



Efecto de la intemperización y proceso en las características físicas, mecánicas y energéticas de briquetas

Weathering and process effects on physical, mechanical and energy characteristics of briquettes

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Resumen:

Las industrias forestales generan residuos de aserrín que son subutilizados, frecuentemente estos se acumulan durante años, lo que puede propiciar incendios y contaminación. Los objetivos de la presente investigación fueron comparar las propiedades energéticas entre el aserrín fresco e intemperizado de la madera de *Pinus pseudostrobus*, y determinar algunas propiedades físicas y mecánicas de briquetas elaboradas a partir de ellos. El aserrín se caracterizó según su distribución granulométrica, contenido de humedad, material volátil, cenizas y carbono fijo, de acuerdo con las normas europeas UNE-EN14774-3 y UNE-EN14775. Las briquetas se elaboraron en una máquina marca *LIPPEL* a presiones de 10 y 15 MPa, y temperaturas de 50, 70 y 90 °C; su calidad se determinó mediante análisis proximales, propiedades físicas y mecánicas. Los valores medios y errores estándares se calcularon a las variables: tipo de aserrín, presión y temperatura. Los datos se analizaron estadísticamente como bloques completos al azar y se realizaron análisis de varianza para determinar la existencia de diferencias estadísticas ($p < 0.05$) entre tratamientos. Cuando las hubo, se hicieron pruebas de Tukey. El aserrín intemperizado mostró mejores propiedades energéticas, al presentar menor porcentaje de humedad (9.12 %) y mayor contenido de carbono fijo (13.84 %); sin embargo, presentó un porcentaje superior de cenizas (0.84 %). Las briquetas con mejor calidad se obtuvieron cuando se utilizó aserrín fresco a 15 MPa de presión y temperatura de 70 °C.

Palabras clave. Aserrín intemperizado, biocombustibles sólidos, contenido de ceniza, densidad, poder calorífico, *Pinus pseudostrobus* Lindl.

Abstract:

Forest industries produce sawdust waste that is not used and which may accumulate for years, becoming a risk of fire and a potential source of pollution. The main objectives of this study were to compare the energy properties between fresh sawdust and weathered sawdust from *Pinus pseudostrobus* wood and determine the physical and mechanical properties of briquettes made from both types of sawdust. The sawdust was characterized according to the particle size distribution, moisture content, volatile material, ash, and fixed carbon, in accordance with European standards UNE-EN14774-3 and UNE-EN14775. The briquettes were produced in a laboratory machine (*LIPPEL*) at pressures of 10-15 MPa and temperatures of 50, 70 and 90 °C. The quality of the briquettes was determined by proximal analysis, and by their physical and mechanical properties. The mean values and standard errors were calculated for all the variables of each type of sawdust, pressure and temperature. Data were statistically analyzed as a randomized complete block, and variance analyses were conducted to determine the existence of statistical differences ($p < 0.05$) between treatments. When statistical differences were found, Tukey tests were performed. The weathered sawdust showed the best energy properties, as it exhibited the lowest moisture content (9.12 %) and the highest fixed carbon (13.84 %); however, it had the highest percentage of ash (0.84 %). The briquettes with the best quality were obtained by using fresh sawdust at a pressure of 15 MPa and a temperature of 70 °C.

Key words. Weathering sawdust, solid biofuels, ash content, density, higher heating value, *Pinus pseudostrobus* Lindl.

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Introduction

Biomass densification is a pretreatment which allows to reduce the volume and homogenize the material in order to render it more efficient as fuel (Filbakk *et al.*, 2011).

The global output of densified sawdust products in the form of pellets in the year 2014 was slightly over 24 Mt; Europe was the main producer, with approximately 62 %, followed by North America, with 34 % (Purohit and Chaturvedi, 2016).

On the other hand, the forest industry and forest exploitations generate residues that are underutilized; these include branches and tree tips, chips, slab wood, and sawdust. García *et al.* (2015) point out that the energetic potential of industrial residues amounts to 3.5 % of the total potential production of Mexico, which is 1 713 PJ (petajoules); this percentage is equivalent to 59.9 PJ. However, these residues are not efficiently utilized, and in most cases they are accumulated for years in areas close to the transformation sites (Oyedepo *et al.*, 2014; Ekhuemelo *et al.*, 2016), due to the lack of a market or to the remoteness of the transformation centers (Walker, 2013).

