRef]
44. Zamudio-Trejo, D. Producción de Pellets de Residuos de Cultivo de Frijol con Máximo Contenido Energético. 2019. Available online: <http://ri-ng.uaq.mx/handle/123456789/1141> (accessed on 9 May 2022).
45. Brand, M.A.; Jacinto, R.C. Apple pruning residues: Potential for burning in boiler systems and pellet production. *Renew. Energy* **2020**, *152*, 458–466. [CrossRef]
46. Gaitán-Alvarez, J.; Moya, R.; Puente-Urbina, A.; Rodríguez-Zuñiga, A. Physical and compression properties of pellets manufactured with the biomass of five woody tropical species of Costa Rica torrefied at different temperatures and times. *Energies* **2017**, *10*, 1205. [CrossRef]
47. Carrillo Alvarado, V.B.; Valenzuela Macías, J.A. Estudio de las posibilidades de peletización de la cáscara de cacao y su utilización como biocombustible. *Rev. Univ. Guayaquil* **2015**, *121*, 79–84.
48. Mani, S.; Sokhansanj, S.; Bi, X.; Turhollow, A. Economics of producing fuel pellets from biomass. *Appl. Eng. Agric.* **2006**, *22*, 421–426. [CrossRef]
49. Carone, M.T.; Pantaleo, A.; Pellerano, A. Influence of process parameters and biomass characteristics on the durability of pellets from the pruning residues of *Olea europaea* L. *Biomass Bioenergy* **2011**, *35*, 402–410. [CrossRef]
50. Núñez-Retana, V.D.; Wehenkel, C.; Vega-Nieva, D.J.; García-Quezada, J.; Carrillo-Parra, A. The bioenergetic potential of four oak species from northeastern Mexico. *Forests* **2019**, *10*, 869. [CrossRef]
51. Velásquez, S.S.; Bernabé, P.A.S.; Velásquez, N.S.S.; Sánchez, A.C.; Rodríguez, L.E.S.; Castañeda, M.A.A. Optimización del proceso de densificación de desechos lignocelulósicos para la conformación de pellets energéticos. *Sciéndo* **2014**, *17*, 73–80. Available online: <https://www.semanticscholar.org/paper/OPTIMIZACION-DEL-PROCESO-DE-DENSIFICACION-DE-PARA-Seijas-Vel%C3%A1squez-Chavez/24f2f1636a9271fb4f34ee41150532ba28b0bf17> (accessed on 9 May 2022).
52. Soto, G.; Nunez, M. Manufacturing Pellets of Charcoal, Using sawdust of *Pinus radiata* (d. don), as a binder material. *Maderas Cienc. Technol.* **2008**, *10*, 129–137.
53. Tauro, R.; Serrano-Medrano, M.; Maser, O. Solid biofuels in Mexico: A sustainable alternative to satisfy the increasing demand for heat and power. *Clean Technol. Environ. Policy* **2018**, *20*, 1527–1539. [CrossRef]