The most common option is to burn the byproducts in the open air, which causes serious problems to humans and to the environment because of the smoke generated by this practice.

A viable alternative for the use of these byproducts is the manufacture of densified products, such as pellets and briquettes. Certain studies have determined the effects of the open-air storage of sawdust at industrial sawmills on the quality of pellets (Arshadi *et al.*, 2008); however, its impact on the quality of briquettes is limited. For this reason, the objectives of the present study were to compare the energy-producing properties of fresh sawdust with those of weathered sawdust of *Pinus pseudostrobus* Lindl. wood, and to determine the density, resistance to compaction, resistance to impact, and higher heating value of briquettes made with both types of sawdust.

Materials and Methods

Sawdust collection and characterization

After a year of weathering, *Pinus pseudostrobus* sawdust was collected in the sawmill of ejido *La Luz*, located in *Iturbide* municipality, *Nuevo León*, Mexico. Fresh sawdust was obtained after the sawing of logs. Both types of sawdust were conditioned to 65 % relative moisture and 20 °C, until they reached a constant weight. The percentage of particles retained in each one of the sieves with No. 4, 6, 8, 20 and 40 meshes was determined; furthermore, the normal distribution of the particle size was estimated using the Kolmogorov-Smirnov procedure (Razali and Wah, 2011). Proximal analyses (of moisture content, volatile matter, ashes, and fixed carbon) and an analysis of the sawdust in bulk were conducted in accordance with the standards ASTM D 1762-84 (2001), UNE-EN-14774 (UNE-EN, 2010 and UNE-EN-15149-2 (UNE-EN, 2011).

Manufacture of briquettes

The briquettes were manufactured in a Lippel™ laboratory briquette-producing machine, with a vertical orientation. The equipment has a solid base, with a cylinder with a 20 mm diameter and an in-built thermostat which allows to modify the temperature, and two pistons that apply the pressure and facilitate the extraction of the briquettes. Each briquette was made using 200 cm³ of sawdust; the pressures utilized were of 10 and 15 MPa, and the temperatures, of 50, 70 and 90 °C; five briquettes with a diameter of 3.3 cm and a length of 8.0 cm were made for each treatment.

Characterization of the briquettes

Density was determined according to the standard UNE-EN-16127 (UNE-EN, 2012). The measurements were carried out 48 hours after the briquettes were manufactured, in order to allow them to stabilize so that the dilation effect would not affect the results (Križan *et al.*, 2015).

Resistance to compaction was obtained by applying a load to the whole length of the briquette, as established by Borowski and Hycnar (2013) and according to the norm ASTM D 143-83 (1994) (Equation 1). The registered value was the force to the breaking point of the briquette; the tests were conducted in a universal Shimadzuc machine with a capacity for 100 kN.

$$RC = L_f \quad (1)$$

Where:

RC = Resistance to compaction

L_f = Force to the point of failure

Resistance to impact was determined by the number of pieces formed when each briquette was dropped onto the ground on three occasions, from a height of two meters (Chaiklangmuang *et al.*, 2008; Rajaseenivasan *et al.*, 2016) (Equation 2).

$$RI = NP \quad (2)$$

Where:

RI = Resistance to impact

NP = Number of manufactured pieces

The heating value of the sawdust and of the briquettes was calculated using the equation developed by Cordero *et al.* (2001), which relates the percentage of fixed carbon to that of the volatile matter (Equation 3).

$$SHP = 0.3563 FC + 0.1755 VM \quad (3)$$

Where:

SHP = Superior heating power (KJ kg⁻¹)

FC = Fixed carbon (%)

VM = Volatile matter (%)

Statistical analyses

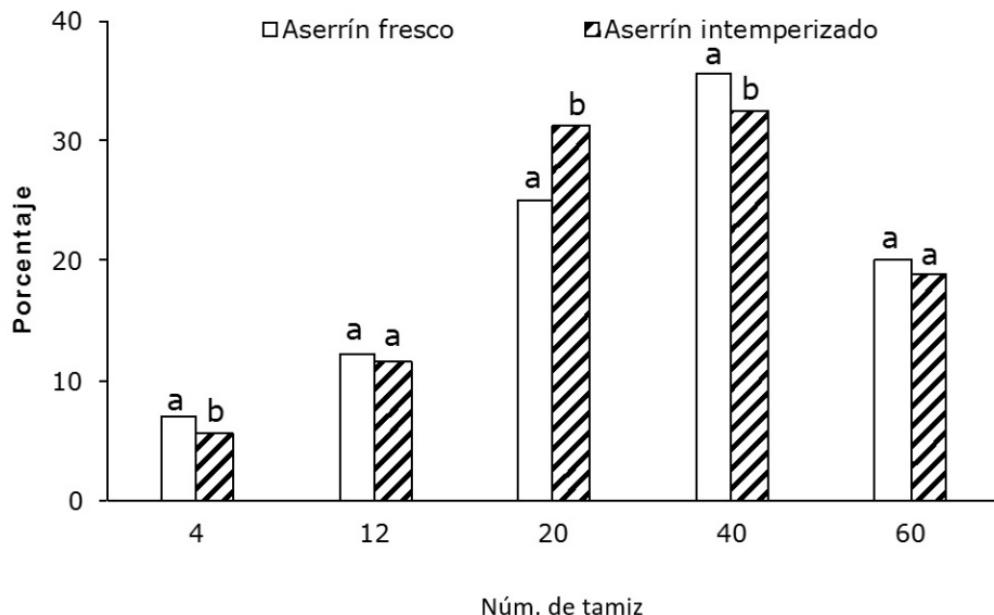
The normality of the data of the variables was determined using the Kolmogorov-Smirnov test (Razali and Wah, 2011). The percentage variables were transformed before the statistical analyses, with the square root of the arc sine (Steel and Torrie, 1960). A factorial design (2 × 2 × 3), in which the sources of variation were: a) type of sawdust, b) pressure, and c) temperature. Each treatment included five repeats.

A variance analysis was applied to the data with a normal distribution in order to determine the existence of statistical differences between treatments ($p<0.05$); when these occurred, Tukey tests were made. Whenever the distribution was not normal, the Kruskal Wallis test was carried out. All the statistical analyses were performed using the R 3.2.1 statistical package (Bolker, 2012).

Results and Discussion

Particle size

The percentages of particles between the two types of sawdust in the sieves with mesh sizes. 4, 20 and 40 were statistically different (Figure 1). The fresh sawdust exhibited higher values for particle sizes with mesh sizes 4 and 40. According to Žandekis *et al.*(2014), particles in the interval of 0.1 to 0.85 mm make products with a better quality. Grover and Mishra (1996) point out that smaller particles are required in order to increase the adhesion with the larger particles. However, the mechanical resistance of the manufactured briquettes was estimated in terms of the balance in the proportion of the smaller and the larger particles.



Porcentaje = Percentage; Núm. de tamiz = Mesh size; Aserrín fresco = Fresh sawdust; Aserrín intemperizado = weathered sawdust.

Bars followed by the same letter in each sieve do not differ statistically for Tukey's mean comparison test with a 95 % confidence interval.

Figure 1. Percentage of fresh and weathered sawdust of the sieves with mesh sizes Nos. 4 (4.76 mm), 12 (1.68 mm), 20 (0.84 mm), 40 (0.42 mm), and 60 (0.35 mm).

Proximal analyses

The moisture content was lower in weathered sawdust (Table 1), which indicates that this type absorbs less moisture under similar conditions, due to hysteresis and to oxygen emission during storage (Alakoski *et al.*, 2016). The moisture content of both types of sawdust corresponds to the interval of 6.39 % to 12.4 % established by Ghaffar *et al.* (2015) and Žandeckis *et al.* (2014) for the manufacture of high-quality briquettes but is 15 % lower than that considered by Grover and Mishra (1996).

Table 1. Moisture content, volatile matter, ashes, and fixed carbon of fresh and weathered *Pinus pseudostrobus* Lindl. Sawdust.

Sawdust	Moisture content (%)	Volatile matter (%)	Ashes (%)	Fixed carbon (%)
Fresh	9.83±0.13a	75.74±0.90a	0.24±0.04b	14.19±0.92a
Weathered	9.12±0.12b	73.72±0.78a	0.83±0.05a	16.33±0.83a

Values followed by the same letter in each column do not differ statistically for Tukey's mean comparison test with a 95 % confidence interval.

The percentage of volatile matter in fresh sawdust coincided with the value established by Brito and Barrichelo (1982), cited by Hansted *et al.* (2016), of 75 to 85 %, while that of weathered sawdust was slightly lower.

As for to the ash content, the percentage in weathered sawdust was statistically higher ($p<0.05$) than that found in fresh sawdust, due to the concentration of inorganic elements during the storage and handling (Thek and Obernberger, 2012; Correa-Méndez *et al.*, 2014). The percentage of ash is an important factor to be considered during combustion at high temperatures, due to the formation of slag in the furnaces (Alakoski *et al.*, 2016). A value of 0.83 %, registered for weathered sawdust, exceeds the accepted limit (0.50 %) for the production of briquettes (ÖNORM, 2000), while the result for fresh sawdust was similar to that cited by Correa-Méndez *et al.* (2014) for *Pinus leiophylla* Schiede. ex Schltdl. & Cham., *P. montezumae* Lamb. and *P. pseudostrobus*.

The percentage of fixed carbon in briquettes made with both types of sawdust exhibited no statistically significant differences; the value for briquettes made from both biomass sources was higher than the one cited by Freitas *et al.* (2016).

Briquettes

Proximal analyses. The lowest moisture content (6.63 %) was found in briquettes made with fresh sawdust at a pressure of 15 MPa and at 70 °C (Table 2). The interaction between the sawdust type and the temperature evidenced significant statistical differences ($P<0.001$) (Table 3). The higher the moisture content, the greater the loss of energy was during combustion, due to water evaporation, which reduced the heating power of the material (Onyenanu *et al.*, 2015). A low moisture content is also desirable in order to reduce the rot caused by fungi, as well as disintegration during transportation.

Table 2. Immediate analyses of briquettes made with fresh and weathered *Pinus pseudotrobus* Lindl. sawdust under various temperatures and pressures.

Pressure (MPa)	Temperature (°C)	Moisture content (%)	Volatile matter (%)	Ashes (%)	Fixed carbon (%)
Fresh sawdust					
10	50	9.41±0.15a	78.62±0.23cd	1.02±0.13a	10.96±0.23c
	70	6.76±0.16c	79.76±0.34abcd	0.87±0.05ab	12.62±0.23ab
	90	7.15±0.14bc	80.87±0.24ab	0.88±0.10ab	11.10±0.31bc
	50	9.34±0.16a	78.75±0.45bcd	0.90±0.09ab	11.01±0.37c
	70	6.63±0.06c	78.52±0.28cd	0.77±0.05abc	14.07±0.29a
	90	7.39±0.27bc	79.43±1.11abcd	0.73±0.07bc	11.56±0.20bc
Weathered sawdust					
15	50	7.72±0.15b	80.98±0.32a	0.28±0.05e	11.03±0.36c
	70	7.48±0.09bc	81.01±0.27a	0.26±0.05e	11.25±0.20bc
	90	8.92±0.21a	78.52±0.26cd	0.62±0.03cd	11.94±0.28bc
	50	7.14±0.14bc	80.26±0.32abc	0.28±0.01e	12.32±0.40bc
	70	7.86±0.16b	80.47±0.48abc	0.42±0.21de	11.25±0.28bc
	90	8.86±0.42a	77.77±0.13d	0.77±0.07abc	12.62±0.47ab

Values followed by the same letter in each column exhibit no statistical difference for Tukey's mean comparison test with a 95 % confidence interval.

Table 3. Variance analysis of the proximal analyses of briquettes made with fresh and weathered *Pinus pseudotrobus* Lindl. sawdust at various temperatures and pressures.

Source of variation	Moisture content		Volatile matter		Ashes		Fixed carbon	
	F	p	F	p	F	p	F	p
Sawdust type (S)	1.568	0.216	3.424	0.070	76.358	0.000	0.235	0.630
Temperature (T)	1.705	0.197	2.174	0.146	7.793	0.007	1.964	0.167
Pressure (P)	0.042	0.839	7.420	0.009	0.001	0.991	5.149	0.027
Interaction (S × T)	54.605	<0.001	33.935	<0.001	22.377	<0.001	0.135	0.715
Interaction (S×P)	0.083	0.775	0.091	0.764	3.711	0.060	0.002	0.967
Interaction (T×P)	0.764	0.386	1.367	0.248	0.144	0.706	0.025	0.874
Interaction (S×T×P)	0.048	0.827	1.311	0.257	0.485	0.489	0.609	0.439

F = F value; p = Level of significance.

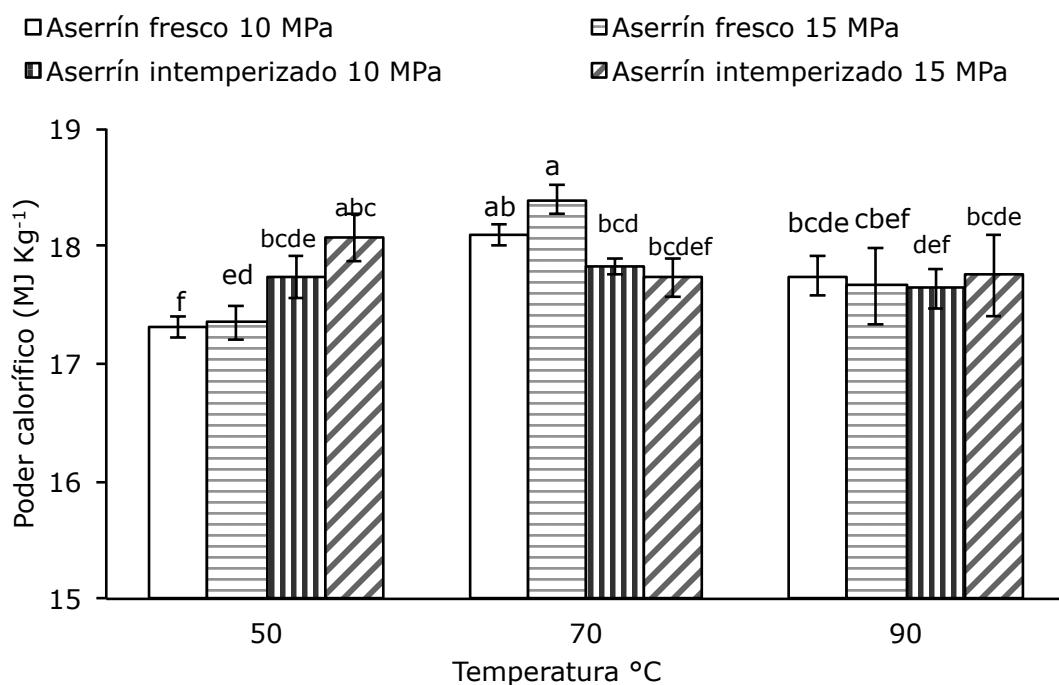
The highest percentage of volatile matter (81.01 %) occurred in briquettes made with weathered sawdust at a pressure of 10 MPa and a temperature of 70 °C (Table 2). The variable pressure and the sawdust:temperature interaction exhibited statistically significant differences (p<0.05) (Table 3).

The reaction index was calculated based on the volatile matter/fixed carbon ratio, and it indicates the potential reaction of the material during pyrolysis (Purohit and Chaturvedi, 2016); therefore, the most reactive briquette was the one made with weathered sawdust at 10 MPa and 50 °C.

The ash content exhibited significant statistical differences (p<0.05) in the variables type of sawdust, temperature and sawdust: temperature and sawdust: pressure interactions (Table 3). The fixed carbon ranged between 10.96 and 14.07 % (Table 2), which is below the 17.87 % cited by Ballarin *et al.* (2010) for *Cupressus lusitanica* Mill. sawdust briquettes.

Heating value

The heating value corresponded to the interval of 17.35 to 18.40 MJ kg^{-1} , of which the highest was for briquettes made with fresh sawdust at a temperature of 70 $^{\circ}\text{C}$ and a pressure of 15 MPa (Figure 2). The results agree with the interval of 16.374 a 19.774 MJ kg^{-1} by Onyenanu *et al.* (2015) for briquettes made with sawdust with various particle size distributions; these values were lower than the 19.678 MJ kg^{-1} indicated by Cardoso *et al.* (2016) for conifer sawdust briquettes, and higher than the 14.68 MJ kg^{-1} documented by Rahaman and Salam (2017) for rice husk briquettes.



Poder calorífico = Heating value; *Temperatura* = Temperature; *Aserrín fresco* = Fresh sawdust; *Aserrín intemperizado* = weathered sawdust.

Bars followed by the same letter in each temperatura do not differ statistically for Tukey's mean comparison test with a 95 % confidence interval.

Figure 2. Heating value of briquettes produced with fresh and weathered *Pinus pseudostrobus* Lindl. sawdust at various pressures and temperatures.

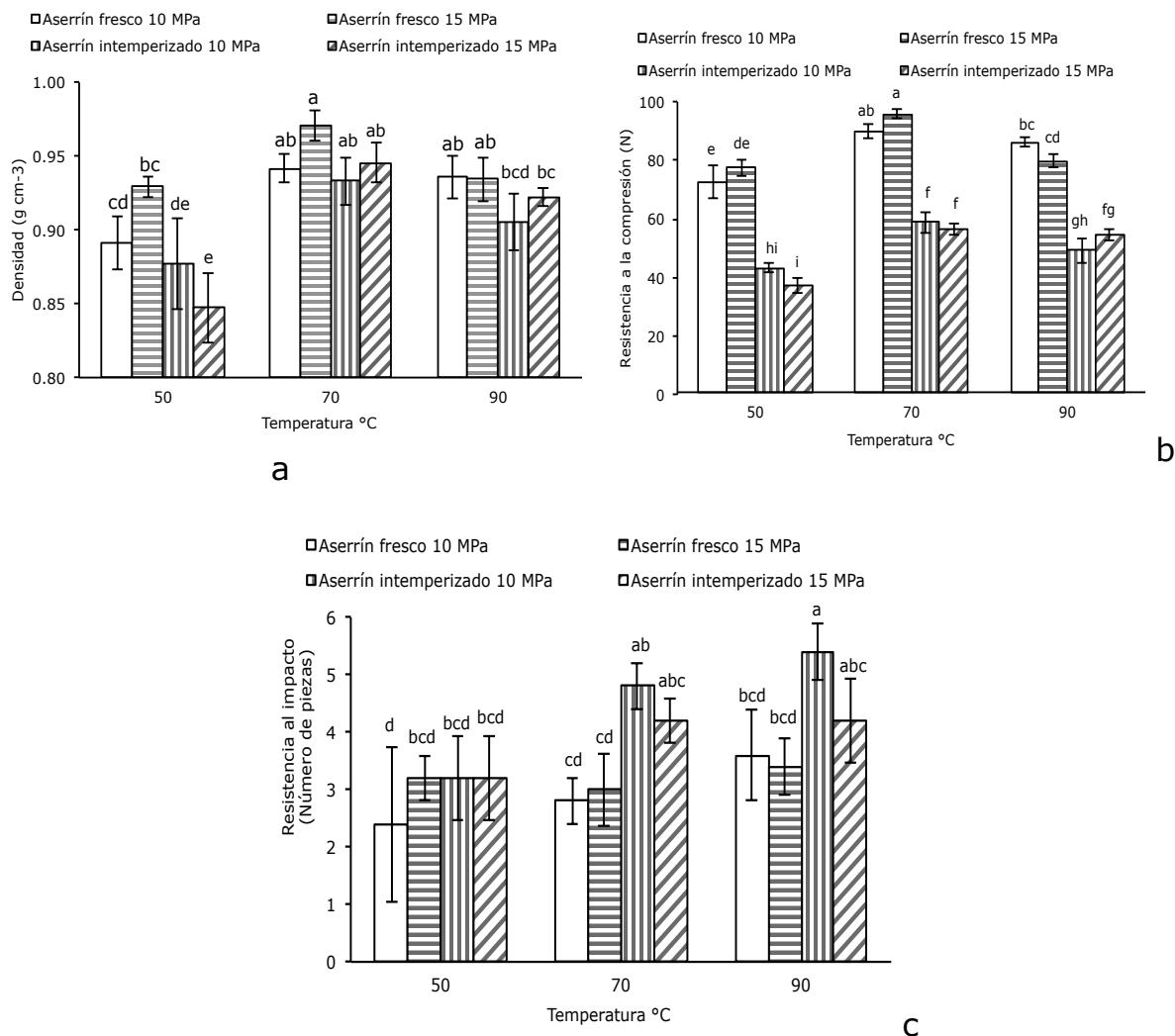
Density

There were significant statistical differences ($p < 0.05$) in the density of the briquettes for the variables type of sawdust, temperature and sawdust:temperature:pressure interaction (Table 4). The highest density was obtained with fresh sawdust at a temperature of 70 °C and a pressure of 15 MPa (Figure 3a). In a similar assay, Filbakket *et al.* (2011) reported denser pellets made with fresh sawdust. On the other hand, Silva *et al.* (2011) point out that different percentages in particle sizes produce differences in the density of biofuels. The higher density is also ascribed to the higher percentage of extracts in fresh sawdust, which may reduce friction during pressing, generating greater mobility of the particles and a better arrangement during pressing.

Table 4. Variance analysis of the physical and mechanical properties of briquettes made with fresh and weathered *Pinus pseudotrobus* Lindl. sawdust at various temperatures and pressures.

Source of variation	Density		Resistance to compaction		Resistance to impact	
	F	p	F	p	F	p
Type of sawdust (S)	14.948	<0.001	317.411	0.000	30.944	<0.001
Temperature (T)	17.768	<0.001	17.712	0.000	22.548	<0.001
Pressure (P)	2.188	0.145	0.016	0.899	0.710	0.403
Interaction (S × T)	2.194	0.145	0.559	0.458	3.452	0.069
Interaction (S × P)	2.166	0.147	0.418	0.521	4.802	0.033
Interaction (T × P)	0.046	0.832	0.000	0.997	5.157	0.027
Interaction (S × T × P)	5.676	0.021	5.804	0.020	0.043	0.837

F = F value; *p* = Level of significance.



Aserrín fresco = Fresh sawdust; *Aserrín intemperizado* = weathered sawdust;
Densidad = Density; *Resistencia a la compresión* = Resistance to compaction;
Resistencia al impacto = Resistance to impact; *Número de piezas* = Number of pieces.

a = Density; b = Resistance to compaction; c Resistance to impact.

Bars followed by the same letter for each sieve do not differ statistically for Tukey's mean comparison test with a 95 % confidence interval.

Figure 3. Physical and mechanical properties of briquettes made with fresh and weathered *Pinus pseudostrobus* Lindl. sawdust at different pressures and temperatures.



Resistance to compaction

The resistance to compaction of the briquettes exhibited statistical differences ($p<0.05$) between the sawdust types, the temperature and the sawdust:temperature:pressure interaction (Table 4). Figure 3 shows that the briquettes made with fresh sawdust at 70 °C and 15 MPa are the most resistant to compaction. The higher resistance of the fresh sawdust briquettes is the result of a greater adhesion between the particles under this condition. Another aspect that affects resistance positively is the higher percentage of particles of less than 0.42 mm (Sieve No. 40). In this regard, Mitchual *et al.* (2013) and Chin and Siddiqui (2000) point out that the particle size affects the mechanical durability of densified products inversely. The percentage of small particles is considered to improve the mechanical resistance up to a certain limit, after which it decreases as the resistance to impact diminishes.

Resistance to impact

The resistance to impact evidenced statistical differences between the sawdust types, temperatures, and the temperature:pressure interaction (Table 4). The fresh sawdust briquettes made at 50 °C and 10 MPa were more resistant to impact as they generated a smaller number of pieces.

Conclusions

Certain energetic quality parameters, such as the high content of volatile matter, the high reactivity index and the low content of ashes make weathered sawdust a better biofuel than fresh sawdust. However, when both sawdust types are densified under equal conditions, briquettes made with weathered material exhibit a lower quality in certain parameters than those produced with fresh sawdust.

The positive bioenergetic parameters exhibit by weathered sawdust briquettes are a lower content of moisture, fixed carbon and ashes, and a higher percentage of volatile matter. While the negative parameters are less density and a lower

resistance to compaction, due to the lower percentage of small particles and to the higher production temperatures, of 70 and 90 °C.

Notwithstanding, weathered sawdust stored at the facilities of forest industries may produce briquettes with high-quality bioenergetic parameters, such as a low content of ashes, and a high density and resistance to compaction, provided that a selection of particles is carried out, and a temperature of 70 °C and a pressure of 10 or 15 MPa are utilized.

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Conflict of interests

The authors no declare conflict of interests.

Contribution by author

Artemio Carrillo-Parra: achievement of the project in which this research was made, support in the selection of variables, structuring and writing of the manuscript; Esteban Contreras Ortiz: development of laboratory work, data capture and analysis, and writing of the manuscript; Fortunato Garza-Ocañas: support in the structure, writing and final review of the manuscript; Maginot Ngangyo Heya: data statistiacal analysis and general review of the manuscript; Guadalupe Rutiaga-Quiñones: general review of the manuscript.

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